Data Repository: Uba, Strecker, and Schmitt

U-Pb radiometric dating

The age controls for the stable isotopic records presented here are from both published sources and U-Pb geochronology performed in this study. We analyzed six zircon separates by U-Pb geochronology with secondary ionization mass spectrometry (SIMS, ion microprobe) using the Cameca ims 1270 at UCLA. For each of the samples, the volcanic ashes were crushed, sieved and the zircons were separated using heavy liquid procedures and magnetic separation techniques. About 10 - 30 zircons per sample were then picked by hand and placed in epoxy mounts, which were polished and gold-coated for analysis. A ~15 nA O^- primary beam with 22.5 keV total impact energy was focused to a ~25 μm diameter spot. Secondary ions were extracted at 10 kV with an energy band pass of 50 eV. O_2 pressure in the sample chamber was adjusted to ~0.002 Pa, which increased Pb sensitivity by ~50 %. In three analytical sessions the $^{206}Pb/^{238}U$ age reproducibility of 33, 19, and 24 analyses (July 4th 2006, August 20th 2006, and April 6th 2007, respectively) of reference zircon AS-3 was between 2 and 3 % (1 standard deviation). All U-Pb ages were corrected for initial ^{230}Th deficit using measured zircon U/Th and whole-rock U/Th = 2.3 (Central Andean ignimbrite average; Schmitt et al., 2002). This correction typically adds ~0.1 Ma to the equilibrium $^{206}Pb/^{238}U$ ages. Ion microprobe results for samples are presented in Data Repository Table 1 and Tera-Wasserburg isochron plots are shown in Data Repository Fig. 1.

Due to extremely slow diffusion of U and Pb in zircon, crystallization ages are essentially unaffected by pre-eruptive crystal storage even at elevated temperatures. This is in contrast to K-Ar (Ar-Ar) or zircon fission track dates, in which any memory of pre-eruptive crystallization is erased. There are also numerous cases where U-Pb zircon ages of young volcanic rocks significantly predate the eruption due to magmatic assimilation of older wall-rocks or mechanical entrainment of zircons from country rock fragments or sediment during eruption and/or deposition, collectively termed “detrital”. Detrital contamination typically results in complex zircon age distributions, whereas zircon age spectra with a single dominant age peak indicate minor assimilation or reworking. Pre-eruptive zircon residence or zircon recycling from just solidified precursor intrusions may cause an overestimation of the eruption age, but crystallization just prior to the eruption is frequently recorded in zircon (e.g., Lanphere et al., 2004), and by targeting zircon rims we aimed to enhance the detection of near-eruption crystallization.

Zircon U-Pb age distributions for four out of six dated tephra samples (TSV 01, TSV 02, Tang 01, Tang 02, Tang 03, Tang 04) show a dominant young age peak with heterogeneous older ages present in varying proportions. After excluding older detrital or xenocrystic zircons, the remaining populations are homogeneous and yield the following average zircon crystallization ages for Puesto Salvacíon samples: 10.4 ± 0.2 Ma (TSV 01; age uncertainties quoted at 95% confidence) and 7.93 ± 0.26 Ma (TSV 02); and Angosto de Pilcomayo: 7.51 ± 0.24 Ma (Tang 02), 7.31 ± 0.20 (Tang 03), and 5.94 ± 0.16 Ma (Tang 04).

U-Pb ages for tephra samples TSV 02 and Tang 02 overlap within uncertainty, and zircons are also indistinguishable in their U abundance (Data Repository Table 1 and Fig. 1). It is therefore permissible to consider both samples as part of the same tephra, but given the continuous and frequent silicic volcanic activity within the Altiplano - Puna region throughout the Miocene, they may as well have originated from separate eruptions. The sample Tang 01 (base of the Angosto de Pilcomayo section) has high proportions of detrital zircons. Because we initially screened the samples by rapid collection of the ^{206}Pb signal and excluded clearly identifiable old grains from further analysis, the reported ages in Data Repository Table 1 and Fig. 1 are thus already biased to younger ages. Closely overlapping U-Pb ages are obtained for three zircon grains from Tang 01 (average 12.4 ± 0.5 Ma). We interpret these ages as maximum depositional ages for each tephra. In summary, the U-Pb zircon chronostratigraphy for tephra samples from the Puesto Salvacíon and Angosto de Pilcomayo sections reveals that deposition of the Yecua formation may have started as early as ~12.5 Ma and was well under way by 10.4 Ma. The base of the overlying Tariquia Formation is dated to ~7.5 Ma, and the age of the upper Tariquia Formation is constrained by the youngest tephra dated at ~6 Ma.

