Timing of the Jiufotang Formation (Jehol Group) in Liaoning, northeastern China, and its implications

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[1] The timing of the Jiufotang Formation remains speculative despite recent progress in the study of the Jehol Biota. In this paper we contribute to this topic with $^{40}Ar/^{39}Ar$ dating on K-feldspar (sanidine and orthoclase) from tuffs interbedded within the fossil-bearing shales of the Jiufotang Formation, from the upper part of the Jehol Group in Chaoyang, Liaoning, northeastern China. $^{40}Ar/^{39}Ar$ step heating analyses of K-feldspar and the SHRIMP U-Pb zircon data indicate that tuffs at the Shangheshou section erupted at 120.3 ± 0.7 million years ago. This result confirms an Aptian age for the Jiufotang Formation that was mainly based on biostratigraphic evidence. It also places stringent controls on the age of the fossils from the formation, providing a minimum age (120 Ma) for the four-winged dinosaur, *Microraptor*, and the seed-eating bird, *Jeholornis*. *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, F. Wang, A. Boven, G. H. Shi, and R. X. Zhu (2004), Timing of the Jiufotang Formation (Jehol Group) in Liaoning, northeastern China, and its implications, *Geophys. Res. Lett.*, *31*, L12605, doi:10.1029/2004GL019790.

1. Introduction

[2] Discoveries of a variety of exceptionally well preserved early birds, feathered dinosaurs, primitive mammals and angiosperms have brought global significance to the Jehol Biota [*Sereno and Rao*, 1992; *Hou et al.*, 1995; *Norell et al.*, 2002], a unique Lower Cretaceous ecosystem in east and central Asia. However, the lack of sufficient and reliable chronostratigraphic data for the Jehol deposits has become a growing predicament not only for explicit comparisons with penecontemporaneous biomes from other regions but also for establishing or evaluating the earliest appearance of many important vertebrate groups and angiosperms [*Jerzykiewicz and Russell*, 1991; *Zhou et al.*, 2003]. The Jehol Group comprises the Yixian Formation and the overlying Jiufotang Formation. The age of both the Yixian and Jiufotang formations have been contentious among paleontologists in the past, varying from the Late Jurassic to Early Cretaceous. Interbedded tuff from the lower Yixian Formation (Sihetun section, Figure 1a) provides the most direct age for the fossil-bearing sediments in the lower Jehol Group [*Swisher et al.*, 1999, 2002; *Wang et al.*, 2001]. Till now no precise age has, however, been obtained for the Jiufotang Formation, the upper part of the Jehol Group. The only radioisotopic age of the Jiufotang Formation has been dated from Tebch, Inner Mongolia, with a $^{40}Ar/^{39}Ar$ date of 110.59 ± 0.52 million years obtained from intrusive basalt within the formation [*Eberth et al.*, 1993], which probably provides a top limit for the Jiufotang Formation. There is, however, no evidence indicating how much younger the basalt is than the fossil-bearing sediments although the pollen from the clastic deposits at Tebch, Inner Mongolia supports a Barremian or Early Aptian age [*Eberth et al.*, 1993].

[3] The Jiufotang Formation mainly comprises lacustrine sandstones, shales, mudstones, and tuffs interbedded within the sediments [*Chang et al.*, 2003]. A number of important vertebrate discoveries have been made from the Jiufotang Formation in the last two decades, such as fish, amphibians, dinosaurs, and early birds. Most notable among them are the four-winged dinosaur *Microraptor gui* [*Xu et al.*, 2003], and the long-tailed seed-eating bird *Jeholornis prima* [*Zhou and Zhang*, 2002a]. Other major vertebrates that were recently discovered at the Shangheshou section include: *Peipiaosteus* and *Protopsephurus*, two of the primitive acipenseriform fish; *Sinopterus*, the first tapejarid pterosaur found outside Brazil [*Wang and Zhou*, 2003]; *Psittacosaurus*, one of the most famous dinosaurs of the Jehol Biota [*Chang et al.*, 2003]; and *Sapeornis*, the largest bird of the Early Cretaceous [*Zhou and Zhang*, 2002b]. The Shangheshou locality has become arguably one of the most productive localities of the Jiufotang Formation, much like the Sihetun locality of the Yixian Formation. Here we report the results of $^{40}Ar/^{39}Ar$ dating of K-feldspars from the tuffs interbedded within the shale at the Shangheshou section and discuss its implications for the evolution of biotic change in the Jehol Biota.

