$^{40}Ar/^{39}Ar$ dating of ignimbrite from Inner Mongolia, northeastern China, indicates a post-Middle Jurassic age for the overlying Daohugou Bed

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[1] The $^{40}Ar/^{39}Ar$ step heating analyses of K-feldspar derived from the ignimbrite in Inner Mongolia, China indicates that they were formed at 159.8 ± 0.8 Ma, which provides a maximum age for the overlying fossil-bearing lacustrine deposits (Daohugou Bed). This result favors a post-Middle Jurassic (Late Jurassic or younger), rather than the Middle Jurassic age for the Daohugou Bed. Such a result is generally consistent with vertebrate biostratigraphic evidence, providing a maximum known age for the first appearance of several major animal groups such as Cryptobranchidae of Urodela (salamanders) and Maniraptora (birds and their closest dinosaurian relatives). *INDEX TERMS:* 1035 Geochemistry: Geochronology; 8404 Volcanology: Ash deposits; 9320 Information Related to Geographic Region: Asia; 9609 Information Related to Geologic Time: Mesozoic. **Citation:** He, H. Y., X. L. Wang, Z. H. Zhou, R. X. Zhu, F. Jin, F. Wang, X. Ding, and A. Boven (2004), $^{40}Ar/^{39}Ar$ dating of ignimbrite from Inner Mongolia, northeastern China, indicates a post-Middle Jurassic age for the overlying Daohugou Bed, *Geophys. Res. Lett.*, *31*, L20609, doi:10.1029/2004GL020792.

1. Introduction

[2] In recent years, a new Mesozoic fresh water fossil locality in Daohugou village, Ningcheng County, southeastern Inner Mongolia, has drawn extensive attention among paleontologists, not only for producing hundreds of beautifully preserved salamanders [*Wang*, 2000; *Gao and Shubin*, 2001, 2003], insects [*Zhang*, 2002], and arboreal dinosaur [*Zhang et al.*, 2002], haired pterosaurs [*Wang et al.*, 2002] and some unpublished mammals, but also for its controversial relationship to the Early Cretaceous Jehol Biota in the adjacent western Liaoning Province, which is well known for preserving exceptional lacustrine fossils including birds, feathered dinosaurs, mammals and angiosperms. Therefore, the Daohugou locality becomes one of the most important Mesozoic fossil sites in East Asia [*Chang et al.*, 2003].

[3] The lake deposits at the Daohugou locality have long been considered as a southward extension of the Yixian Formation, the lower part of the Jehol Group. Recently, the deposits at this locality were newly named the Daohugou Bed, as the lowest part of the Yixian Formation [*Wang*, 2000; *Wang et al.*, 2000, 2002]. On the other hand, some other workers proposed a new formation, the Daohugou Formation to distinguish it from the Yixian Formation of the Jehol Group, and the concept of the Pre-Jehol Biota has been coined to indicate both its similarities to, and differences from, the Jehol Biota [*Zhang*, 2002]. Since the name of the Daohugou Formation was not appropriately set up with a stratigraphic section, we would prefer to use the Daohugou Bed in this paper [*Wang*, 2000].

[4] Like its debatable stratigraphic correlation, the age of the deposits has also been very controversial, ranging from the Middle Jurassic to the Late Jurassic or the Early Cretaceous. Vertebrate paleontologists generally prefer a Late Jurassic or Early Cretaceous age for the deposit [*Wang et al.*, 2000; *Ji and Yuan*, 2002]. Invertebrate paleontologists however, in particular, those working on insects and conchostracans disagree that it is part of the Yixian Formation. Some even went further to suggest that the Daohugou deposits could be correlated to the Middle Jurassic (Bathonian) Jiulongshan Formation [*Ren et al.*, 2002].