References

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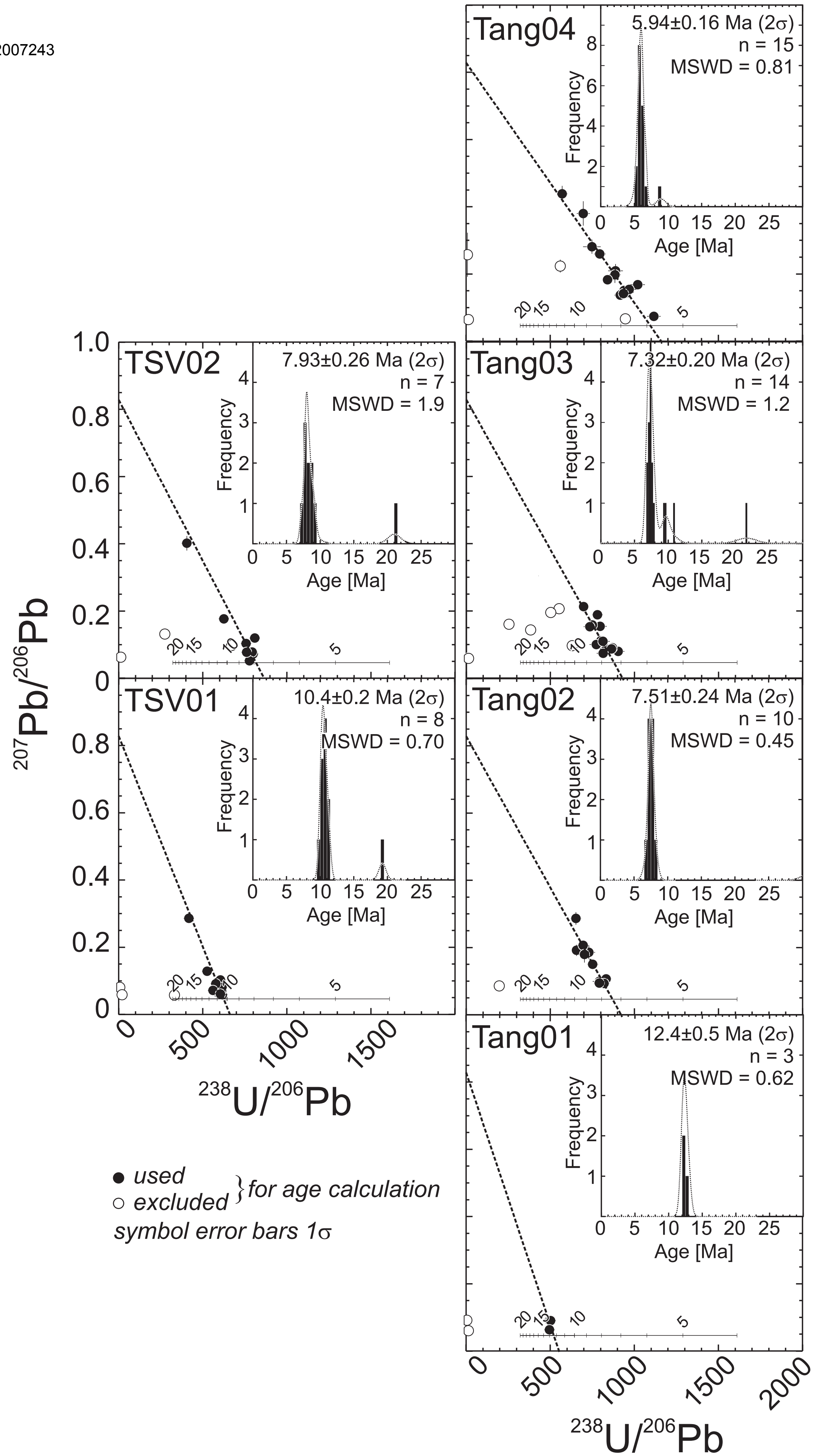

Tang04
5.94±0.16 Ma (2σ)
n = 15
MSWD = 0.81
TSV02
7.93±0.26 Ma (2σ)
n = 7
MSWD = 1.9
Tang03
7.32±0.20 Ma (2σ)
n = 14
MSWD = 1.2
TSV01
10.4±0.2 Ma (2σ)
n = 8
MSWD = 0.70
Tang02
7.51±0.24 Ma (2σ)
n = 10
MSWD = 0.45
Tang01
12.4±0.5 Ma (2σ)
n = 3
MSWD = 0.62
Frequency
Age [Ma]
207Pb/206Pb
238U/206Pb
● used
○ excluded
for age calculation
symbol error bars 1σ

