Dates and rates in ancient lakes: $^{40}Ar-^{39}Ar$ evidence for an Early Cretaceous age for the Jehol Group, northeast China

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Abstract: The correlation of freshwater sediments in small, fault-bound basins in Liaoning Province, northeast China, known as the Jehol (or Rehe) Group, has been a subject of debate for many years, with biochronological estimates ranging from Late Jurassic to the Cretaceous periods. We have applied the laser $^{40}Ar-^{39}Ar$ technique to volcanic intercalations and lacustrine sediments from the Yixian Formation at the base of the Jehol Group. Minerals and whole-rock chips from the upper parts of the Yixian Formation give concordant ages with a mean of 121.1 $\pm$ 0.2 Ma (1σ). Ages for samples near the base of the Yixian Formation give 121.4 $\pm$ 0.6 and 122.9 $\pm$ 0.3 Ma, and appear to be synchronous or only slightly older than the top of the formation. Integrated ages of 122 Ma for glaucony from the lacustrine sediments lying stratigraphically between the upper and lower parts of the Yixian are in very good agreement with the absolute age framework provided by the volcanic units. Such disseminated facies of this clay show promise for directly dating lacustrine sediments. All of the above $^{40}Ar-^{39}Ar$ dates provide an absolute calibration of the Yixian Formation, and show that the whole formation was deposited entirely within Early Cretaceous time over an interval of no more than 2–3 Ma.

Résumé : La corrélation des sédiments d'eau douce dans les petits bassins bordés par des failles de la province de Liaoning, dans le nord-est de la Chine, assignés au Groupe de Jehol (ou de Rehe), a été l'objet de nombreuses discussions durant plusieurs années, avec des estimations biochronologiques variant des périodes Jurassique tardif à Crétacé. Nous avons appliqué la technique laser $^{40}Ar-^{39}Ar$ aux matériaux des intercalations volcaniques et aux sédiments lacustres de la Formation de Yixian, à la base du Groupe de Jehol. Les minéraux et les éclats de roche des portions supérieures de la Formation de Yixian fournissent des âges concordants dont la moyenne est de 121,1 $\pm$ 0,2 Ma (1σ). Les âges obtenus pour les échantillons prélevés près de la base de la Formation de Yixian donnent 121,4 $\pm$ 0,6 et 122,9 $\pm$ 0,3 Ma, étant synchrones ou à peine plus vieux que ceux du sommet de la formation. Les âges intégrés de 122 Ma sur la glauconie des sédiments lacustres, stratigraphiquement localisés entre les parties supérieure et inférieure du Yixian, sont en bon accord avec l'âge absolu que procurent les unités volcaniques. Ainsi, les faciès disséminés de cette argile deviennent potentiellement intéressants pour dater directement les sédiments lacustres. Toutes les dates $^{40}Ar-^{39}Ar$ citées ci-haut autorisent une calibration d'âge absolu pour la Formation de Yixian, et elles démontrent que tous les matériaux de cette formation ont été déposés entièrement durant le Crétacé précoce sur un intervalle n'excédant pas 2–3 Ma.

[Traduit par la rédaction]

Introduction

The most frequently used way to determine the time of the deposition of sedimentary strata is to relate their fossils to a global biochronological record which itself has been calibrated by absolute ages. The application of such an approach relies on the presence of globally ranging index fossils typical of the marine record. However, restricted or nonmarine sediments frequently lack index fossils, and can be difficult to correlate with global chronologies. Although not always possible, the most desirable solution to these correlation problems is to obtain an independent absolute time scale from a sedimentary sequence itself to compare with the international standard.

One such dating problem concerns the age of the late Mesozoic Jehol Group, Liaoning Province, northeast China. This sedimentary sequence is important for several reasons: (*i*) it contains a fertile biological record integral to the understanding of vertebrate evolution, including the origin of

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certain groups of freshwater fish, the extinction and survival of several dinosaurian groups, and the appearance of carinate birds; (*ii*) fossil remains of organisms preserved in the Jehol sequence are widely distributed across China, Mongolia, and Siberia, indicating that the correlation of Jehol strata has broad regional implications; and (*iii*) based on observed similarities and differences of fossil taxa from these and stratigraphically adjacent sediments compared to those found on the other continents, the Jehol Group may record the time of the end of the mid-Mesozoic isolation of the Asian continent (Russell 1993). Endemic taxa evolved during the isolation of Asia have contributed to the difficulties in unambiguously correlating Chinese sedimentary sequences with international scales. Moreover, the sediments were deposited in numerous, isolated, and heavily faulted lacustrine basins. Thus, the endemic nature of the Jehol biota, coupled with its preservation in restricted, small lake basins, has been a source of much uncertainty in assessing the age of the Jehol Group.

