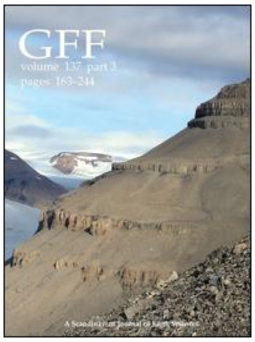

GFF

ISSN: 1103-5897 (Print) 2000-0863 (Online) Journal homepage: https://www.tandfonline.com/loi/sgff20

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To cite this article: Sandra L. Kamo & Alberto C. Riccardi (2009) A new U–Pb zircon age for an ash layer at the Bathonian–Callovian boundary, Argentina, GFF, 131:1-2, 177-182, DOI: 10.1080/11035890902867605

To link to this article: https://doi.org/10.1080/11035890902867605

GFF volume 131 (2009), pp. 177–182. *Article*

A new U–Pb zircon age for an ash layer at the Bathonian–Callovian boundary, Argentina

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Kamo, S.L. & Riccardi, A.C., 2009: A new U–Pb zircon age for an ash layer at the Bathonian–Callovian boundary, Argentina. *GFF*, Vol. 131 (Pt. 1–2, June), pp. 177–182. Stockholm. ISSN 1103-5897.

Abstract: A U–Pb zircon age of 164.64 ± 0.2 Ma (95% confidence level) is reported for an ash bed, at the Bathonian–Callovian (late Middle Jurassic) boundary, determined by isotope dilution thermal ionisation mass spectrometry from individual, chemically abraded grains. The volcanic ash layer occurs within the Chacay Melehue Formation, Chacay Melehue section, Neuquén Province, central west Argentina, above the last record of ammonites of the regional *Lilloettia steinmanni* Standard Zone, and, stratigraphically, where the first of those of the regional *Eurycephalites vergarensis* Standard Zone appears, generally referred to as the uppermost Bathonian and the lowermost Callovian, respectively. This ash layer represents the only known datable horizon worldwide that is directly related to a well-documented ammonite faunal succession at this boundary. The U–Pb zircon age is older than one previously reported for the same bed and closer to an estimation of 164.7 ± 4 Ma for the boundary based on the scaling durations of ammonite zones to their subzones in the sub-boreal standard zonation. The new age agrees better with the age model for the Oxfordian through Bathonian M-sequence magnetic anomalies in the Pacific and contributes to the radioisotopic age calibration of the Jurassic time scale.

Keywords: U-Pb geochronology, ammonites, biostratigraphy, Bathonian, Callovian, zircon.

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Manuscript received 3 November 2008. Revised manuscript accepted 1 February 2009.

Introduction

Chronostratigraphy of the upper Bathonian and lower Callovian has been based on the well-established ammonite zonal standard of northwestern Europe (see Callomon & Dietl 2000). Additional standards have been established for other regions of the globe based on occurrences of rich, diverse ammonoid faunas, although biostratigraphic correlation has been problematic due to high faunal endemism. Notwithstanding these difficulties, at least one correlation has been presented (see Westermann 1996).

One of the most important of these standards corresponds to the eastern Pacific area, extending from the southern Andes to southern Mexico (Riccardi et al. 1989; Sandoval et al. 1990; Hillebrandt et al. 1992). A key section of this area is located in Chacay Melehue, Neuquén Province, central-west Argentina (Fig. 1), where a rich ammonoid fauna has been studied and used to propose the main zones recognised along the eastern Pacific (Riccardi et al. 1989; Riccardi & Westermann 1991a, 1991b).

The identification of volcaniclastic rocks interbedded with an ammonite-bearing marine sequence that extends from the Lower to Upper Jurassic system (Pliensbachian to Oxfordian) prompted the initiation of a project in the 1980s with the goal of establishing a detailed Early and Middle Jurassic chronostratigraphy calibrated using radioisotopic ages. As a preliminary result, two ages were obtained for two levels located at the Bathonian–Callovian boundary and lower Callovian (see Odin et al. 1992).