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sample	grain	spot	analysis date	$^{238}U/^{206}Pb$	$^{238}U/^{206}Pb$ 1 s.e.	$^{207}Pb/^{206}Pb$	$^{207}Pb/^{206}Pb$ 1 s.e.	Correlation of Concordia Ellipses	$^{206}Pb/^{238}U$ Age [Ma]	±1s.e. [Ma]	U ppm	UO/U	% $^{206}Pb^*$	remarks
TSV01	10	1	7/4/2006	594.2	17.6	0.0631	0.0026	-0.09	10.7	0.3	1496	9.1	97.9	
TSV01	11	1	7/4/2006	616.5	16.9	0.0588	0.0018	-0.02	10.4	0.3	2015	9.4	98.4	
TSV01	2	1	7/4/2006	611.2	16.0	0.0706	0.0026	-0.07	10.3	0.3	2416	9.3	96.9	
TSV01	3	1	7/4/2006	605.0	16.9	0.1026	0.0041	-0.21	10.0	0.3	754	9.7	92.8	
TSV01	5	1	7/4/2006	6.3	0.5	0.0806	0.0018	0.06	939	72	186	10.4	98.7	†
TSV01	5	2	7/4/2006	20.4	0.6	0.0588	0.0005	-0.01	306	9	524	9.0	99.2	†
TSV01	6	1	7/4/2006	417.5	13.1	0.2860	0.0080	0.04	10.8	0.5	1895	9.2	69.3	
TSV01	7	1	7/4/2006	613.5	16.4	0.0805	0.0034	-0.12	10.1	0.3	1054	9.6	95.6	
TSV01	8	1	7/4/2006	577.7	17.2	0.0913	0.0050	-0.23	10.6	0.3	2294	9.2	94.3	
TSV01	8	2	7/4/2006	561.2	16.6	0.0716	0.0032	-0.05	11.2	0.3	1534	8.9	96.8	†
TSV01	9	1	7/4/2006	331.6	9.1	0.0573	0.0016	0.09	19.2	0.5	1425	9.2	98.6	†
TSV01	12	1	8/20/2006	525.8	16.1	0.1289	0.0050	0.07	11.1	0.4	1645	8.1	89.4	†
TSV01	13	1	8/20/2006	605.0	18.8	0.0607	0.0030	-0.07	10.6	0.3	2583	8.1	98.2	
								w.m.	**10.4**					
								±1s	**0.1**					
								MSWD	**0.70**					
								n	**8**					
TSV02	1	1	7/4/2006	12.5	0.4	0.0629	0.0009	0.01	492	15	133	9.2	99.2	†
TSV02	10	1	7/4/2006	799.4	24.5	0.0725	0.0020	0.16	7.89	0.25	3687	9.0	96.6	
TSV02	11	2	7/4/2006	793.7	23.7	0.0780	0.0032	0.10	7.89	0.25	1932	8.9	95.9	
TSV02	2	1	7/4/2006	625.8	21.1	0.1767	0.0110	-0.13	8.66	0.39	587	9.2	83.3	†
TSV02	3	1	7/4/2006	809.1	26.2	0.1196	0.0116	-0.12	7.31	0.29	545	9.3	90.6	
TSV02	4	1	7/4/2006	672.0	23.1	0.0976	0.0063	-0.26	9.05	0.34	1305	9.1	93.4	†
TSV02	5	2	7/4/2006	777.6	27.8	0.0521	0.0021	-0.11	8.33	0.30	2207	9.2	99.2	
TSV02	7	1	7/4/2006	757.0	24.4	0.1030	0.0064	-0.15	7.99	0.28	780	9.5	92.7	
TSV02	9	1	7/4/2006	405.5	21.4	0.4016	0.0219	-0.26	8.75	1.10	479	9.0	54.5	
TSV02	12	1	8/20/2006	760.5	27.9	0.0772	0.0050	0.02	8.23	0.32	867	8.0	96.0	
TSV02	13	1	8/20/2006	274.0	10.7	0.1317	0.0072	-0.21	21.0	1.0	431	8.0	89.1	†
								w.m.	**7.93**					
								±1s	**0.13**					
								MSWD	**1.35**					
								n	**7**					
Tang01	2	1	7/4/2006	13.8	0.5	0.0605	0.0018	0.17	448	14	119	9.4	99.4	†
Tang01	3	1	7/4/2006	495.8	14.1	0.0908	0.0043	-0.07	12.3	0.4	1162	9.2	94.3	
Tang01	4	1	7/4/2006	502.3	16.5	0.0902	0.0035	0.18	12.2	0.4	1186	9.0	94.4	
Tang01	5	1	7/4/2006	495.5	16.6	0.0634	0.0020	0.01	12.8	0.4	2002	8.9	97.8	