2. Samples, Analyses and Results

[4] The tuff samples were collected from the Jiufotang Formation at the Shangheshou locality (120.38°E, 41.59°N), Chaoyang City, Liaoning Province, northeastern China (Figure 1a). The stratigraphic sequence of this section consists of fossil-bearing shales and the Quaternary sediments, which unconformably cover the shales (Figure 1b). Two volcanic ash horizons are present in the section and have been labeled as L3001 and L3002 (Figure 1c). Birds and feathered dinosaurs were rich at a level (main fossil-bearing shale) about 1.5 meters below the L3001 (Figure 1b). Field excavations at this section by the Institute of Vertebrate Paleontology and Paleoanthropology in 2000 have resulted in the discoveries of hundreds of vertebrate fossils including fish, turtles, lizards, dinosaurs and birds.

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[5] Samples were collected from two yellowish tuff layers, in the field. Sample L3001 (Figure 1c) was from the lower layer which is ~5 cm thick, and Sample L3002 (Figure 1c) from the upper layer. Since Sample L3001 is fresher than L3002 it was chosen for laboratory analysis. In addition, Sample Lx9 was collected from a 1–2 mm thick ash layer within the lower tuff layer (Figure 1c). This thin ash layer is characterized by well defined boundaries with overlying and underlying tuffs, the richness of pyrogenic crystals, continuous distribution and good preservation (Figure 1d), indicating its air-fall origin [*Fisher and Schmincke*, 1984]. Thin section studies and electron probe micro-analysis show that there is no significant compositional difference between Sample L3001 and Lx9, and both are composed of sanidine and orthoclase (10–15%), plagioclase (30–40%), quartz (30–40%), altered biotite (10–15%) and accessory minerals such as zircon and opaque minerals.

[6] Tuff samples were crushed and sieved between 80–100 mesh (200–120 μm) fractions, and washed 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 stacked in a quartz vial. Ga1550-biotite standards, optical CaF_2 and K-glass monitors were stacked between the samples. The vials were shielded with cadmium foil (0.5 mm thick) and irradiated in position H8 of the 49-2 reactor, Beijing, China, for 47.5 hours.

[7] After the irradiation and a period of cooling, the total fusing of standards and argon 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. Every standard yields a J-value, combined with the sample position in the capsule, the line fit was done and J-value of each sample was calculated. The uncertainty of J-value is one standard deviation of mean and does not take into account uncertainties in the "absolute" age of the monitor mineral. We use the reference age 98.79 ± 0.96 Ma [*Renne et al.*, 1998] for GA-1550 biotite standard to calculate our results, so the age can be compared directly with the most recent compilation of $^{40}Ar/^{39}Ar$ age for the Sihetun tuff which is 125.0 ± 0.1 Ma with a relative

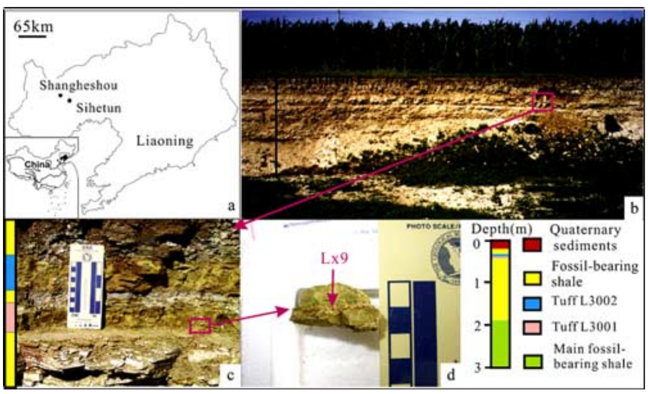

Figure 1. (a) Sketch map showing the location of the studied section, (b) Sampling section, (c) Sampling location, and (d) Tuff Lx9 interbedded in tuff L3001.

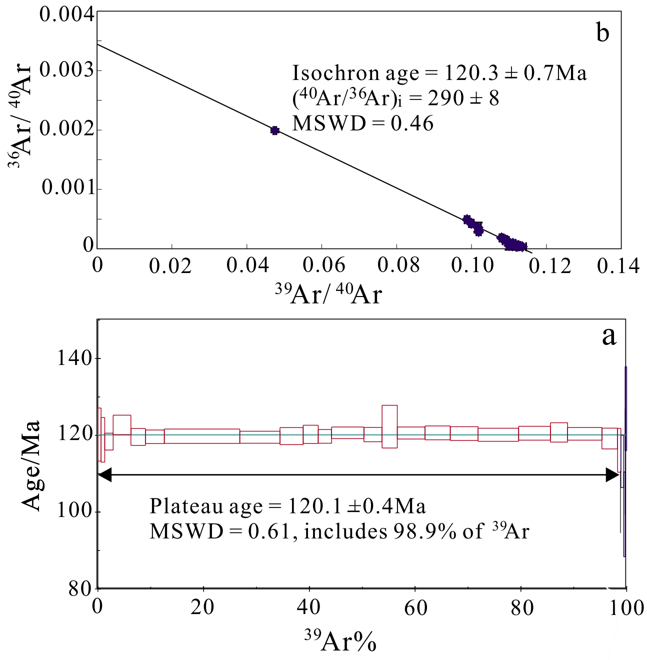

Figure 2. (a) Age spectrum of Lx9, (b) inverse isochron plot of Lx9.