Radioisotopic ages for the Jurassic, including the Bathonian–Callovian stages, have been discussed in detail by Pálfy et al. (2000a, 2000b) and presented in an updated summary by Ogg (2004). From these summaries, it is clear that the Bathonian–Callovian boundary is poorly constrained by absolute ages and the possibility of determining the age of a biochronologically defined boundary by direct isotopic dating at or in the immediate vicinity of the boundary has been considered as rarely occurring in the Jurassic (Pálfy et al. 2000b). Thus, the only available U–Pb age of 160.5 ± 0.3 Ma from the Chacay Melehue section (Odin et al. 1992) was used to assign a chronogram age of 160.4 + 1.1/− 0.5 Ma to the base of the Callovian (Pálfy et al. 2000b).

As this age was difficult to reconcile with the age model for the Oxfordian through Callovian M-sequence magnetic anomalies in the Pacific, an age of 164.7 ± 4 Ma was assigned to the base of the Callovian in the 2004 geologic time scale (Ogg 2004), by scaling according to equal subzones from computed ages for base Bajocian and Oxfordian.

To clarify the age of this important boundary and to aid in refining the Jurassic time scale, it became apparent that the original ash layer dated by Odin et al. (1992) should be

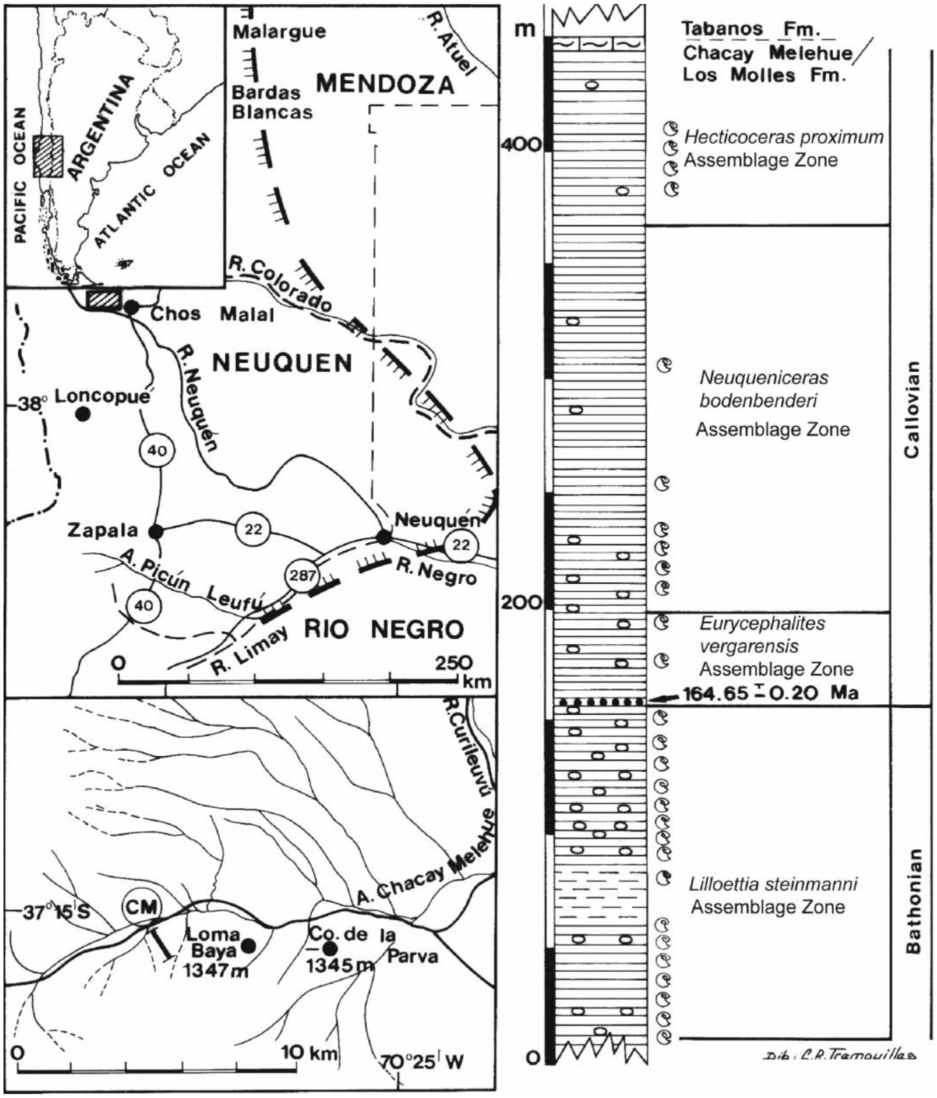

Fig. 1. Location of the Chacay Melehue section in western Argentina and simplified stratigraphic log of the studied Bathonian–Callovian succession with recognised biostratigraphic units.

redated with modern isotope dilution thermal ionisation mass spectrometry (ID-TIMS) methods, especially given that this is the only known volcanic layer directly associated with the Bathonian–Callovian boundary.

Regional geology

The Neuquén Basin is an approximately N–S-oriented back-arc basin comprising a ~7 km thick Mesozoic–Cenozoic sedimentary succession that formed during the extension of Pangea in the early Mesozoic. Sedimentation began in the Triassic with volcanic and coarse-grained continental deposits, and from the Late Triassic onwards a narrow marine corridor settled in the northern area of the basin. This sea expanded towards the rest of the basin in the Pliensbachian–Toarcian, producing a westward-prograding clastic system and deep-basin turbidites, periodically interrupted by volcanic events. In the Chacay Melehue area (Fig. 1), where the structure and succession of the whole area has been extensively studied (cf. Llambias et al. 2007 for a recent account), the Mesozoic is excellently represented.

