Direct U-Pb dating of Cretaceous and Paleocene dinosaur bones, San Juan Basin, New Mexico

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ABSTRACT

Vertebrate fossils have been important for relative dating of terrestrial rocks for decades, but direct dating of these fossils has heretofore been unsuccessful. In this study we employ recent advances in laser ablation in situ U-Pb dating techniques to directly date two dinosaur fossils from the San Juan Basin of northwestern New Mexico and southwestern Colorado, United States. A Cretaceous dinosaur bone collected from just below the Cretaceous-Paleogene interface yielded a U-Pb date of 73.6 ± 0.9 Ma, in excellent agreement with a previously determined ^{40}Ar/^{39}Ar date of 73.04 ± 0.25 Ma for an ash bed near this site. The second dinosaur bone sample from Paleocene strata just above the Cretaceous-Paleogene interface yielded a Paleocene U-Pb date of 64.8 ± 0.9 Ma, consistent with palynologic, paleomagnetic, and fossil-mammal biochronologic data. This first successful direct dating of fossil vertebrate bone provides a new methodology with the potential to directly obtain accurate dates for any vertebrate fossil.

INTRODUCTION

Vertebrate fossils have always intrigued humans, and the evolution of the animals whose remains these fossils represent has provided valuable clues about the biochronology of the rock strata in which they are preserved. However, because of the rarity and often endemic nature of these fossils, mostly found in continental rocks, only a spotty knowledge of these animals' lifestyles, distribution through time and space, evolution, and even their basic metabolism currently exists. Precise age determinations of rocks containing vertebrate fossils have been made only rarely (Fassett, 2009). A potential weakness in determining the age of a fossil based on the age of the strata containing the fossil is the possibility that the fossil in question may have been reworked from older strata. The direct dating of fossil bone could preclude the reworking hypothesis. We herein report the first successful direct U-Pb age determinations for two fossil dinosaur bones using an in situ laser ablation–multicollector–inductively coupled plasma–mass spectrometer technique (LA-MC-ICP-MS).

STUDY AREA AND PREVIOUS PUBLICATIONS

The San Juan Basin of northwestern New Mexico and southwestern Colorado (United States) (Fig. 1) has been an area of intense geologic study for 100 yr because of its rich endowment of vertebrate fossils concentrated in rocks adjacent to the Cretaceous-Paleogene boundary plus its enormous natural gas and coal resources, mostly concentrated in upper Cretaceous rocks. Previously published chemical data obtained for suites of Paleocene and Cretaceous dinosaur bone from the San Juan Basin (Fassett et al., 2002; Fassett, 2009) indicated that the outer surfaces of Paleocene dinosaur bone contained hundreds of parts per million U, whereas Cretaceous bone contained <50 ppm U. Average sums of rare earth elements (REE) in the outer surfaces of Paleocene bones are ~1500 ppm compared to twice that in Cretaceous bones, and the chondrite-normalized REE patterns for Paleocene bones were noticeably less fractionated. In the two bone samples selected for this study, fission-track radiography indicated a relatively homogeneous distribution of U, and ^{238}U decay series measurements indicated close approach to radioactive equilibrium. The sample collection localities and complete chemical data for these fossils were reported in Fassett et al. (2002) and Fassett (2009).

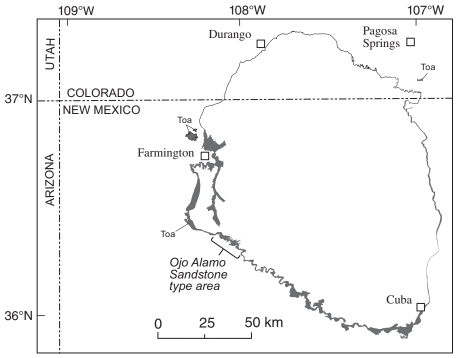

Figure 1. Index map of San Juan Basin, New Mexico and Colorado. Dinosaur bones analyzed in this study were collected from Ojo Alamo Sandstone type area in southwestern part of basin. Toa—Ojo Alamo Sandstone.

