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High-precision U–Pb ages in the early Tithonian to early Berriasian and implications for the numerical age of the Jurassic–Cretaceous boundary

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1. Sample preparation

Ash beds were crushed using a tungsten mill. The crushed samples were then sieved at 300 μm. A water flotation method was used to separate and remove the very fine grain size material (less than 20 microns), resulting in a fine material with mineral grains ranging from 20 to 300 μm. The material was then separated with a magnetic separator at a current of 1.8A using a Frantz unit. The non-magnetic material was then run through heavy liquid density separation method using Methyl Iodide from Geoliquids Inc. Minerals denser than 3.33g/cm^3 sank and collected, were zircons were expected to be found. Single grains zircons were then hand-picked using a transmitted light microscope.

2. Laboratorial procedures

Ten to twenty grains were selected for annealed at 900^{o}C for 48 hours. Grains from each sample were then separately chemically abraded, in bulk, for 12 hours in 3 ml Teflon beakers at 210^{o}C with 12N HF inside a pressure dissolution vessel. Following chemical abrasion, the grains were then individually placed inside 3ml Teflon beakers rinsed and cleaned. The cleaning of grains consisted of several steps. In the first step, grains are left for 12 hours (overnight) in 6.2N HCl inside 3ml Savilex beaker at 80^{o}C on a hot plate. In the second step, the grains were cleaned using 7N HNO_3, which consisted of a 45 min hot bath on a sonicator with and an additional 60 min hot bath on hotplate at 80^{o}C. The 7N HNO_3 was then removed from the beakers and the process repeated three times before moving to total dissolution of the grains. Total dissolution consisted of grains being placed individually in microcapsules in 12N HF for 48 hours at 210^{o}C. After total dissolution, microcapsules were then placed on a hotplate at 120^{o}C uncapped to evaporate the remaining HF remaining from the total dissolution. Subsequently, 6.2N HCl was added to the microcapsules and put back in the oven for 12 hours at 180^{o}C for total conversion. After the conversion of acids, the microcapsules were then placed on a hot at 80^{o}C to evaporate the 6.2N HCl remaining from conversion. After evaporation, 3.1N HCl was added to the microcapsules before eluting Pb and U using micro columns. The micro columns were first cleaned using four steps alternating 6.2N HCl and ultrapure H_2O. Samples were collected in 7ml savillex beakers. Cleaning of 7 ml beakers are done in a three-step process of 12h in 6.2N HCl, 12h in 12N HF, and 12h of 6.2N HCl on the hotplate at 80^{o}C. General chemical abrasion procedures are followed after Mattinson (2005).

3. Data acquisition

After the full laboratorial procedures, the samples were dissolved into a silica gel and gently placed on outgassed Re filaments. Isotopic ratios were acquired using Thermal Ionization Mass Spectrometry. Samples were run using a either the Thermo Finnigan TRITON or Isotopx Phoenix in the Department of Earth Sciences, University of Geneva, Switzerland. Lead (Pb) measurements were acquired in dynamic mode on the SEM (Triton) or the Daly (Pheonix). Auto focusing and Peak centering were performed at the beginning of each block, which consisted of 20 cycles each. Lead baseline was monitored by mass 203.5. Interferences on mass ^{202}Pb and ^{205}Pb were monitored by masses 201 and 203, respectively. EARTHTIME ^{202}Pb-^{205}Pb-^{235}U-^{233}U and EARTHTIME ^{205}Pb-^{235}U-^{233}U were used as spikes (Parrish et al., 2006; Parrish and Krogh, 1987). Each measured ratio was corrected for fractionation using a ^{202}Pb/^{205}Pb of 0.99989 when the ^{202}Pb-^{205}Pb-^{235}U-^{233}U and EARTHTIME was used. When EARTHTIME ^{205}Pb-^{235}U-^{233}U was used Pb fractionation was assumed to be 0.13±0.5 % a.m.u (2 S.D). All common Pb measured was assumed to be from laboratorial blanks. Any sample that contained over 1pg of common Pb was considered to be contaminated and discarded.