The Jurassic beds exposed at Chacay Melehue form the eastern slope of the N–S-oriented Cordillera del Viento. The Cordillera del Viento Formation comprises Triassic porphyritic rocks of andesitic to rhyolitic composition and is unconformably overlain by about 200 m of bedded flows of basalts, andesitic basalts and rhyolites of the Milla Michicó Formation. These Triassic units are unconformably overlain by about 1300–1400 m of marine sediments, ranging in age from Pliensbachian to Oxfordian. The Lower Jurassic comprises about 350 m of Pliensbachian tuffaceous sandstones and siltstones in the lower part interbedded with basaltic rocks, i.e. the La Primavera Formation. The Toarcian–Aalenian consists of about 150 m of bituminous black shales and argillites. Above 30–40 m of mudstone, arkose and conglomerates, with argillites in the upper part, and about 600–700 m of black shales, with turbidites near the base and, in the middle part, with interbedded white tuffaceous sandstones, containing a rich Upper Bajocian–Lower Callovian ammonoid fauna (Fig. 1). This Toarcian–Callovian sequence is usually ascribed to the Los Molles Formation originally described by Weaver (1931) for the Zapala area, although the Callovian shales have also been named the Chacay Melehue Formation (cf. Riccardi & Damborenea 1993).

This sequence is successively overlain by 4–15 m of white gypsum, the Tabanos Formation; about 150 m of dark grey shales and mudstones, in the lower part with yellowish grey, fine- to coarse-grained sandstones and in the upper part with interbedded siltstones and limestones, and 40–60 m of dark grey micritic limestones and calcareous breccias, i.e. the Lotena, La Manga and Auquilco formations or their equivalents. The whole sequence is overlain by continental red pelites, sandstones and conglomerates of the Tordillo Formation (Kimmeridgian), on top of which rests the marine Mendoza Group (Tithonian–Barremian).