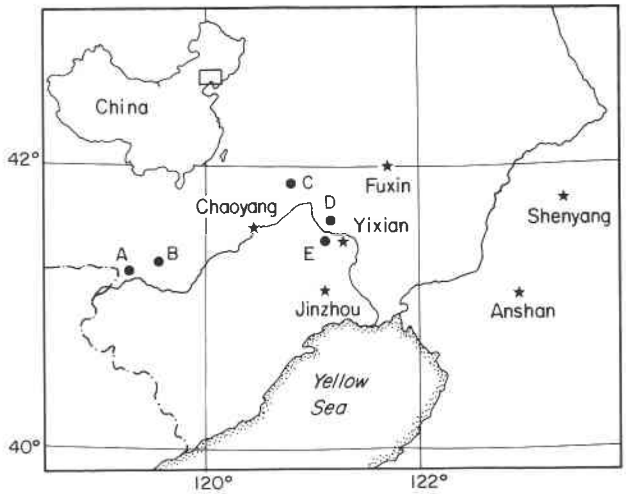
The Jehol Group is often subdivided into four lithostratigraphic units (Yixian, Jiufotang, Shahai, and Fuxin formations) of variable thickness, ranging up to approximately 1500 m (Wang et al. 1990). Biochronological estimates of the age of the sequence have been the subject of controversy and can be grouped into three preferences: Late Jurassic (e.g., bivalves; Yu 1987; Yu et al. 1987); both Jurassic and Early Cretaceous (e.g., conchostracans; Wang 1987); and Early Cretaceous (e.g., palynomorphs; Mao et al. 1990). Available radiometric ages for Jehol strata do not resolve the problem. Published ages for the base of the Yixian Formation, which consist of a series of intercalated sedimentary and igneous horizons (cf. Wang et al. 1990), include 137 ± 7 Ma using K–Ar and 142.5 Ma using Rb–Sr (Wang 1983; Wang and Diao 1984). K–Ar ages for the Fuxin Formation reported in Mao et al. (1990) range from 100 to 137 Ma. These dates suggest that at least the lower part of the Jehol Group is Late Jurassic or Early Cretaceous in age.

Here we report the initial results of a laser $^{40}Ar-^{39}Ar$ study of single mineral grains – rock chips from intercalated volcanic and sedimentary horizons in the Jehol sediments. We focused our attention on the Yixian Formation, to provide a maximum age for the Jehol Group, and thereby constrain the ages of Jehol biota.

Samples and techniques

The samples are derived from many of the historically most important sections in the type region of the Jehol Group, the Lingyuan–Chaoyang–Beipiao and Yixian–Fuxin basins located in Western Liaoning Province (Fig. 1) (Wang et al. 1990). The laser fusion technique at the Toronto laboratory has been described elsewhere (Layer et al. 1987). Radiogenic ^{40}Ar is calculated by assuming that the initial $^{40}Ar/^{36}Ar$ ratios in the Mesozoic were similar to that of modern atmosphere (295.5). For the most chronologically useful gas fractions of the samples (i.e., heating steps >600°C), measured $^{40}Ar/^{36}Ar$ ratios are on the order of $1-3 \times 10^4$. Therefore, the amounts of radiogenic ^{40}Ar determined are relatively insensitive to small departures (i.e., a few percent) from the assumed initial atmospheric value. However, ages from the data have also been derived from isochron plots to test this assumption. In many instances, the fractions corresponding to plateau segments are very uniformly radiogenic. The same fractions show very little spread of points on an isochron plot and consequently yield poorly defined initial ratios. In such cases, if the determined initial $^{40}Ar/^{36}Ar$ ratios are not significantly different than 295.5, we consider the plateau age to represent the best estimate of the age.

Fig. 1. Sample locations (●) of the Yixian Formation in Liaoning Province, northeastern China. A, Ershilipu; B, Daxinfangzi; C, Beipao; D, Zoujiagou; E, Jingangshan.