Uranium measurements were made in static mode on Faraday cups 10^{12} Ω resistors as UO_2^+. Auto focusing and peak centering was performed at the beginning of every block, with each block consisting of 20 cycles each. Baselines were monitored on ±0.5 mass units. ^{238}U/^{235}U of the sample and blank was assumed to be 137.88 (Chen and Wasserburg, 1980). The oxide correction in U measurements was assumed ^{16}OU/^{18}OU = 0.002. Uranium decay constant values were used from Jaffey et al. (1971).

Raw U-Pb data was reduced using U-Pb Redux (Bowring et al., 2012) and the data reported in TS.1. Uncertainty and error propagation algorithm used in Redux software is described in McLean et al. (2011). For the initial Th disequilibrium deviation was calculated using ^{208}Pb and a Th/U of the magma was assumed to be 3.5 (Blackburn et al., 2013).

4. Ash bed age interpretation

All zircons considered in the age distribution of the ash are interpreted as ash-fall deposits from near-by volcanic eruptions, even though the exact source cannot be known. Individual zircon grains from each sample were analyzed and subsequently the youngest subset of grains from the age distribution of each sample were selected for the final weighted mean age. Ages reported in Fig. 2 and throughout the text are ^{206}Pb/^{238}U weighted mean Th-corrected ages.

Uncertainties are reported as X/Y/Z; X includes analytical uncertainty, Y includes additional tracer (ET2535) calibration uncertainty, and Z includes additional ^{238}U decay constant uncertainty. The final weighted mean ages are interpreted as a depositional age for the ash beds. Chemical abrasion is assumed to eliminate the effect of Pb loss in individual grains. Therefore, dispersion of individual grains bigger than the analytical uncertainty in individual grains were assumed to record prolonged residence of zircon in the magmatic systems as well as intramagmatic recycling.

5. Sample Description

5.1. Ash bed LL3

Ash bed LL3 is located at stratigraphic height 54 m in the Las Loicas section. Zircon yield was moderate to high ca. > 50 grains. Zircons crystals ranged from 40-80 μm in size. Grains were mainly rounded with aspect ratio of 1:3 with rare prismatic crystals being rare. Radiogenic Pb ranged from 3 to 10 pg (TS.1). A total of nine grains were selected to represent the age distribution of the sample, with 5 grains being discarded for being too old (>~150 Ma) and considered either inherited grains or prolonged magmatic residence, or magmatic recycling. A total of four younger grains (z23, z25, z26, z32) that overlapped were considered for the final weighted mean age of the ash bed. The final weighted mean age of the ash bed is 139.238 ± 0.049/0.061/0.16 Ma, MSWD = 0.56

5.2.Ash bed LL9

Ash bed LL9 is located in stratigraphic height 41 m in the Las Loicas section. Zircon yield was moderate to high ca. > 50 grains. Zircons crystals ranged from 40-80 μm in size. Grains were mainly prismatic with aspect ratio of 1:8. Radiogenic Pb ranged from 2.5 to 14 pg (TS.1). A total of eleven grains were selected to represent the age distribution of the sample, with 7 grains being discarded for being too old (>~150 Ma) and considered either inherited grains or prolonged magmatic residence, or magmatic recycling. A total of four younger grains (z2, z12, z51, z55) that overlapped were considered for the final weighted mean age of the ash bed of 139.956 ± 0.063/0.072/0.17 Ma, MSDW 0.34

5.3.Ash bed LL10

Ash bed LL10 is located in stratigraphic height 31 m in the Las Loicas section. Zircon yield was moderate to high ca. > 50 grains. Zircons crystals ranged from 40-80 μm in size.

Grains were mainly prismatic with aspect ratio of 1:8. Radiogenic Pb ranged from 1 to 6 pg (TS.1), notably lower than other the other samples. A total of eight grains were selected to represent the age distribution of the sample, with 4 grains being discarded for being too old (>~150 Ma) and considered either inherited grains or prolonged magmatic residence, or magmatic recycling. A total of four younger grains (z41, z42, z44, z45) that overlapped were considered for the final weighted mean age of the ash bed of 140.338 ± 0.083/0/091/0.18 Ma, MSWD = 1.1

5.4. Ash bed LL13

Ash bed LL13 is located in stratigraphic height 2 m in the Las Loicas section. Zircon yield very low ca. > 10 grains. Zircons crystals ranged from 40-80 μm in size. Grains were mainly prismatic with aspect ratio of 1:8. Radiogenic Pb ranged from 7 to 8 pg (TS.1). The only three three grains that overlapped and reasonably respecting stratigraphic superposition were selected to represent the age of the sample, with 7 grains being discarded for being too (>~450 Ma) and having no geological meaning to the age of the sample. Grains that are much older than the youngest population are hard to interpret if they are inherited grains or detrital, therefore they were discarded. A total of 3 younger grains (z9, z33, z34) that overlapped were considered for the final weighted mean age of the ash bed of 142.039 ± 0.058/0.069/0.17 Ma, MSWD 3.5.