SAMPLES

The first bone sample analyzed for this study (22799-D; Fassett, 2009) was a limb bone fragment from a fine-grained sandstone bed in the upper part (late Campanian) of the upper Cretaceous Kirtland Formation, 6.1 m below the base of the Paleocene Ojo Alamo Sandstone. This locality is in the Ojo Alamo Sandstone type area in the southwestern part of the San Juan Basin (Fig. 1). This bone was in rock strata at virtually the same stratigraphic level as a volcanic ash bed from which a ^{40}Ar/^{39}Ar single-crystal sanidine age of 73.04 ± 0.25 Ma had been obtained previously (Fassett and Steiner, 1997; Fassett et al., 2002; Fassett, 2009); the bone collection site and the dated ash bed localities are only 3.5 km apart. Because of this excellent independent age control, this bone was our control sample.

The second dinosaur bone analyzed (BB1; Fassett, 2009) is a large fragment from the left femur of the sauropod dinosaur *Alamosaurus sanjuanensis*. This fossil was weathering out of a coarse-grained, reddish-brown, iron-cemented sandstone bed 1.8 m above the base of the Ojo Alamo Sandstone. The exact age of this specimen was unknown, but because of the pristine nature of its cortical surface and its Paleocene geochemical signature, it was interpreted to be in place and not reworked from underlying Cretaceous strata. This specimen was subsequently collected by R.M. Sullivan of the State Museum of Pennsylvania and labeled SMPVP-1138; the prepared specimen was illustrated in figure 2 of Lucas and Sullivan (2000). The complete chemical analyses and exact collection localities of both bones were reported in Fassett et al. (2002) and Fassett (2009).

U-PB ANALYSES

Sample 22799-D

For sample 22799-D, a longitudinal thick section was prepared for imaging and isotopic analysis. For sample BB1, both longitudinal and cross-sectional sections were prepared (Fig. 2) to test whether section orientation was critical to obtaining good quality data. Sections were examined with backscattered electron imaging and element (Ca, P, S, Si, Mn) mapping using a JEOL8900 electron microprobe at the University of Alberta to search for visible structural and chemical variations within the

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samples and to help guide selection of analysis sites. The U-Pb analyses were conducted with a Nu Plasma MC-ICP-MS coupled to a UP213 New Wave laser-ablation workstation. The MC-ICP-MS houses 12 Faraday and 3 ion counter detectors and is configured to allow for simultaneous collection of masses ^{204}Pb-^{206}Pb-^{207}Pb (on ion counters) and ^{203}Tl-^{205}Tl-^{235}U-^{238}U (on Faradays; 10, 11). Operating conditions during U-Pb bone analysis were similar to protocols established for U-bearing minerals (outlined elsewhere; Simonetti et al., 2005, 2006, 2008). The Durango apatite was used as an internal standard to monitor laser-induced elemental fractionation and instrument drift. Each analysis consisted of a single 160-µm-diameter laser spot (Figs. 2A and 2B) measured over a 60 s data collection cycle. Under these analysis conditions a relatively strong Pb signal was obtained (^{206}Pb signals varied between ~2 × 10^5 and 2 × 10^6 cps).

U-Pb results for 127 spot analyses obtained during two analytical sessions for bone sample 22799D are presented in Table DR1 in the GSA Data Repository[1] and on a ^{206}Pb/^{238}U age versus ^{238}U/^{204}Pb diagram (Fig. 3A) to evaluate the effects of postdepositional disturbance and uranium mobilization. There are two patterns in the data scatter in Figure 3A. The first is a decrease in age with increase in ^{238}U/^{204}Pb ratio. This pattern is attributed to a relatively young uranium-gain event (possibly ca. 20 Ma). Therefore, the majority of high ^{238}U/^{204}Pb ratios (i.e., ratios >~500) in this sample do not record primary age information. A second population of analyses has both low ^{238}U/^{204}Pb (<1000) and young age (younger than 40 Ma), reflecting an age disturbance that is not linked to secondary uranium mobility. The remainder of the analyses (n = 36) have low ^{238}U/^{204}Pb (<500) and ages that vary between ca. 60 and 80 Ma. The oldest of these analyses (n = 25; black symbols in Fig. 3; excluding two analyses with ^{206}Pb/^{238}U ages older than 80 Ma) are interpreted to represent undisturbed sites in the specimen and together yield a weighted average ^{206}Pb/^{238}U date of 73.6 ± 0.9 Ma (mean square of weighted deviates, MSWD = 1.06; Fig. 3B). This date agrees, within analytical uncertainty, with the ^{40}Ar/^{39}Ar sanidine age of 73.04 ± 0.25 Ma obtained from the nearby ash bed, ~4 m stratigraphically higher in the section, and is therefore interpreted to be a good estimate for the age of deposition and probably for primary diagenesis of this Cretaceous fossil bone.