Results

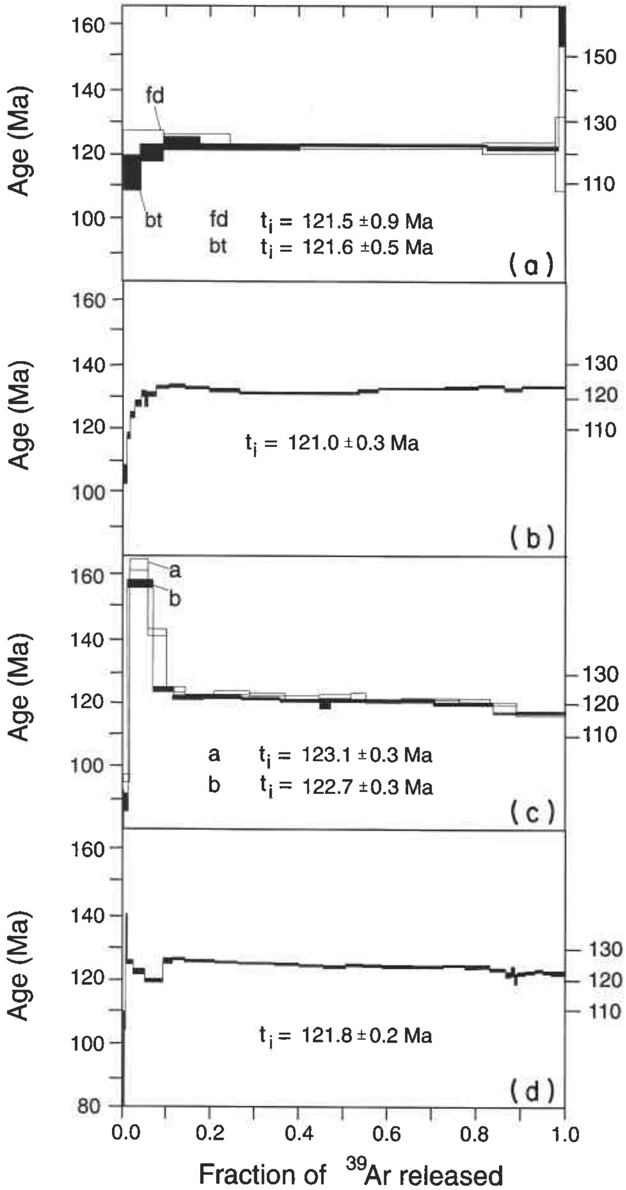
Upper Yixian Formation

The section at Jingangshan represents the fourth intercalation of the Yixian Formation (Wang et al. 1990). Pristine minerals separated from an otherwise altered volcanic breccia (sample 6) give concordant age fractions (Fig. 2*a*). The integrated ages of 121.5 ± 0.9 and 121.6 ± 0.5 Ma for plagioclase and biotite, respectively, are indistinguishable from their respective plateau ages and each other, and effectively date the formation of the breccia (Table 1).

Biotite from a diabase (sample 5) from a slightly lower stratigraphic position shows more internal age discordance in its age spectrum (Fig. 2*b*). This pattern features low but increasing ages for the first 5% of the gas release, giving way to a plateau with a slight sag in the middle. This spectral pattern is very common for biotite, and the age discordance has often been attributed to redistribution of ^{39}Ar due to recoil (e.g., Lo and Onstott 1989). Therefore, considering the spectral discordance, the best approximation to the age of this sample is given by the integrated age of 121.0 ± 0.3 Ma.

Duplicate analyses of rock chips from the diabase show very reproducible gas release patterns and good plateaux. Plateau ages of 123.1 ± 0.3 and 122.7 ± 0.3 Ma are identical within uncertainties (Fig. 2*c*). However, the age spectral shape at low temperature is suggestive of some excess Ar in these samples. It is not surprising, therefore, that isochron treatment of the data for these chips indicates initial isotopic ratios of 346 ± 9 and 333 ± 7, which are significantly higher than those of the modern atmospheric value (Table 1). Therefore, isochron ages of 120.9 ± 0.4 and 120.8 ± 0.4 Ma may provide better age approximations. These ages are significantly lower than the plateau ages; however, they

Fig. 2. $^{40}Ar-^{39}Ar$ age spectra for samples from the Upper Yixian Formation. (*a*) Sample 6 plagioclase (fd) and biotite (bt). (*b*) Biotite from sample 6. (*c*) Diabase (analyzed in duplicate). Integrated ages (t_i) are indicated below spectra. (*d*) Sample 10 basalt. Uncertainties associated with individual fractions are 1σ and do not include the error in *J*.



are in good agreement with the integrated age for the above-mentioned biotite.