Biostratigraphy

Ammonites present in the Jurassic of central-west Argentina have been extensively studied, especially in the last 35 years and, as a result, a detailed biostratigraphic framework has been developed and correlated with the European biochronologic standard (Riccardi et al. 2000; Riccardi 2008).

In the Chacay Melehue area, the Bathonian–Callovian stratigraphy and/or ammonoid systematics have been studied by Dellape et al. (1979), Westermann (1981), Riccardi (1984a, 1984b, 1985), Westermann & Riccardi (1985), Riccardi et al. 1989, Riccardi & Westermann (1991a, 1991b) and Parent (1997, 1998). The Bathonian–Callovian fauna of Chacay Melehue is mainly endemic, although it has some taxa in common with those occurring in other well-studied sections from northern Chile and southern Mexico (Riccardi et al. 1989; Sandoval et al. 1990; Riccardi & Westermann 1991a, 1991b; Hillebrandt et al. 1992; Westermann 1996). Due to extreme faunistic provincialism, an independent set of Standard Zones was defined for the whole area, which includes, from below, the *Lilloettia steinmanni*, *Eurycephalites vergarensis* and *Neuqueniceras bodenbenderi* Zones. The Bathonian–Callovian boundary was placed between the *L. steinmanni* and *E. vergarensis* Zones (Riccardi et al. 1989; Hillebrandt et al. 1992).

In Chacay Melehue, the late Bathonian *L. steinmanni* Zone is characterised, besides the dominant East Pacific Eurycephalitinae and *Neuqueniceras* s. str., by the cosmopolitan *Choffatia* genus. The ammonoid fauna superjacent to the *L. steinmanni* Zone and included in the *E. vergarensis* Zone is characterised by endemic species of Eurycephalitinae, together with cosmopolitan and relatively long-ranging perisphinctids and oppelids. Thus, a precise age cannot be established. The overlying *N. bodenbenderi* Zone is characterised by the index species and some East Pacific Eurycephalitinae. Only the overlying *Hecticoceras proximum* Zone is characterised by an association of Tethyan Oppeliidae indicating the latest early Callovian (Riccardi et al. 1989).

In northern Chile, however, *L. steinmanni* (Spath) and *Choffatia jupiter* (Steinmann) occur with, or just above, *Hecticoceras (Prohecticoeceras) retrocostatum* (Grossouvre), which in Europe and northwest Africa characterises the upper *H. retrocostatum* Zone, while *Epistrenoceras histricoides* (Rollier), *Hecticoceras (Prohecticoceras) blanazanese* Elmi, *Hemigarantiana* sp. and *Eohecticoceras* sp. indicate the early *H. retrocostatum* Zone. The overlying *E. vergarensis* and *N. bodenbenderi* Zones are again, as in Chacay Melehue, characterised by endemic ammonites (Riccardi et al. 1989; Hillebrandt et al. 1992; Gröschke & Hillebrandt 1994; Westermann 1996).

In Mexico, again, the *L. steinmanni* Zone is characterised by *L. steinmanni* and *C. jupiter*, as in Chacay Melehue, in association with *Eohecticoceras* sp., and as in northern Chile overlying beds with *E. histricoides* and *P. blanazense*. Thus, the *L. steinmanni* Zone has been correlated with the *H. retrocostatum*–(?) *Clydoniceras discus* Zones. Again, the *E. vergarensis* Zone is mainly characterised by the index species and does not include any age diagnostic ammonoid. However, here, the overlying *N. bodenbenderi* Zone contains Mediterranean species, such as *Oxycerites (Paroecotraustes) cf. bronni* (Zeiss), *Rehmannia cf. grossouvrei* (Petitclerc), *Parapatoceras distans* (Baugier and Sauze), *Paracuariceras cf. incisum* Schindewolf and *Jeanneticeras malbosi* Elmi, which indicate top *Macrocephalites herveyi* to basal *M. gracilis* Zone (Sandoval et al. 1990; Hillebrandt et al. 1992; Westermann 1996).