5.5. Ash bed LY5

Ash bed LY5 is located 1.5 m below the contact of the Vaca Muerta Fm. and the Tordillo Fm (FS. 1B). Zircon yield was high ca. > 150 grains. Zircons crystals ranged from 20-100 μm in size. Grains were mainly prismatic with aspect ratio of 1:8 and rounded grains with aspect ratio of 1:3 were also very common. The ash bed is located in the Tordillo Fm, a silisiclastic unit. This sample had a significant amount of inherited and or detrital grains in the sample distribution. In a first batch of dated grains by CA-ID-TIMS the age distribution had a very large interval 180-450 Ma. Therefore, to optimize time we scanned the distribution of ages of the sample via LA-ICP-MS U-Pb geochronology. Grains were imaged via Cathodoluminescence and 250 grains were analyzed. Subsequently, the twenty youngest grains (135-145 Ma), based on Concordia ages, were selected to be analyzed via CA-ID-TIMS to obtain a reliable depositional age for the ash bed. A total of seven grains were selected to represent the age distribution of the sample, with thirteen grains being discarded for being too old (>~150 Ma) and considered either inherited grains or prolonged magmatic residence or magmatic recycling. A total of four younger grains (z10, z20, z38, z44) that overlapped were considered for the final weighted mean age of the ash bed of 147.112 ± 0.078/0/088/0.18 Ma, MSWD = 0.81. Radiogenic Pb ranged from 1 to 6 pg (TS.1).

5.6. Ash bed MZT-81

Ash bed MZT-81 is located in stratigraphic height ca. 22.5 m in the Mazatepec section. Zircon yield high ca. > 100 grains. Zircons crystals ranged from 40-80 μm in size. Grains were mainly prismatic with aspect ratio of 1:8. Radiogenic Pb ranged from 1 to 6 pg (TS.1), notably lower than other the other samples. A total of eight grains were selected to represent the age distribution of the sample, with 4 grains being discarded for being too old (>~150 Ma) and considered either inherited grains or prolonged magmatic residence, or magmatic recycling. A total of four younger grains (z4, z6, z8, z10) that overlapped were considered for the final weighted mean age of the ash bed of 140.512 ± 0.031/0/048/0.16 Ma, MSWD = 0.56

6. Age-depth modelling - Bchron Code

```
library(Bchron)

mydata2 = read.table(file='\\Users\\fortesd0\\Documents\\R\\win-
library\\3.4\\Bchron\\OregonPliens.txt', header=TRUE)
GlenOut = Bchronology(ages=mydata2$ages,
                    ageSds=mydata2$ageSds,
                    calCurves=mydata2$calCurves,
                    positions=mydata2$position,
                    positionThicknesses=mydata2$thickness,
                    ids=mydata2$id
                    predictPositions=seq(0,8400,by=10),iterations = 10000)
plot(GlenOut,main="NeuquenBchron",xlab='Age (Ma)',ylab='Depth (cm)',las=1)
summary(GlenOut)
summary(GlenOut, type='convergence')
summary(GlenOut, type='outliers')


Output <- cbind(apply(GlenOut$thetaPredict, 2, quantile,  probs = c(.0025)),
     apply(GlenOut$thetaPredict, 2, quantile,  probs = c(.5)),
     apply(GlenOut$thetaPredict, 2, quantile,  probs = c(.975)))
write.csv(Output, file = 'whatever.csv', quote=FALSE, row.names = FALSE)


acc_rate = summary(GlenOut, type = 'acc_rate')
```