A basalt unit (sample 10) occurs about 10 m above the third sedimentary intercalation in the Yixian type section at Zoujiagou. Integrated ages for duplicates giving ages of 120.2 $\pm$ 0.3 and 121.8 $\pm$ 0.2 Ma are slightly variable (Table 1). The age spectrum of a step-heated sample yields a relatively flat plateau over most of the gas release, although there is a slight tilt towards younger ages for higher temperature fractions, rendering the plateau age questionable (Fig. 2*d*). The isochron approach resolves this slight bias and yields indistinguishable ages of 121.3 $\pm$ 2.3 and 121.4 $\pm$ 0.7 Ma for intermediate and higher temperature data, respectively, on separate isochrons (Table 1).

Lower Yixian Formation

Two volcanic horizons constrain the first sedimentary intercalation of the Yixian Formation (Lower Yixian), namely an andesite from the type locality at Daxinfangzi (sample 2), and a basalt from Beipiao (sample 4). Specimens of the andesite give integrated ages of 119 Ma, slightly lower compared with those of the Upper Yixian, suggesting that the samples have undergone some ^{40}Ar loss (Table 1). However, the samples give reproducible age spectra and identical plateau ages for gas fractions representing 40–50% of their total ^{39}Ar volumes (Fig. 3*a*). A weighted mean estimate for the two samples yields an age of 122.9 $\pm$ 0.3 Ma.

The basalt from Beipiao is situated approximately 230 m above the base of the Yixian Formation. Integrated ages from subsamples are significantly higher than those of the andesite, indicating that this rock has undergone less ^{40}Ar loss (Table 1). However, the range of integrated ages from 121 to 123 Ma is over seven times the typical analytical uncertainties and suggests that some samples have suffered small amounts of Ar loss. The age spectrum of one of these replicates features two plateaux at 123 and 121 Ma, corresponding to lower and higher temperatures, respectively (Fig. 3*b*). Higher ages for low- to intermediate-temperature portions relative to the higher temperature portions are common features of the $^{40}Ar-^{39}Ar$ age spectra of basalts. These features have been attributed to ^{39}Ar recoil affecting the phases that degas at low temperatures (e.g., Baksi and Farrar 1991). We are currently investigating possible recoil artifacts in fine-grained rock in detail. However, considering the present status of such age spectra, we regard the higher temperature plateau age of 121.2 $\pm$ 0.3 Ma as a better estimate of the age of this sample.

Ershilipu sediments

White lacustrine sediments at Ershilipu occupy a stratigraphic position between the dated units of the upper and lower parts of the Yixian Formation. Glaucony-bearing sediments were sampled about 100 m above the volcanic rocks associated with the first intercalation. Glaucony facies in these sediments exist as planar disseminated masses located parallel to bedding laminae. Glaucony is composed of very fine crystallites, and a previous $^{39}Ar-^{40}Ar$ study of the pelletoidal variety has shown that it is subject to large-scale redistribution of neutron-generated Ar isotopes, in addition to significant ^{39}Ar recoil loss causing the apparent ages to be too old (Smith et al. 1993). However, the disseminated form of glaucony has not been previously tested using $^{40}Ar-^{39}Ar$, and we are unaware of any K–Ar studies of this habit. Duplicate glaucony samples give indistinguishable integrated ages of 122.1 $\pm$ 0.2 and 122.5 $\pm$ 0.3 Ma (Table 1). These ages are in excellent agreement with the strict chronostratigraphic constraints imposed by the $^{40}Ar-^{39}Ar$ ages of the upper and lower Yixian Formation. Thus, these samples appear to have undergone no significant ^{39}Ar loss. Perhaps, unlike pelletoidal glaucony, these samples lack the connected pathways that facilitate the escape of ^{39}Ar during neutron irradiation.

Table 1. Summary of $^{40}Ar-^{39}Ar$ ages from the Yixian Formation of the Jehol Group, northeastern China.