Thus, the Late Bathonian *L. steinmanni* Zone, characterised, besides the dominant East Pacific Eurycephalitinae, *Neuqueniceras* s. str. and *C. jupiter*, by cosmopolitan species, has been dated as *H. retrocostatum* (?) *C. discus* Zone. For the superjacent *E. vergarensis* Zone, characterised by endemic species of Eurycephalitinae, together with cosmopolitan and relatively long-ranging perisphinctids and oppelids, a precise age cannot be established. However, the overlying *N. bodenbenderi* Zone, which in southern Mexico also contains Mediterranean species, has been dated as top *M. herveyi* to basal *M. gracilis* Zone. Therefore, the stratigraphic position of the *E. vergarensis* Zone, between the better dated *L. steinmanni* and *N. bodenbenderi* Zones, indicates earliest Callovian (≈*M. herveyi* Zone), although it could cross the stage boundary.

Analytical methods

The U–Pb dating method used in this study was ID-TIMS. Zircon grains were thermally annealed and leached to ensure removal of radiation-damaged zones that may have undergone Pb loss due to alteration, as described by Mattinson (2005), and applied and modified by Mundil et al. (2004). Mattinson (2005) called this pre-treatment application for chemical abrasion thermal ionisation mass spectrometry "CA-TIMS". Grains were placed in a muffle furnace at ~1000°C for 60 h to anneal damaged lattice sites, followed by leaching in concentrated hydrofluoric acid (HF) in Teflon dissolution vessels at 200°C for 6 h. Grain weights were estimated using photomicrographs: uncertainties in weight affect only U and Pb concentrations and not age information. The grains were washed prior to dissolution and a ^{205}Pb–^{233}U–^{235}U spike (EARTHTIME tracer solution ET535: see www.earth-time.org) was added to the Teflon dissolution capsules during sample loading. Zircon was dissolved using ~0.1 ml HF and ~0.02 ml of 7 N nitric acid (HNO_3) in Teflon dissolution bombs at 200°C after the method outlined by Krogh (1973) for 5 days, and then dried to a precipitate, followed by addition of ~0.08 ml of 3.1 N hydrochloric acid overnight. Anion-exchange column separation was performed to isolate U and Pb, which were then dried in ~10 μl of 0.05 N phosphoric acid (H_3PO_4) and loaded directly onto out-gassed rhenium filaments with silica gel (Gerstenberger & Haase 1997). Pb and U were analysed with a VG354 mass spectrometer using a Daly collector in pulse-counting mode. All common Pb was assigned to the isotopic composition of the procedural Pb blank (and it is noted that correcting for initial Pb using Stacey & Kramers' (1975) Pb evolution model and assuming a blank of 0.5 pg has a negligible effect on the ^{206}Pb–^{238}U age). Dead time of the measuring system for Pb is 22.8 and 20.8 ns for U. The mass discrimination correction for