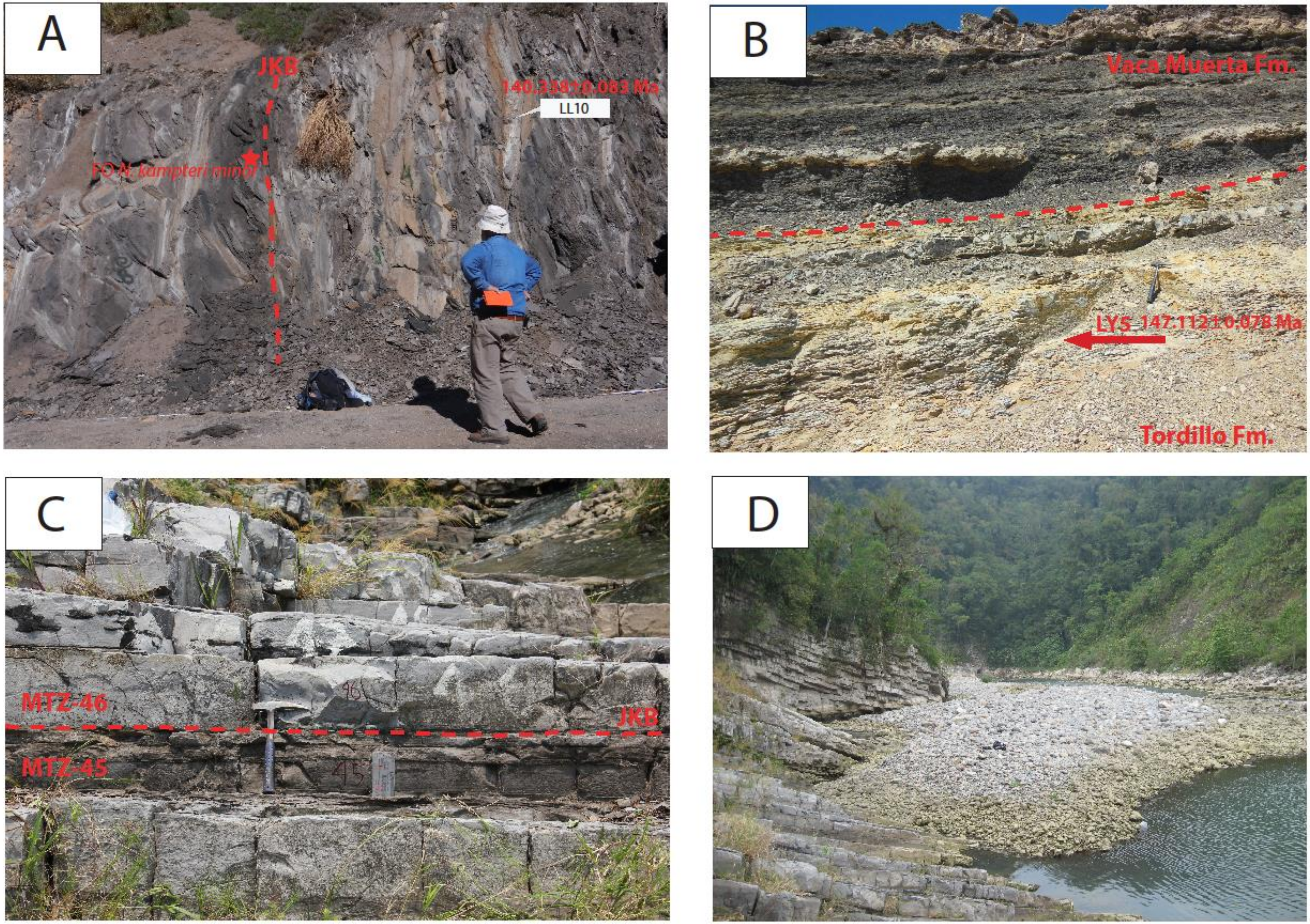
```
plot(acc_rate[,'age_grid'], acc_rate[,'50%'], type='l', ylab = 'cm per year', xlab = 'Age (k cal
years BP)', ylim = range(acc_rate[,-1]))
lines(acc_rate[,'age_grid'], acc_rate[,'2.5%'], lty='dotted')
lines(acc_rate[,'age_grid'], acc_rate[,'97.5%'], lty='dotted')
sed_rate = summary(GlenOut, type = 'sed_rate', useExisting = FALSE)
plot(sed_rate[,'position_grid'], sed_rate[,'50%'], type='l', ylab = 'Years per cm', xlab = 'Depth
(cm)', ylim = range(sed_rate[,-1]))
lines(sed_rate[,'position_grid'], sed_rate[,'2.5%'], lty='dotted')
lines(sed_rate[,'position_grid'], sed_rate[,'97.5%'], lty='dotted')

write.csv(sed_rate, file = 'NeuquenBchron_sed_rates.csv', quote=FALSE, row.names =
FALSE)
write.csv(GlenOut, file = 'NeuquenBchron_sed_rates.csv', quote=FALSE, row.names =
FALSE)
```