[5] Recently, *Chen and Zhang* [2004] have obtained Sensitive High Resolution Ion Micro Probe mass spectrometer (SHRIMP) U-Pb ages of the zircon and $^{40}Ar/^{39}Ar$ step heating ages of the biotite and K-feldspar for the andesite, rhyolite and a kind of reddish igneous rock at the Daohugou locality. The age is about 159–165 Ma, however, isotopic dates on volcanics from the Daohugou locality have not helped to resolve the controversy because the relationship of these dated volcanics to fossil-bearing shale is not clear. Here we report the results of $^{40}Ar/^{39}Ar$ dating of K-feldspars from the Daohugou ignimbrite, which are directly in contact with the shale and discuss its implications for the stratigraphic and biotic comparisons with the Early Cretaceous Jehol Biota in northeastern China.

2. Samples, Analyses and Results

[6] The Daohugou locality lies near the border between southern Inner Mongolia and Lingyuan of western Liaoning Province, northeastern China (Figure 1). The lacustrine deposits at the Daohugou locality mainly comprise grayish or gray tuffaceous shales and mudstones with tuffaceous breccia at the bottom, and various tuff layers interbedded in the shales and mudstones. The ignimbrite, which were given the field name as andesite, are extensively distributed in the Daohugou basin upon the shales and were previously

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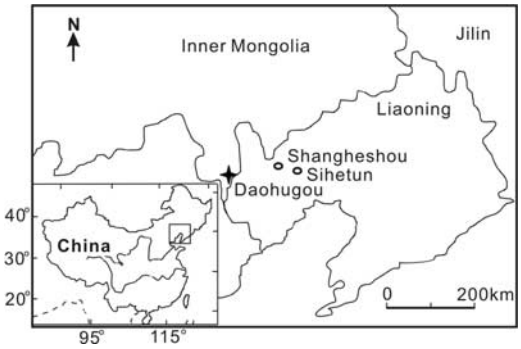

Figure 1. Sketch map showing the location of Daohugou section.

believed to overly the Daohugou fossil bearing shales conformably though no evidence has been reported [*Ren et al.*, 2002; *Chen and Zhang*, 2004]. However, a newly discovered contact zone (119.22°E, 41.32°N) between the ignimbrite and the shale results in a completely different explanation. The section shows a reversed sequence, from the bottom to the top, the shales conformably contact the red layer, which is composed of mineral components from weathered ignimbrite (Figure 2). The shale deposits obviously represent a later event than the ignimbrite; hence, the dating of the ignimbrite will provide a maximum age for the fossil-bearing sediments.

[7] Sample L3013 was collected from the ignimbrite at the Daohugou locality (119.24°E, 41.32°N). Field excavations at this section by the Institute of Vertebrate Paleontology and Paleoanthropology in 2002 resulted in discoveries of hundreds of fossils including lizards, dinosaurs and insects in the overlying fossil-bearing shales.

[8] Thin section study shows that the ignimbrite L3013 is mainly composed of fine volcanic ash (80–90%), sanidine and orthoclase crystal (5–15%) and biotite crystal (5–10%). Since K-feldspar is fresher than biotite and it was chosen for laboratory analysis.

[9] Ignimbrite samples were crushed and sieved between 80–100 mesh (200–120 μm) fractions, and rinsed with distilled water. After heavy liquid separation the K-feldspars (sanidine and orthoclase) were obtained and washed with acetone in an ultra-sonic bath for 20 minutes. Cleaned K-feldspars were wrapped in Al foil and irradiated together with Ga1550-biotite standards, optical CaF_2 and K-glass monitors in position H8 of the 49-2 reactor, Beijing, China, for 47.5 hours with 0.5 mm cadmium foil shield. The reference age for GA-1550 biotite is 98.79 ± 0.96 Ma [*Renne et al.*, 1998]. Ca, K correction factors were calculated from the CaF_2 and K-glass monitors: $(^{40}Ar/^{39}Ar)_K = 1.13 \times 10^{-2}$, $(^{39}Ar/^{37}Ar)_{Ca} = 7.24 \times 10^{-4}$, $(^{36}Ar/^{37}Ar)_{Ca} = 2.39 \times 10^{-4}$.