Type	Sample no.	Figure no.	Sample	Locality	Integrated age (Ma)[a]	Plateau age (Ma)	Percent ^{39}Ar[b]	Isochron age (Ma)	Initial $^{40}Ar/^{36}Ar$[c]	SumS/ $(n-2)$[d]
Upper Yixian Formation										
Basalt	10	—	Rock chip	Zoujiagou	120.2±0.3	One-step fusion				
		2*d*	Rock chip		121.8±0.2	122.8±0.2	43	121.3±2.3	580±440	0.68
								121.4±0.7	230±80	0.72
Volcanic breccia	6	2*a*	Plagioclase	Jingangshan	121.5±0.9	121.5±0.9	100	121.4±1.1	300±60	0.20
		2*a*	Biotite		121.6±0.5	121.5±0.5	90	121.6±0.4	277±120	0.35
Diabase	5	2*b*	Biotite		121.0±0.3					
		2*c*	Rock chip	Jingangshan	123.1±0.3	122.1±0.3	73	120.9±0.4	346±9	1.03
		2*c*	Rock chip		122.7±0.3	122.1±0.3	59	120.8±0.4	333±7	0.52
Ershilipu sediments										
Glaucony		—	Rock chip	Ershilipu	122.1±0.2	One-step fusion				
		3*c*	Rock chip		122.5±0.3					
Lower Yixian Formation										
Andesite	2	3*a*	Rock chip	Daxinfangzi	119.5±0.4	122.5±0.5	42	122.9±0.3	280±60	0.48
		3*a*	Rock chip		119.2±0.2	123.0±0.3	49			
Basalt	4	—	Rock chip	Beipiao	121.8±0.4	One-step fusion				
		—	Rock chip		123.1±0.3	One-step fusion				
		3*b*	Rock chip		120.8±0.3	121.2±0.3	39	121.4±0.6	220±140	0.51

Notes: ^{40}K decay constants: $\lambda_\beta = 4.962 \times 10^{-10}$ year^{-1}, $\lambda_{(e+e)} = 0.581 \times 10^{-10}$ year^{-1}; uncertainties given at 1σ.

[a]Ages calculated using 1.071 Ga monitor Hb−3gr, giving an average J value of 0.0052; data are corrected for mass discrimination, Ca-derived neutron generated Ar isotopes and inlet blanks of 4×10^{-12} to 10^{-11} mL STP ^{40}Ar.

[b]Fraction of ^{39}Ar used in calculation of plateau age.

[c]Ratio derived from isochron.

[d]SumS is the sum of the squares of the deviation from the best-fit line; n is the number of points.

The spectrum of a step-heated sample shows much internal discordance indicative of ^{39}Ar recoil redistribution (Fig. 3*c*). However, unlike the pattern characteristic of pelletoidal glaucony, the older apparent ages (up to 150 Ma) indicative of areas showing net ^{39}Ar loss appear at relatively low-temperature portions of the spectrum. Furthermore, some relatively high-temperature fractions approach 120 Ma, suggesting that some areas of the sample were not grossly affected by ^{39}Ar recoil redistribution (Fig. 3*c*).

Discussion: age of the Yixian Formation

A summary of the age results for the Yixian Formation is shown in Fig. 4. $^{40}Ar-^{39}Ar$ ages of 121.2 ± 0.3 Ma for a basalt from Beipiao and 122.9 ± 0.3 Ma for an andesite from Daxinfangzi provide an absolute calibration for the emplacement of the base of the Yixian Formation. Although both of these units are tied to the first intercalation, their positions relative to each other are uncertain, due to the difficulty in precise correlations between their respective basins. Nonetheless, the ages unambiguously fix the Jehol Group to be no earlier than the Cretaceous. These results confirm the younger Early Cretaceous age estimate for the Jehol Group based on palynomorphs (Mao et al. 1990).

The $^{40}Ar-^{39}Ar$ ages for the upper parts of the Yixian Formation, based on mineral and whole-rock chips, are analytically indistinguishable and give a weighted mean age of 121.1 ± 0.2 Ma. Using the older of the two results from the Lower Yixian, 122.9 Ma, gives a time span of 1.8 ± 0.3 Ma for the entire Yixian.

The dates on the upper and lower parts allow us to estimate the sedimentation rates for the Yixian strata. Using the extremes in the estimated stratigraphic thickness between the first and fourth intercalation, combined with the extreme limits of the corresponding time span estimated for the $^{40}Ar-^{39}Ar$ dated volcanic control points on the intercalations, gives an estimate of 500−2000 m · Ma^{-1} for the average sedimentation rate for the Yixian Formation. This range is higher than 250 m · Ma^{-1} estimated for Mesozoic lacustrine sediments of the Newark Series (Olsen 1986), and more in accordance with the figure of 800 m · Ma^{-1} derived from Pliocene lacustrine sediments of the Hadar Formation (Walter 1994). Evidently, accumulation rates in the Jehol basins were very high.