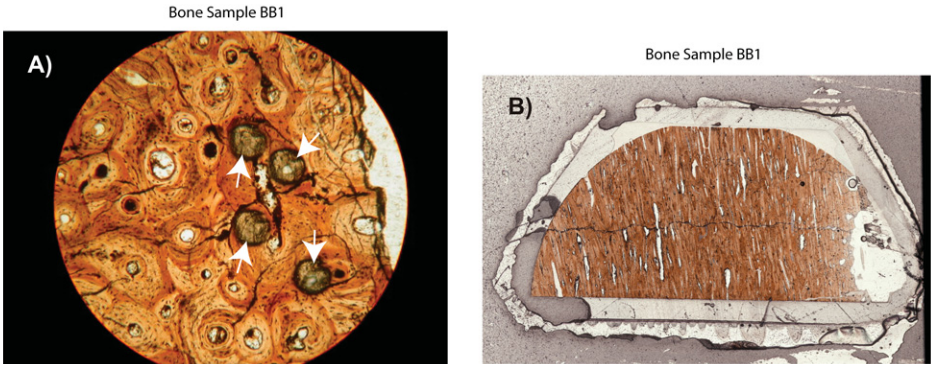

Figure 2. Polished slabs of dinosaur bone sample BB1 collected from Paleocene Ojo Alamo Sandstone in southern part of San Juan Basin, New Mexico and Colorado. A: Cross section; note four laser spots (white arrows); laser spots are 160 µm in diameter. B: Longitudinal section; slide is 2.5 cm wide.