[10] The total fusion of standards and step-heating analyses of samples were performed at the $^{40}Ar/^{39}Ar$ Geochronology Lab, Institute of Geology and Geophysics, Chinese Academy of Sciences, on a MM5400 mass spectrometer operating in a static mode. The data were corrected for system blanks, mass discriminations, interfering Ca, K derived argon isotopes, and the decay of ^{37}Ar since the time of the irradiation. The decay constant used throughout the calculations is $\lambda = (5.543 \pm 0.010) \times 10^{-10}$ a^{-1}, as recommended by *Steiger and Jäger* [1997]. Detail of the analysis and data processing procedures are outlined in *He et al.* [2004]. The results of the $^{40}Ar/^{39}Ar$ experiments are shown in Table 1 and plotted as age spectrum and isotope correlation diagrams in Figure 3.

[11] The K-feldspar separated from ignimbrite L3013 yields a concordant age spectrum (Figure 3a). Three consequent steps, which account for 58.8% of the total ^{39}Ar released, define a plateau age of 160.4 ± 0.8 Ma (2σ). An inverse isochronal age of 159.8 ± 0.8 Ma (2σ, MSWD = 8.9), calculated from all steps, is in good agreement with the plateau age. The $^{40}Ar/^{36}Ar$ intercept of 302.7 ± 9.6 (2σ) is not distinguishable from the air ratio, indicating that there's no apparent excess argon contamination (Figure 3b). Therefore, 159.8 ± 0.8 Ma (2σ) is the good estimate of the eruption age of ignimbrite L3013.

3. Discussion and Conclusions

[12] $^{40}Ar/^{39}Ar$ step heating of the K-feldspar from the ignimbrite at the Daohugou locality gives an isochron age of 159.8 ± 0.8 Ma, corresponding to the latest Middle Jurassic (Callovian) or the earliest Late Jurassic (Oxfordian). Considering the vastly different isotope system behavior, this result is more or less concordant with the SHRIMP U-Pb zircon age (165.5 ± 1.5 Ma, 1σ, MSWD = 0.75) for the andesite reported by *Chen and Zhang* [2004]. They argued that such a dating result indicates the Daohugou deposit, which was presumed to be overlaid by the volcanic rock, must be older than 165 Ma, thus lends further support to its referral to the Middle Jurassic (Bathonian). However, the field examination of the relationship between the ignimbrite and deposits has resulted in a completely different conclusion about the age of the Daohugou Bed. The direct contact between the ignimbrite and the Daohugou sediments near the Daohugou village (119.22°E, 41.32°N) shows no baking of the sediments near the volcanic rocks,

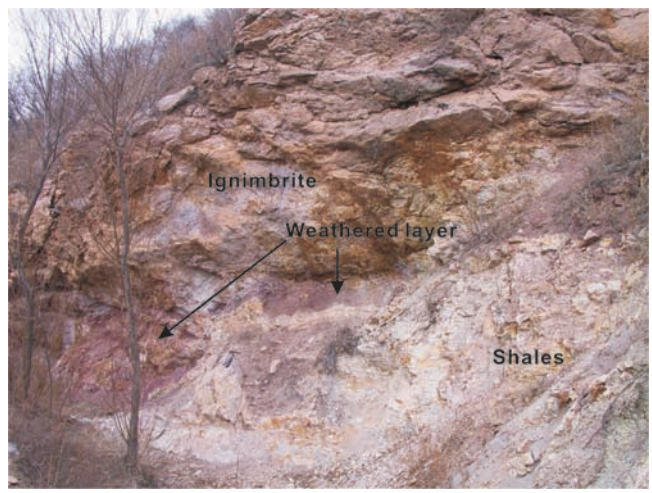

Figure 2. The direct contact between the weathered layer of the ignimbrite and the Daohugou shales near the Daohugou village (119.22°E, 41.32°N). The presence of the weathered layer of the ignimbrite indicates that the shales were deposited later than the ignimbrite, thus the section shows a reversed sequence, i.e., the upper ignimbrite is older than the lower shales.