Tang01	7	1	7/4/2006	4.2	0.1	0.0916	0.0005	0.00	1382	38	468	8.9	99.5	†
								w.m.	**12.41**					
								±1s	**0.24**					
								MSWD	**0.62**					
								n	**3**					
Tang02	10	1	7/4/2006	196.4	7.1	0.0859	0.0061	-0.17	31.2	1.2	451	9.2	95.0	†
Tang02	4	1	7/4/2006	728.9	38.0	0.1859	0.0204	-0.39	7.36	0.54	494	9.1	82.1	
Tang02	5	1	7/4/2006	655.7	30.6	0.1917	0.0166	-0.12	8.10	0.52	347	9.2	81.4	
Tang02	6	1	7/4/2006	694.9	21.0	0.2063	0.0084	0.06	7.43	0.30	995	9.3	79.5	
Tang02	7	2	7/4/2006	791.1	39.9	0.0994	0.0037	0.03	7.68	0.41	1111	9.3	93.2	
Tang02	8	1	7/4/2006	652.3	31.7	0.2865	0.0190	-0.19	6.94	0.57	238	9.3	69.3	
Tang02	9	1	7/4/2006	751.9	32.0	0.1503	0.0137	-0.34	7.52	0.41	682	9.2	86.7	
Tang02	11	1	8/20/2006	831.9	29.5	0.1064	0.0102	-0.23	7.24	0.30	776	7.9	92.3	
Tang02	12	1	8/20/2006	819.0	28.8	0.0931	0.0083	0.03	7.49	0.29	914	8.1	94.0	
Tang02	13	1	8/20/2006	703.7	31.9	0.1792	0.0240	-0.29	7.70	0.52	559	7.9	83.0	
Tang02	14	1	8/20/2006	791.1	29.0	0.0945	0.0124	0.06	7.73	0.32	792	8.0	93.8	
								w.m.	**7.51**					
								±1s	**0.12**					
								MSWD	**0.45**					
								n	**10**					
Tang03	1	1	4/6/2007	779.4	29.2	0.1884	0.0045	-0.12	6.86	0.32	665	7.1	81.8	
Tang03	2	1	4/6/2007	254.3	12.9	0.1601	0.0146	-0.16	21.7	1.4	80	6.9	85.5	†
Tang03	5	1	4/6/2007	902.5	37.6	0.0795	0.0050	-0.21	6.94	0.30	938	6.9	95.7	
Tang03	6	1	4/6/2007	800.6	35.0	0.1111	0.0055	0.08	7.48	0.36	1124	6.7	91.7	
Tang03	7	1	4/6/2007	697.4	30.2	0.2125	0.0085	0.14	7.38	0.42	958	6.8	78.7	
Tang03	8	1	4/6/2007	772.8	40.7	0.0999	0.0035	-0.15	7.87	0.44	1917	7.0	93.1	
Tang03	11	1	4/6/2007	13.0	0.5	0.0603	0.0006	0.03	477	18	223	7.0	99.5	†
Tang03	12	1	4/6/2007	551.6	34.4	0.2068	0.0132	-0.33	9.38	0.80	769	6.7	79.5	
Tang03	13	1	4/6/2007	502.0	32.8	0.1955	0.0198	-0.37	10.5	0.9	1092	6.6	80.9	†
Tang03	14	1	4/6/2007	831.9	37.0	0.0868	0.0028	0.05	7.45	0.35	2437	6.7	94.8	
Tang03	16	1	4/6/2007	868.8	33.6	0.0901	0.0035	0.00	7.09	0.29	939	6.9	94.4	
Tang03	17	1	4/6/2007	13.1	0.5	0.0587	0.0008	0.03	472	19	229	6.8	99.7	†
Tang03	20	1	4/6/2007	796.2	38.9	0.1539	0.0100	0.06	7.08	0.41	445	6.8	86.2	
Tang03	21	1	4/6/2007	863.6	41.2	0.0867	0.0038	-0.12	7.18	0.36	804	6.7	94.8	
Tang03	22	1	4/6/2007	813.7	37.9	0.0742	0.0040	-0.20	7.74	0.37	1232	6.8	96.4	
Tang03	23	1	4/6/2007	629.3	31.6	0.0963	0.0029	0.05	9.69	0.52	1630	6.5	93.6	†
Tang03	24	1	4/6/2007	749.1	43.1	0.1581	0.0078	-0.31	7.46	0.52	798	6.9	85.7	
Tang03	26	1	4/6/2007	813.0	40.1	0.1092	0.0067	-0.22	7.39	0.40	319	6.9	92.0	
Tang03	27	1	4/6/2007	735.3	38.3	0.1527	0.0093	-0.15	7.67	0.48	498	6.6	86.4	
								w.m.	**7.32**					