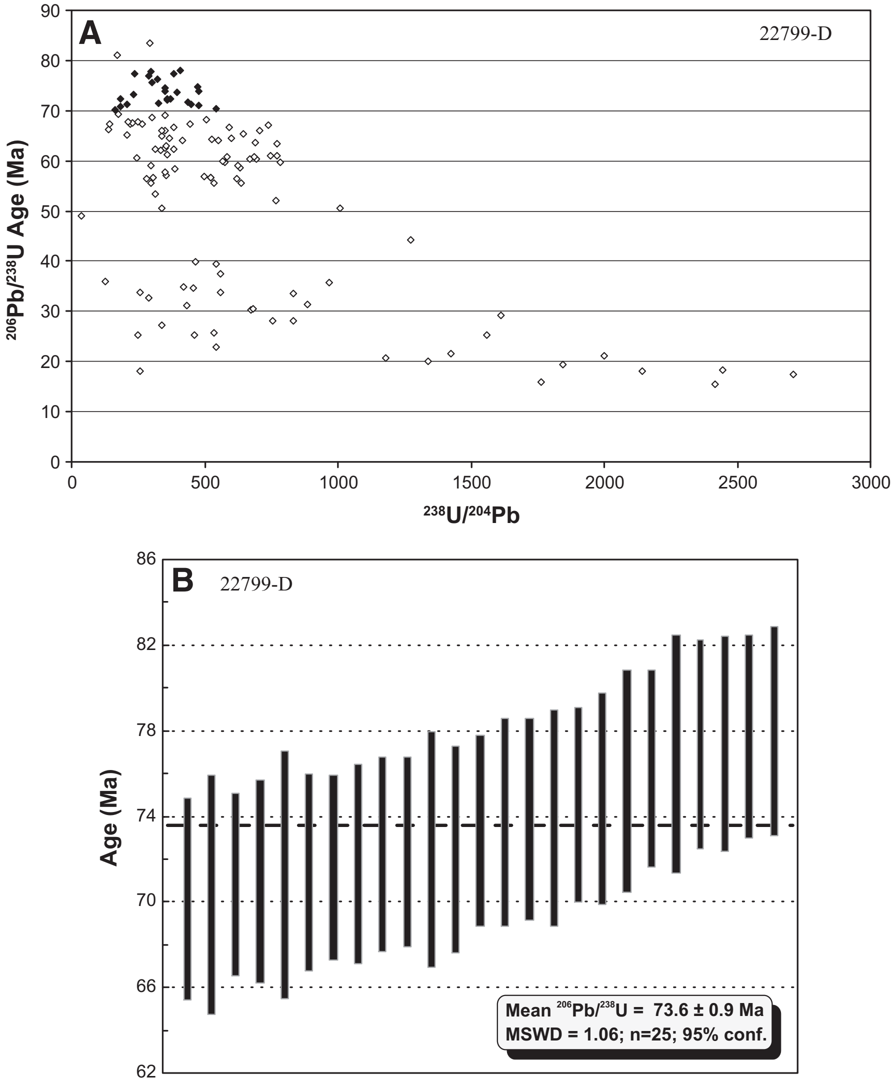

Figure 3. U-Pb results for control dinosaur bone 22799-D collected from Cretaceous Kirtland Formation in southern part of San Juan Basin, New Mexico and Colorado. A: ^{206}Pb/^{238}U age versus ^{238}U/^{204}Pb age diagram showing composition of unaltered parts of bone (solid symbols). B: Weighted average ^{206}Pb/^{238}U plot. MSWD—mean square of weighted deviates; conf.—confidence.

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[1] GSA Data Repository item 2011069, Tables DR1 and DR2, laser-ablation MC-ICPMS results for two dinosaur bone samples from strata adjacent to the Cretaceous-Paleocene interface, San Juan Basin, New Mexico, USA, is available online at www.geosociety.org/pubs/ft2011.htm, or on request from editing@geosociety.org or Documents Secretary, GSA, P.O. Box 9140, Boulder, CO 80301, USA.

Sample BB1

The U-Pb results for 80 spot analyses for two sections from bone sample BB1 are presented in Table DR2 and Figure 4. The data were collected during two analytical sessions, the first on a cross section and the second on a longitudinal section. The U-Pb data for BB1 also show some scatter toward higher ^{238}U/^{204}Pb ratios (the highest value is 1291), but in general this sample displays much less scatter in both age and ^{238}U/^{204}Pb than the control sample 22799D (cf. Figs. 3A and 4A). The age results from the two BB1 sections differ to some degree, but overlap within analytical uncertainty; the longitudinal section yielded a date of 64.6 ± 1.0 Ma (MSWD = 1.1, n = 50) and the cross section yielded a date of 63.0 ± 1.3 Ma (MSWD = 5.3, n = 50). If the two extreme outliers are omitted from the calculation, the ages obtained for both sections are slightly more precise with a significant reduction in the scatter for the cross-section data of 64.8 ± 0.9 Ma (MSWD = 0.84, n = 48) and of 62.8 ± 0.8 Ma (MSWD = 1.6, n = 28) (dates reported in Figs. 4B and 4C). If all the data excluding the outliers are considered together, a date of 63.4 ± 0.6 Ma (MSWD = 1.3, n = 76) is obtained.

On the basis of paleomagnetic data, one of us (Fassett, 2009) determined that the base of the Ojo Alamo Sandstone is 65.2 Ma, 0.3 m.y. younger than the 65.5 Ma age for the Cretaceous-Paleogene boundary of Gradstein et al. (2004); thus, a hiatus of ~7.8 m.y. separates Cretaceous and Tertiary strata in the southern San Juan Basin. If these age constraints are correct and bone BB1 is not reworked, then it must have a depositional and/or diagenetic age younger than 65.2 Ma; thus our U-Pb age of 64.8 Ma for dinosaur bone sample BB1 is in excellent agreement with these age constraints.