Table 1. $^{40}Ar/^{39}Ar$ Analytical Data for K-Feldspar Separate of Ignimbrite L3013[a]

Temp (°C)	$^{40}Ar/^{39}Ar \pm 2$ s.d.	$^{36}Ar/^{39}Ar \pm 2$ s.d. (10^{-3})	Ca/K	^{39}Ar (10^{-14} moles)	^{39}Ar Cum (%)	$^{40}Ar^*$ (%)	$^{40}Ar^*/^{39}Ar$	Apparent Age ± 2 s.d. (Ma)
800	11.22684 ± 0.03	1.66 ± 0.05	0.24	2.31	3.00	95.64	10.74	167.3 ± 1.5
900	10.362 ± 0.01	0.74 ± 0.01	0.34	16.13	23.96	97.89	10.14	158.5 ± 1.3
970	10.34061 ± 0.01	0.18 ± 0.00	0.39	23.84	54.94	99.50	10.29	160.7 ± 1.4
1020	10.40661 ± 0.01	0.29 ± 0.01	0.45	13.34	72.27	99.17	10.32	161.1 ± 1.4
1080	10.4082 ± 0.01	0.68 ± 0.01	0.73	8.05	82.73	98.08	10.21	159.5 ± 1.4
1150	10.29559 ± 0.01	0.70 ± 0.02	0.95	5.92	90.42	97.98	10.09	157.7 ± 1.3
1200	10.31532 ± 0.02	0.42 ± 0.01	0.66	5.13	97.09	98.79	10.19	159.2 ± 1.4
1260	10.49683 ± 0.02	1.43 ± 0.04	1.94	1.50	99.03	95.97	10.07	157.4 ± 1.4
1330	11.63423 ± 0.18	5.34 ± 0.13	2.87	0.50	99.68	86.43	10.06	157.2 ± 2.8
1400	14.5533 ± 0.37	13.54 ± 0.52	3.83	0.18	99.91	72.50	10.55	164.6 ± 4.6
1500	22.00698 ± 2.16	35.68 ± 4.03	4.49	0.07	100.00	52.09	11.46	178.1 ± 18.9

[a]Weight = 33.25 mg, J = 0.009052 ± 0.00004.

excluding the possibility of the ignimbrite having intruded later. Therefore, the overlying Daohugou Bed lake deposits clearly represent a geological event later than the ignimbrite.

[13] The dating result of the Daohugou ignimbrite provides a maximum age for the deposits that are characteristic of abundant salamander and insect fossils, and arboreal theropod dinosaurs and haired pterosaurs. This result lends support to a post-Middle Jurassic (e.g., Late Jurassic/Early Cretaceous) age rather than the Middle Jurassic age for the Daohugou Bed. Thus, despite the claim of the earliest known (Middle Jurassic) crown-group urodeles (closest relative of living salamanders) occurring from the Daohugou locality in Inner Mongolia, the age of the underlying ignimbrite does not seem to support such a conclusion. And it also implies that the earliest known arboreal maniraptoran had not been discovered before the Late Jurassic.

[14] Vertebrate studies generally favor a Late Jurassic/Early Cretaceous view for the Daohugou Bed. For instance, the Daohugou salamander assemblage is generally comparable to that of the Late Jurassic Karatau Formation in Kazakhstan, and it also contains elements comparable to the typical Jehol assemblage [*Wang*, 2004]. The studies on the pterosaurs in this region arrive at approximately the same conclusion: the short tailed rhamphorhynchoid pterosaur *Jeholopterus* is referable to the Anurognathidae, which currently comprises only 4 genera [*Wang et al.*, 2002]. The other three genera are from the Late Jurassic limestone in Solnhofen, the Late Jurassic Karatau Formation in Kazabstau, and the Early Cretaceous Yixian Formation in Liaoning, respectively [*Zhou et al.*, 2003; *Zhou*, 2004]. The only reported dinosaur from the Daohugou locality is *Epidendrosaurus*, which is an arboreal maniraptoran theropod very close to the transition to birds [*Zhang et al.*, 2002].