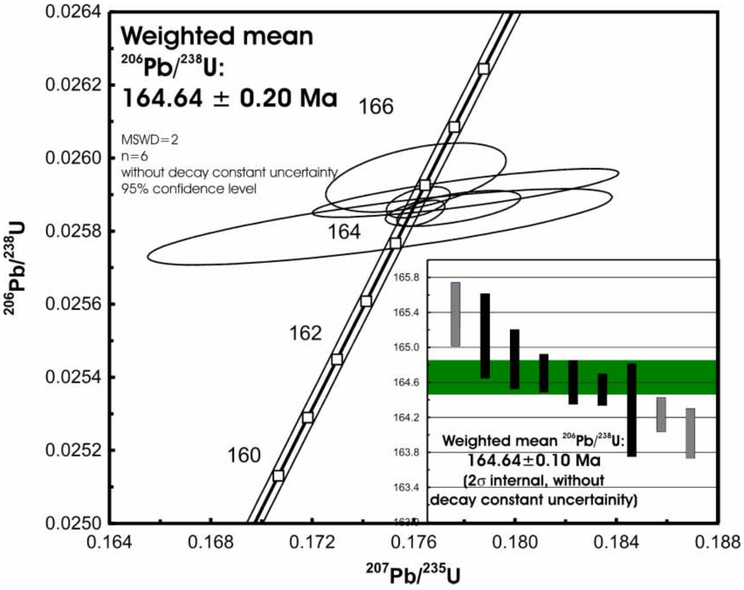

Fig. 2. U–Pb concordia diagram showing data for individual, chemically abraded zircon crystals from an ash bed at the Bathonian-Callovian boundary, Chacay-Melehue Formation, Neuquén Province, Argentina. Error ellipses shown at the 2σ level.

the Daly detector is constant at 0.05% per atomic mass unit. Daly characteristics were monitored using the SRM982 Pb standard. Thermal mass discrimination correction for Pb is 0.10% per atomic mass unit. A correction for ^{230}Th disequilibrium was applied, assuming Th/U of 4.2 in the magma from which the zircon crystallised. Decay constants are those of Jaffey et al. (1971). All age errors quoted in the text and Table 1 and error ellipses in the concordia diagram, are given at the 95% confidence level (Fig. 2). Plotting and age calculations are from Ludwig (2003).

Results

A weighted mean ^{206}Pb–^{238}U age of 164.64 ± 0.20/0.27 Ma (95% confidence level, decay constant error not included/decay constant error included) was determined from six CA-TIMS zircon analyses (MSWD or mean square of the weighted deviates = 2). In an effort to reduce or eliminate potential bias from the inclusion of an analysis containing a trace of inherited material, or from minor Pb loss (despite the annealing and leaching pre-treatment), the oldest and two youngest data were excluded from the mean age. If the TuffZirc algorithm of Ludwig (2003) is used for the total data set, the same six data are extracted and give an age of 164.65 + 0.48/− 0.37 Ma. If all nine are included in the mean, the age is within error at 164.54 ± 0.27 Ma (95% confidence level; excludes decay constant error) with a MSWD of 7.3. We conclude that the 164.64 ± 0.2 Ma age interpretation is the most robust, and that by using the ET535 spike solution, the age is amenable to future direct inter-laboratory comparisons and interpolations with similarly derived U–Pb zircon ages in the Jurassic time scale.

Discussion

The only other radioisotopic ages for the Bathonian–Callovian boundary were obtained from two ash layers, from two different sections in western Canada. One of them, sample 13 dated at 158.4 ± 0.8 Ma (Pálfy et al. 2000a), is poorly constrained biostratigraphically, because the only ammonoid taxon observed at this locality is the genus *Iniskinites*, which was first considered to be restricted to the Bathonian, but is now known to range into the Callovian (Westermann et al. 2002). The other, sample 14 dated at 162.6 + 2.9 − 7 Ma (Pálfy et al. 2000a), is from a tuff located c. 5 m above an assemblage with *Cadoceras cf. moffiti* (Imlay) and c. 4 m below an assemblage with *Kepplerites loganianus* (Whiteaves). These two ammonoids belong to the Boreal Realm; the first has been ascribed to the upper Bathonian whilst the second is considered to be early Callovian (Callomon 1984). The identifications, however, are in open nomenclature, and the whole fauna does not seem to have been studied in detail. Nevertheless, the ash layer can be considered to lie close to the Bathonian–Callovian boundary and the age obtained, and its error, falls within our age obtained for Chacay Melehue (Table 1).

Conclusions

A volcanic ash layer at the Bathonian–Callovian (late Middle Jurassic) boundary within the Chacay Melehue Formation, Chacay Melehue section, Neuquén Province, Argentina has yielded a U–Pb zircon age of 164.64 ± 0.2 Ma (95% confidence level). The bed rests above the regional *L. steinmanni* Standard Zone, where ammonites of the *E. vergarensis* Standard Zone occur. This ash layer is the only known datable horizon directly related to a well-documented ammonite faunal succession at the Bathonian–Callovian boundary.