DISCUSSION

Bone sample BB1 displays the most consistent U-Pb results obtained for the two dinosaur bones analyzed and appears to be relatively undisturbed by any postdepositional diagenetic processes. There is some scatter in the ^{238}U/^{204}Pb ratios (Table DR2; Fig. 4A), indicating uranium addition to this bone, but this does not correlate with a change in the apparent ^{206}Pb/^{238}U dates; therefore, this variation was likely acquired by the bone during initial diagenesis. The weighted-mean ^{206}Pb/^{238}U dates obtained for the cross section of 62.8 ± 0.8 Ma (n = 28, MSWD = 1.6) and the longitudinal section of 64.8 ± 0.9 Ma (n = 48, MSWD = 0.8) are only just distinguishable within the quoted analytical uncertainties; however, we interpret the 64.8 Ma date to be closer to the time of initial bone diagenesis. This date was obtained on a longitudinal bone slice (orientation similar to that of our control sample), and we recommend that future attempts at U-Pb dating of vertebrate bone be conducted on longitudinal, near-cortical-surface sections. We attribute the slightly younger, weighted-mean date for BB1A to possibly reflect a protracted period (1 k.y. to 1 m.y.) for complete uranium mineralization to occur. Some of the analysis spots in this section were obtained from locations near the cortical surface and others from deeper within the bone. We anticipate U mineralization accompanying bone recrystallization to have progressed from the outside to the center; thus ages obtained from the inside of the bone should be slightly younger than the burial age.