[15] The Daohugou Biota is unambiguously different from that of the Jehol Biota despite the presence of the evidence of a close connection. As in the case of invertebrates and plants, the vertebrate assemblage of the Daohugou Biota shows more primitive appearance than the Jehol Biota. Therefore, the determination of the age of the Daohugou Bed is critical for our understanding of the origin of various animal groups as well as the Jehol Biota.

[16] Despite the dating reported in this paper, the exact age of the Daohugou Bed in Inner Mongolia remains unsolved as there is yet no evidence showing how long the gap existed between the deposits and the volcanic base. It is, however, noteworthy that at the Daohugou locality, the fossil-bearing sediments are also found to overlie the Tuchengzi Formation in a few sites. The Tuchengzi Formation comprises gray, green to reddish sandstones, conglomerates and shales and is unconformably overlied by the Yixian Formation in many sites of Liaoning and its neighboring areas. Earlier $^{40}Ar/^{39}Ar$ dating of the upper part of the Tuchengzi Formation in Beipiao, Liaoning Province gave a mean age of 139.4 ± 0.2 Ma [*Swisher et al.*, 2002]. Therefore, it appears that the Daohugou Bed is most likely younger than this age. Considering the abovementioned contacts and dating, as well as the primitive appearance of the Daohugou Biota compared to the typical Jehol Biota, we propose that the Daohugou Bed may represent the Early Cretaceous intermontaneous deposits between the Tuchengzi and the Yixian formations.

[17] In summary, the data acquired for the Inner Mongolian ignimbrite is from the latest Middle Jurassic (Callovian) or the earliest Late Jurassic (Oxfordian), thus the overlying fossil-bearing Daohugou Bed must be Late Jurassic or

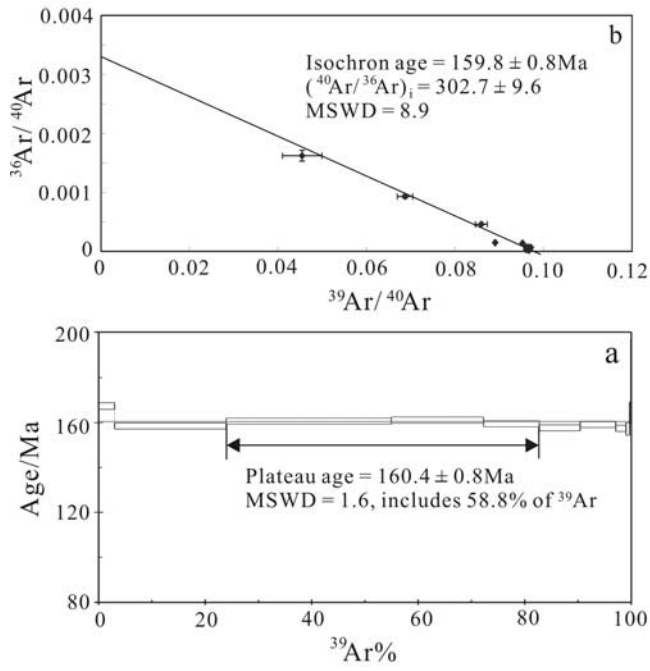

Figure 3. (a) Age spectrum of L3013; (b) inverse isochron plot of L3013.

younger in age. This result is in contrast to a previously proposed Middle Jurassic age for these deposits. Therefore, it is also obvious that more fossil evidence than those known from the Daohugou locality is needed to claim that the early diversification of salamanders was well underway by the Middle Jurassic. The determination of the maximum age for the Daohugou Bed is also important, as it has reduced the temporal gap between the Daohugou fossil assemblage in Inner Mongolia and the typical Jehol Biota in Liaoning Province, and bears significance on our understanding of several other important vertebrate groups such as pterosaurs, theropods and mammals that have been well represented in these deposits.

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