age of 28.02 Ma for Fish Canyon Sanidine monitor mineral [*Swisher et al.*, 2002]. Ca, K correction factors were calculated from the CaF_2 and K-glass monitors: $(^{40}Ar/^{39}Ar)_K = 1.13 \times 10^{-2}$, $(^{39}Ar/^{37}Ar)_{Ca} = 7.24 \times 10^{-4}$, $(^{36}Ar/^{37}Ar)_{Ca} = 2.39 \times 10^{-4}$. Ca/K ratios are deduced from the relative production rates for $^{37}Ar_{Ca}$ and $^{39}Ar_K$, whereby Ca/K = $1.77 \times (^{37}Ar_{Ca}/^{39}Ar_K)$. The constant was calculated from co-irradiated MMhb-1.

[8] Samples were loaded into Christmas tree and degassed at 600°C for 10 minutes before being incrementally heated in a double vacuum furnace. The gases released during each step were purified by 2 SAES getters (operated at 350°C and 100°C, respectively) before they were introduced into the mass spectrometer for Ar isotopes measuring. The ^{40}Ar, ^{39}Ar, ^{38}Ar, ^{37}Ar and ^{36}Ar isotopic abundance are determined through linear extrapolation at time zero of peak intensities. These data (see auxiliary material[1]) were corrected for system blanks, mass discriminations, interfering Ca, K derived argon isotopes, and the decay of ^{37}Ar since the time of 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]. Plateau age was calculated by ISOPLOT [*Ludwig*, 1999], and the isochron age was calculated by CHROPLOT.

[9] The K-feldspar separated from tuff Lx9 yields a concordant age spectrum (Figure 2a). Twenty-three consequent steps, which account for 98.9% of the total ^{39}Ar released, define a plateau age of 120.1 ± 0.4 Ma (2σ). An inverse isochronal age of 120.3 ± 0.7 Ma (2σ), calculated from all steps that formed the plateau, is in agreement with the plateau age. The $^{40}Ar/^{36}Ar$ intercept of 290 ± 8 (2σ) is

[1]Auxiliary material is available at ftp://ftp.agu.org/apend/gl/2004GL019790.

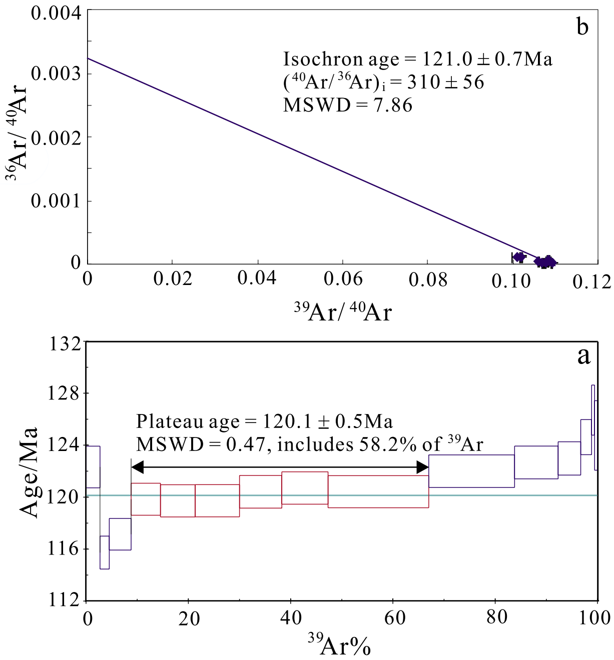

Figure 3. (a) Age spectrum of L3001, (b) inverse isochron plot of L3001.

not distinguishable from the air ratio, and MSWD = 0.46 (Figure 2b). Homogenously distributed radiogenic Ar, and low Ca/K ratios indicate little alteration and no apparent xenocrystic contamination of the K-feldspar separated from tuff Lx9. Therefore, the eruption age of tuff Lx9 (120.3 ± 0.7 Ma) is reliable and represents the deposit time of the fossil-bearing shales at the Shangheshou section.

[10] The K-feldspar separated from tuff L3001 gives a slightly saddle shaped age spectrum with a minimum age of 115.7 Ma and a maximum age of 126.7 Ma in the highest temperature steps (Figure 3a). Despite this, the 6 consequent steps, which account for 58.2% of the total ^{39}Ar released, define a plateau age of 120.1 ± 0.5 Ma (2σ). Isotopic ratios from steps are too scattered to define a reasonable inverse isochron (Figure 3b).