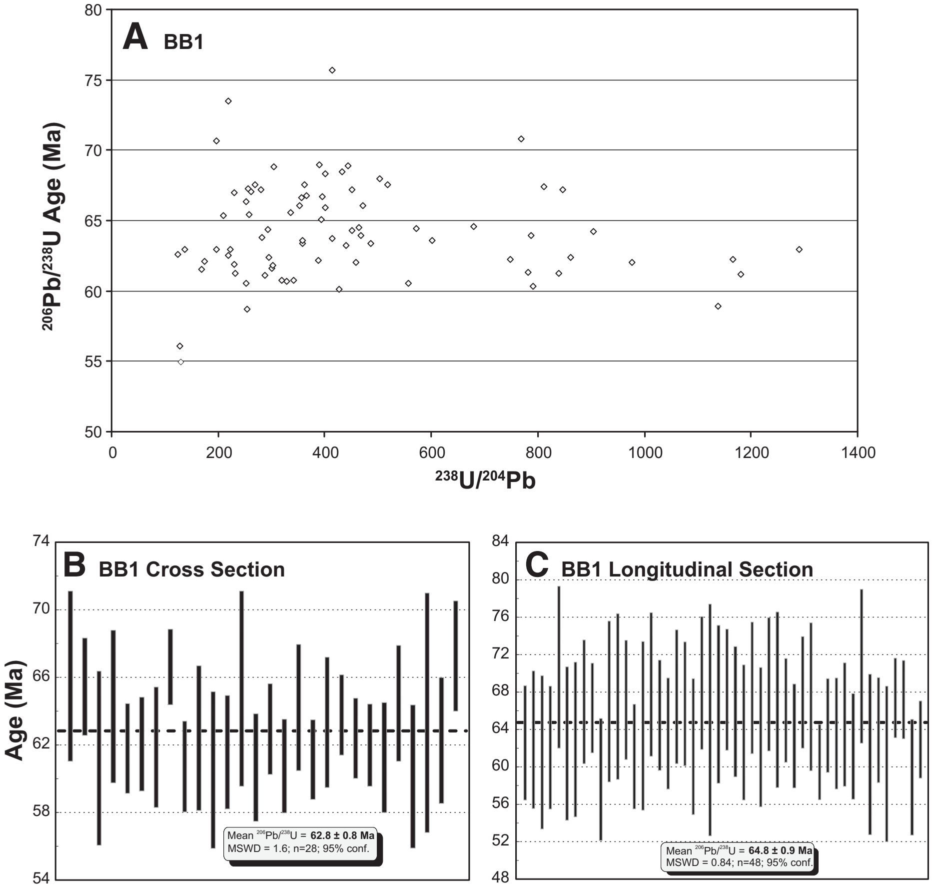

Figure 4. U-Pb results for bone BB1. A: ^{206}Pb/^{238}U age versus ^{238}U/^{204}Pb age diagram showing relatively uniform age range regardless of ^{238}U/^{204}Pb composition. B, C: Weighted average ^{206}Pb/^{238}U plots. MSWD—mean square of weighted deviates; conf.—confidence.

An alternative interpretation of the 64.8 Ma U-Pb age obtained for BB1 is that it records a secondary age of mineralization that could be distinctly younger than its age of deposition. However, a growing body of evidence from relatively recent studies of fossil bone mineralization (Trueman and Benton, 1997; Trueman et al., 2006) provides strong evidence for extremely rapid recrystallization and mineralization of buried bones. Kohn (2008, p. 3758), for example, stated: "Modeled uptake rates for fossil teeth yield a strict minimum bound on durations of about one decade to one century. The similarity of diffusion profiles in teeth, irrespective of depositional ages ranging from ca. 30 ka to >30 Ma, implies that uptake occurred quickly, with a maximum duration of a few tens of kyr for typical fossil enamel; *faster uptake is implied for typical fossil bone and dentine*" [our italics].

Thus it is likely that the age of 64.8 Ma for bone sample BB1 reflects an age no more than a few tens of thousands of years (at most) younger than the time of death of this animal and burial of its femur.

Paleomagnetic studies of the Ojo Alamo Sandstone in the southern San Juan Basin indicate that bone BB1 came from a stratigraphic level in the lowermost part of paleomagnetic chron C29n (Fassett, 2009). The base of magnetochron C29n has an age of 65.118 Ma (Gradstein et al., 2004). An altered volcanic ash bed in the Nacimiento Formation 125 m above the base of the Ojo Alamo Sandstone yielded a ^{40}Ar/^{39}Ar age of 64.0 ± 0.4 Ma (Fassett et al., 2010). Thus existing geochronologic data constrain the age of bone sample BB1 to a 1.1 m.y. time interval, between 65.1 and 64.0 Ma. The age of 64.8 Ma for BB1 is therefore in perfect agreement with these age constraints. In addition, this coincidence of ages lends further credence to the argument that bone recrystallization takes place very soon after burial.

CONCLUSIONS

A previous attempt to date dinosaur bones by U-Pb using isotope dilution-thermal ionization mass spectrometry by Romer (2001) was unsuccessful. However, the in situ U-Pb dating technique developed for this study was successful and opens the door to the direct dating of other vertebrate fossils and, if those fossils are not reworked, the strata that contain them. All too often, vertebrate paleontologists have had no precise way to directly date their fossils; thus the ages of vertebrate-bone assemblages have often been the subject of wide disagreement (e.g., Clemens and Williamson, 2005). In addition, because of the endemic nature of many vertebrate species, vertebrate paleontologists have had difficulties in determining the time equivalence of taxa from different areas. Because vertebrate fossil collection sites are rare throughout the world, attempts to determine biogeographic diversity of taxa and the evolution and radiation of vertebrates have been handicapped by a lack of knowledge of the precise ages of these fossils. Direct dating of vertebrate fossils using the techniques described herein has the potential to revolutionize this biochronologic aspect of vertebrate paleontology.

We believe that the 64.8 ± 0.9 Ma age obtained for the longitudinal section BB1B provides an accurate age for the deposition and diagenesis of this dinosaur bone. Moreover, because this bone was dated directly, its age supports the geochemical data indicating that this bone was not reworked from underlying Cretaceous strata. This direct U-Pb age for bone BB1 also provides independent evidence suggesting the possible survival of some dinosaurs into the Paleocene in the San Juan Basin area, as proposed in Fassett (1982, 2009), Fassett et al. (1987, 2002), and Fassett and Lucas (2000).

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