7. Supplementary Figure 1 – Fig. S1

	Conusphaera mexicana	*Watznaueria barnesiae*	*Watznaueria fossacincta*	*Cyclagelosphaera margerelii*	*Watznaueria britannica*	*Cyclagelosphaera deflandrei*	*Watznaueria communis*	*Hexalithus noeliae*	*Zeugrhabdotus embergeri*	*Polycostella senaria*	*Cocosfera*	*Helenea chiastia*	*Watznaueria manivitiae*	*Nannoconus kamptneri minor*	*Nanoconus sp.*	*Retecapsa octofenestrata*	*Retecapsa surirella*	*Nannoconus steinmannii*	*Nannoconus globulus*	*Zeugrhabdotus erectus*
MZT-92		X		X														X		
MZT-87		X	X	X	X								X	X	X		X	**X**	X	X
MZT-84		X														X				
MZT-69		X		X						X										
MZT-68				X	X				X	X					X					
MZT-65		X	X					X		X		X	X	**X**						
MZT-58		X	X	X					X		X									
MZT-55		X		X						X										
MZT-51		X	X																	
MZT-47		X		X	X	X														
MZT-45	**X**	X	X						X											
MZT-30	X	X		X	X	X	X	X												
MZT-25		X																		
MZT-16		X	X																	
MZT-15	X	X	X	X																
MZT-12	X	X	X	X																
MZT-6	**X**	X	X	X	X															

Supplementary Figure 1. Stratigraphic distribution of calcareous nannofossils of Mazatepec section, Mexico.

8. Supplementary Figure 2 – Fig. S2 - Field Figures


A
JKB
140.338±0.083 Ma
LL10
FO N. kampteri minor
B
Vaca Muerta Fm.
LYS 147.112±0.078 Ma
Tordillo Fm.
C
MTZ-46
JKB
MTZ-45
D

Supplementary Figure 2 – Field photos. A) Field figure from the Las Loicas section. Location of ash bed LL10 and the location of the JKB in the section. B) Field figure of the lower part of the La Yesera section where the contact between the Vaca Muerta and the Tordillo formations, and the location of ash bed LY5. C) location of the JKB in the Mazatepec section in Mexico, see bed MTZ-46 and MTY-45 (see Lopey-Matinez et al. 2013) D) Outcrop view of the Mazatepec section.