The new age is older than the U–Pb zircon age of 160.5 ± 0.3 Ma for the same bed (Odin et al. 1992). In fact, it is closer to an estimation of 164.7 ± 4 Ma based on scaling durations of ammonite zones to their subzones in the sub-boreal standard zonation for the Bathonian–Callovian boundary, and is more in agreement with the age model for the Oxfordian through Bathonian M-sequence magnetic anomalies in the Pacific (Ogg 2004).

The new age obviates the need for interpolation and chronogram estimation for this stage boundary and contributes to the radioisotopic age calibration of the Jurassic time scale.

Table 1. U–Pb isotopic data for single zircons from an ash layer at the Bathonian–Callovian boundary, Chacay Melehue, Neuquén Province, Argentina.

No.	Weight (μg)	Pb_{tot} (pg)	Pb_{com} (pg)	U (ppm)	Th/U	$^{206}Pb/^{204}Pb$	$^{207}Pb/^{206}Pb$	2σ	$^{206}Pb/^{238}U$	2σ	$^{207}Pb/^{235}U$	2σ	Rho concordia	$^{206}Pb/^{238}U$ Age (Ma)	2σ	$^{207}Pb/^{235}U$ Age (Ma)	2σ
1	4.6	13.8	1.3	104	0.74	626	0.04952	0.00048	0.025986	0.000058	0.1774	0.0019	0.487	165.38	0.36	165.8	1.6
2	3.0	9.0	0.6	105	0.74	825	0.04922	0.00075	0.025947	0.000077	0.1761	0.0029	0.491	165.13	0.48	164.7	2.5
3	2.0	24.3	2.9	401	0.97	473	0.04985	0.00130	0.025905	0.000054	0.1781	0.0049	0.824	164.87	0.34	166.4	4.3
4	4.3	15.7	0.7	120	1.01	1290	0.04938	0.00026	0.025879	0.000035	0.1762	0.0010	0.501	164.70	0.22	164.8	0.9
5	4.8	14.3	0.8	103	0.79	1082	0.04980	0.00057	0.025862	0.000040	0.1776	0.0022	0.600	164.60	0.25	166.0	1.9
6	2.0	19.0	0.7	320	0.91	1633	0.04946	0.00019	0.025849	0.000029	0.1763	0.0008	0.544	164.52	0.18	164.9	0.7
7	3.1	8.5	1.6	97	0.75	318	0.04908	0.00198	0.025812	0.000085	0.1747	0.0075	0.790	164.28	0.53	163.5	6.5
8	3.9	18.8	1.2	169	0.75	917	0.04955	0.00033	0.025803	0.000031	0.1763	0.0013	0.544	164.23	0.20	164.8	1.1
9	4.3	15.7	0.9	124	0.88	984	0.04961	0.00063	0.025769	0.000045	0.1763	0.0024	0.576	164.01	0.28	164.8	2.1

Notes: Pb_{tot} is the total amount of Pb excluding blank. Pb_{com} is the common Pb assuming the isotopic composition of laboratory blank: 206/204-18.221; 207/204-15.612; 208/204-39.360 (2σ errors of 2%). 206/204 corrected for fractionation and common Pb in the spike; Pb/U ratios also corrected for blank. Correction for ^{230}Th disequilibrium in 206/238 and 207/206 assuming Th/U of 4.2 in the magma. Th/U calculated from radiogenic $^{208}Pb/^{206}Pb$ ratio and $^{207}Pb/^{206}Pb$ age assuming concordance. Disc is percent discordance for the given $^{207}Pb/^{206}Pb$ age. Rho concordia is correlation coefficients of X–Y errors on the concordia plot.

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