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sample	*grain*	*spot*	*analysis date*	*$^{238}U/^{206}Pb$*	*$^{238}U/^{206}Pb$* 1 s.e.	*$^{207}Pb/^{206}Pb$*	*$^{207}Pb/^{206}Pb$* 1 s.e.	*Correlation of Concordia Ellipses*	*$^{206}Pb/^{238}U$ Age [Ma]*	*±1s.e. [Ma]*	*U ppm*	*UO/U*	*% ^{206}Pb**	*remarks*
								±1s	**0.10**					
								MSWD	**1.16**					
								n	**14**					
Tang04	1	1	7/4/2006	1115.9	40.6	0.0742	0.0045	-0.15	5.65	0.21	2329	9.2	96.4	
Tang04	2	1	7/4/2006	747.9	52.7	0.2811	0.0212	-0.16	6.10	0.70	265	9.0	70.0	
Tang04	3	1	7/4/2006	559.9	28.8	0.2234	0.0192	-0.10	8.98	0.68	219	8.9	77.3	†
Tang04	5	1	7/4/2006	888.1	43.9	0.2092	0.0204	0.06	5.83	0.41	267	9.3	79.1	
Tang04	7	1	7/4/2006	1019.9	43.2	0.1684	0.0109	0.01	5.41	0.28	277	9.2	84.4	
Tang04	8	1	7/4/2006	885.0	34.1	0.1977	0.0246	-0.31	5.95	0.37	324	8.8	80.6	
Tang04	21	1	7/4/2006	841.0	31.0	0.1827	0.0101	0.09	6.41	0.30	1508	9.0	82.6	
Tang04	30	1	7/4/2006	571.4	26.7	0.4387	0.0237	-0.19	5.69	0.72	820	9.3	49.8	
Tang04	31	1	7/4/2006	794.9	34.6	0.2598	0.0135	-0.04	5.97	0.39	393	9.4	72.7	
Tang04	32	1	7/4/2006	939.0	35.4	0.1507	0.0112	0.22	6.03	0.27	636	9.2	86.6	
Tang04	33	1	7/4/2006	916.6	34.8	0.1372	0.0111	-0.14	6.29	0.29	1212	9.4	88.4	
Tang04	34	1	7/4/2006	13.1	0.5	0.0643	0.0012	0.00	471	17	268	9.3	99.0	†
Tang04	22	1	7/4/2006	957.9	38.7	0.1456	0.0096	-0.24	5.96	0.29	858	9.2	87.3	
Tang04	23	1	7/4/2006	954.2	34.5	0.1440	0.0082	-0.08	5.98	0.26	552	9.2	87.5	
Tang04	24	1	7/4/2006	8.4	0.4	0.2574	0.0653	0.02	547	66	46	9.1	74.2	†
Tang04	25	1	7/4/2006	947.0	26.5	0.0664	0.0018	0.02	6.71	0.19	5001	9.5	97.4	†
Tang04	26	1	7/4/2006	969.0	42.4	0.1542	0.0079	0.17	5.81	0.30	603	9.5	86.2	
Tang04	27	1	7/4/2006	935.5	27.3	0.1419	0.0065	0.13	6.12	0.21	1380	9.1	87.8	
Tang04	28	1	7/4/2006	696.9	38.6	0.3799	0.0365	0.22	5.39	0.66	197	9.3	57.3	
								w.m.	**5.94**					
								±1s	**0.08**					
								MSWD	**0.81**					
								n	**15**					

* radiogenic
† not used for weighted average calculations
UO^+/U^+ calibration range

7/4/2006	8.4-9.7
8/20/2006	7.9-8.6
4/6/2007	6.5-7.3