9. Data tables

9.1. U-Pb geochronology data table TS.1

	Dates (Ma)				Composition						
Fraction	206Pb/ 238U <Th> **a**	±2σ abs	207Pb/ 235U **b**	±2σ abs	Th/ U **c**	Pb* (pg) **d**	Pbc (pg) **e**	206Pb/ 238U **f**	±2σ %	207Pb/ 235U **f**	±2σ %
LL3											
z1	139.43	0.11	138.0	1.1	0.97	10.4	0.75	0.021852	0.080	0.1456	0.88
z2	139.36	0.11	138.75	0.76	0.88	9.76	0.43	0.021842	0.079	0.14642	0.59
z11	139.47	0.12	138.7	1.3	0.80	3.37	0.27	0.021858	0.088	0.1464	0.98
z21	139.45	0.11	139.8	1.1	0.97	7.21	0.48	0.021856	0.082	0.1476	0.84
z22	139.44	0.12	139.2	1.2	0.79	4.10	0.22	0.021853	0.086	0.1469	0.93
z23	**139.16**	**0.14**	**136.4**	**1.5**	**0.90**	**4.68**	**0.42**	**0.021809**	**0.10**	**0.1437**	**1.2**
z25	**139.244**	**0.067**	**136.71**	**0.70**	**0.75**	**5.08**	**0.21**	**0.021822**	**0.048**	**0.14413**	**0.55**
z26	**139.27**	**0.10**	**139.45**	**0.77**	**0.79**	**7.78**	**0.37**	**0.021826**	**0.075**	**0.14721**	**0.59**
z32	**139.23**	**0.13**	**139.2**	**1.1**	**0.90**	**6.41**	**0.37**	**0.021820**	**0.093**	**0.1469**	**0.88**
LL9											
z2	**139.98**	**0.11**	**144.6**	**5.2**	**0.73**	**8.68**	**1.02**	**0.021938**	**0.077**	**0.1531**	**3.9**
z5	140.191	0.063	141.2	1.2	0.47	14.0	0.40	0.0219707	0.045	0.1492	0.90
z7	140.19	0.14	141.7	2.3	0.53	8.56	0.46	0.021971	0.097	0.1498	1.8
z12	**139.99**	**0.16**	**150**	**11**	**0.37**	**1.94**	**0.55**	**0.021938**	**0.11**	**0.159**	**8.2**
z33	140.456	0.071	141.3	1.0	0.47	10.3	0.24	0.022013	0.051	0.1493	0.78
z34	140.814	0.078	141.5	1.7	0.78	5.62	0.21	0.022071	0.056	0.1495	1.3
z51	**139.87**	**0.22**	**153**	**15**	**0.37**	**2.54**	**0.96**	**0.021919**	**0.16**	**0.162**	**11**
z52	140.67	0.11	143.6	5.0	0.81	3.13	0.34	0.022048	0.078	0.1519	3.7
z53	140.18	0.14	140.7	2.5	0.65	5.33	0.29	0.021970	0.10	0.1486	1.9
z54	140.35	0.11	142.4	3.3	0.71	3.72	0.27	0.021997	0.080	0.1506	2.5
z55	**139.945**	**0.097**	**141.5**	**2.5**	**0.50**	**6.57**	**0.39**	**0.021932**	**0.070**	**0.1495**	**1.9**
LL10											
z11	140.51	0.13	140.1	1.4	0.82	4.08	0.37	0.022023	0.096	0.1479	1.1
z12	141.20	0.35	140.2	3.8	0.93	1.01	0.24	0.022134	0.25	0.1481	2.9
z13	140.61	0.26	139.6	3.0	0.93	2.57	0.49	0.022039	0.18	0.1474	2.3