[11] $^{40}Ar/^{39}Ar$ dating on volcanic ash is frequently hampered by excess argon or xenocrystic contamination and both of them featured a saddle-shaped spectrum [*Lo Bello et al.*, 1987]. Thus we infer tuff L3001 might have undergone excess argon or xenocrystic contamination.

[12] To address the xenocrystic contamination and possible excess argon, the Sensitive High Resolution Ion Micro Probe mass spectrometer (SHRIMP) U-Pb zircon dating of tuff L3001 was also undertaken at Beijing SHRIMP Lab, Institute of Geology, Chinese Academy of Geological Science. Twenty-one analyses on 21 zircon grains or fragments from sample L3001 were performed. Except three outliers, 11 analyses constitute a coherent group and yield a weighted mean $^{206}Pb/^{238}U$ age of 124 ± 4 Ma (2σ, X^2 = 0.88). This weighted mean age is interpreted to be the crystallizing age of zircons, which can be used to constrain the lower limit age of the volcanic ash. Other 7 analyses constitute another coherent group and yield a weighted mean $^{206}Pb/^{238}U$ age of 250 ± 8 Ma (2σ, X^2 = 0.97), indicating that the saddle shaped spectrum of L3001 was the result of the xenocrystic contamination.

[13] Considering the vastly different isotope system behavior of the zircon and K-feldspar, the congruence between L3001 K-feldspar $^{40}Ar/^{39}Ar$ ages (120.1 ± 0.5 Ma), Lx9 K-feldspar $^{40}Ar/^{39}Ar$ ages (120.3 ± 0.7 Ma) and SHRIMP U-Pb zircon age (124 ± 4 Ma) argues that the xenocrystic contamination of tuff L3001 was not severe, and 120 Ma represents the eruption age of tuffs from the Jiufotang Formation at the Shangheshou section.

3. Discussion and Conclusions

[14] $^{40}Ar/^{39}Ar$ step heating of the K-feldspar from the tuffs of the Shangheshou locality provides the direct age for the fossil-bearing sediments of the Jiufotang Formation. It also represents the first isotopic dating for the Jiufotang Formation in Liaoning Province. The age of the Jiufotang deposits (120.3 ± 0.7 Ma) is about 10 Ma older than the basalt (110.59 ± 0.52 Ma) from the Jiufotang Formation at Tebch, Inner Mongolia, which most likely represents a much later volcanic event. The dating result is consistent with the previous proposal of an Aptian age for the Jiufotang Formation based on recent vertebrate biostratigraphic evidence [*Wang and Zhou*, 2003]. The age of the Jiufotang Formation is only slightly younger than the age of the Yixian Formation, showing that the Jiufotang Formation basically represents a continuation of the lake deposits after the Yixian Formation in this region.

[15] The duration of the whole Jehol sedimentation remains a contentious issue despite published radiometric dating results. Previous estimation of the duration of the Jehol sedimentation between 121 and 114 Ma [*Smith et al.*, 1995] appears a shortened and conservative estimate because much older ages for the lower Yixian Formation (125 Ma at the Sihetun locality) and the basal most Yixian Formation (about 128 Ma at the Lujiatun locality) have been obtained [*Wang et al.*, 2001]. The Jehol Biota was also suggested to have existed for 18 Ma assuming the date of 110 Ma of the intrusive basalt in the Jiufotang Formation as a tentative upper limit of the biota [*Zhou et al.*, 2003]. The $^{40}Ar/^{39}Ar$ date of the Jiufotang Formation, however, casts some doubts on this upper limit of the formation. At the moment, we can probably only conclude that the Jehol sedimentation had a duration of more than 8 Ma (between 128 Ma and 120 Ma) from Barremian to Aptian of the late Early Cretaceous.

[16] Establishing the age of the Jiufotang Formation is critical in evaluating the evolutionary significance of various important vertebrate groups discovered from the Jehol Group. For instance, it bears important implications for the study of the early evolution and differentiation of early birds. It provides the minimum age for truly branched feathered dromaeosaurid dinosaurs [*Xu et al.*, 2003]. It also provides the minimum age for the appearance of the largest Early Cretaceous bird *Sapeornis*, the long-tailed seed-eating bird *Jeholornis*, and the fish eating ornithurine bird *Yanornis*, which possessed nearly modern flight apparatus.

[17] The K-feldspar $^{40}Ar/^{39}Ar$ age (120.3 ± 0.7 Ma) of tuff Lx9 provides an unambiguous age for the fossil-bearing sedimentation of the lacustrine Jiufotang Formation, and corresponds to an early Aptian age of the late Early Cretaceous. This result is consistent with recent biostratigraphic evidence.

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