	z14	140.68	0.23	139.5	2.6	1.10	2.78	0.45	0.022052	0.16	0.1473	2.0
	z41	**140.37**	**0.21**	**138.0**	**2.2**	**0.84**	**2.17**	**0.28**	**0.022001**	**0.15**	**0.1456**	**1.7**
	z42	**140.29**	**0.12**	**140.3**	**1.2**	**1.14**	**6.31**	**0.45**	**0.021990**	**0.085**	**0.1481**	**0.94**
	z44	**140.32**	**0.16**	**139.8**	**1.2**	**1.02**	**4.86**	**0.32**	**0.021994**	**0.12**	**0.1476**	**0.96**
	z45	**140.55**	**0.27**	**138.3**	**2.8**	**0.87**	**1.42**	**0.19**	**0.022029**	**0.19**	**0.1459**	**2.2**
MZT-81												
	z4	**140.478**	**0.079**	**140.57**	**0.74**	**0.42**	**10.4**	**0.54**	**0.022016**	**0.057**	**0.14848**	**0.57**
	z6	**140.504**	**0.083**	**140.41**	**0.85**	**0.53**	**9.91**	**0.60**	**0.022021**	**0.060**	**0.14830**	**0.65**
	z7	141.03	0.12	141.7	1.3	0.43	3.31	0.30	0.022103	0.088	0.1498	0.97
	z8	**140.518**	**0.047**	**140.39**	**0.25**	**0.53**	**11.1**	**0.17**	**0.0220228**	**0.033**	**0.14828**	**0.19**
	z10	**140.528**	**0.061**	**140.22**	**0.45**	**0.53**	**10.9**	**0.33**	**0.0220243**	**0.044**	**0.14809**	**0.34**
	z11	140.85	0.10	140.4	1.1	0.48	6.49	0.51	0.022075	0.075	0.1483	0.83
	z12	141.22	0.29	141.4	3.4	0.47	2.47	0.60	0.022134	0.21	0.1494	2.6
	z15	141.02	0.14	140.1	1.4	0.53	5.98	0.58	0.022102	0.099	0.1479	1.1
LL13												
	z9	**142.106**	**0.085**	**142.49**	**0.76**	**0.64**	**8.39**	**0.41**	**0.022275**	**0.060**	**0.15066**	**0.57**
	z33	**141.93**	**0.11**	**142.2**	**1.2**	**0.76**	**7.49**	**0.51**	**0.022247**	**0.076**	**0.1503**	**0.88**
	z34	**142.05**	**0.12**	**142.6**	**1.1**	**0.93**	**7.09**	**0.39**	**0.022267**	**0.081**	**0.1508**	**0.80**
LY5												
	z10	**147.8**	**1.1**	**144**	**13**	**0.71**	**0.665**	**0.56**	**0.02319**	**0.77**	**0.152**	**9.7**
	z19	148.07	0.42	146.8	4.9	0.51	1.49	0.51	0.023221	0.29	0.1556	3.6
	z20	**147.12**	**0.26**	**144.5**	**3.0**	**0.77**	**3.92**	**0.75**	**0.023071**	**0.18**	**0.1529**	**2.2**
	z22	148.01	0.36	147.0	3.8	0.39	1.34	0.34	0.023211	0.25	0.1558	2.8
	z38	**146.99**	**0.29**	**145.3**	**3.3**	**0.74**	**2.07**	**0.45**	**0.023050**	**0.20**	**0.1539**	**2.5**
	z44	**147.118**	**0.086**	**146.49**	**0.82**	**0.75**	**3.37**	**0.15**	**0.023071**	**0.058**	**0.15520**	**0.60**
	z67	147.740	0.093	147.09	0.96	0.49	6.20	0.41	0.023168	0.064	0.1559	0.70

a) Corrected for initial Th/U disequilibrium using the radiogenic 208Pb and Th/U (magma)= 3.5
b) Isotopic dates calculated using 238= 1.55125 E-10 (Jaffey et al. 1971) and 235=9.8485E-10 (Jaffey et al. 1971)
c) Th contents calculated from radiogenic 208Pb and 239^{Th}-corrected 206Pb/207Pb date of the sample, assuming concordance between U-Pb and Th-Pb systems
d) Total mass of radiogenic Pb
e) Total mass of common Pb
f) Measured ratios correct for fractionation, tracer and blank.

9.2. Age-depth model data table TS.2

Stratigraphic Height (m)	Age (Ma)	Std Err (2 S.D) (Ma)
54	139.24	0.05
53	139.30	0.15
52	139.35	0.20
51	139.41	0.22
50	139.46	0.23
49	139.51	0.23
48	139.57	0.24
47	139.62	0.23
46	139.68	0.24
45	139.73	0.23
44	139.79	0.22
43	139.84	0.21
42	139.90	0.17
41	139.96	0.07
40	140.00	0.10
39	140.03	0.12
38	140.07	0.12
37	140.11	0.13
36	140.15	0.13
35	140.18	0.13
34	140.22	0.13
33	140.27	0.12
32	140.31	0.11
31	140.35	0.08
30	140.42	0.23
29	140.48	0.30
28	140.54	0.37

Stratigraphic Height (m)	Age (Ma)	Std Err (2 S.D) (Ma)
15	141.31	0.56
14	141.37	0.56
13	141.43	0.52
12	141.50	0.51
11	141.56	0.50
10	141.62	0.49
9	141.68	0.47
8	141.74	0.44
7	141.80	0.40
6	141.86	0.36
5	141.92	0.33
4	141.97	0.23
3	142.04	0.06
2	142.10	0.32
1	142.15	0.42

27	140.60	0.41
26	140.66	0.43
25	140.72	0.46
24	140.78	0.50
23	140.84	0.50
22	140.90	0.52
21	140.96	0.53
20	141.02	0.55
19	141.08	0.56
18	141.14	0.55
17	141.20	0.56
16	141.26	0.56

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