Although we have not dated the upper formations of the Jehol Group, it is possible to estimate limits to the duration of Jehol sedimentation. Relatively advanced (tricolpate) flowering plants have recently been discovered in the Fuxin Formation, which has led to the suggestion that the unit cannot be older than Aptian (Mao et al. 1990). Therefore, using

Fig. 3. $^{40}Ar-^{39}Ar$ age spectra for samples from the Lower Yixian Formation (*a* and *b*) and Ershilipu sediments (*c*). (*a*) Sample 2 andesite (analyzed in duplicate). (*b*) Sample 4 basalt. (*c*) Disseminated glaucony. Integrated ages (t_i) are indicated below spectra.

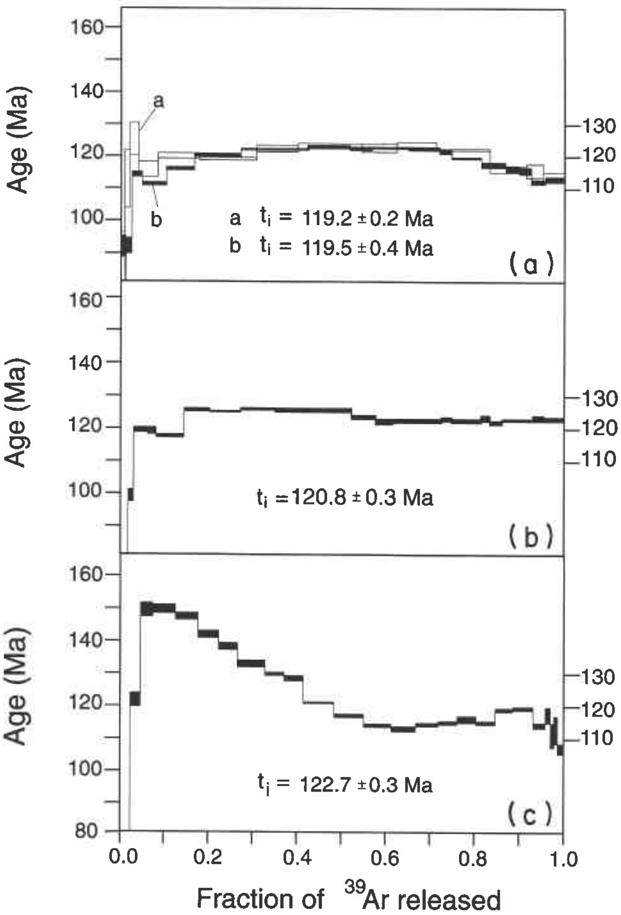
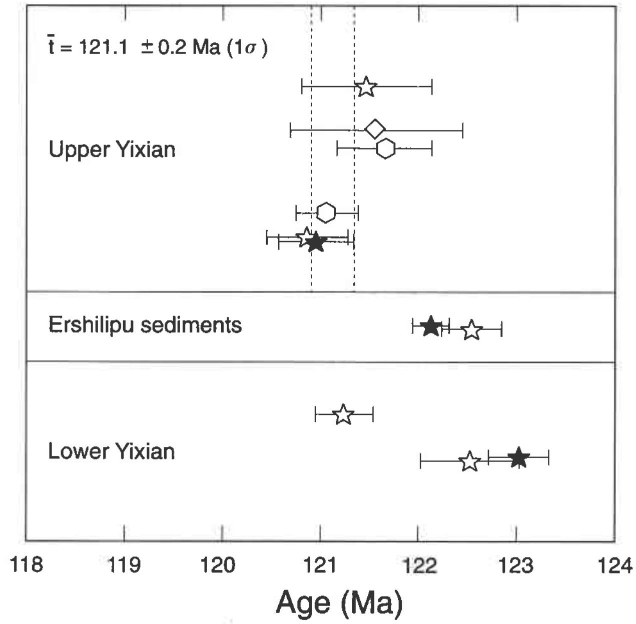


Fig. 4. Summary of preferred $^{39}Ar-^{40}Ar$ ages for the Yixian Formation (see text for discussion). ☆, rock chips (closed symbols indicate duplicate analyses); ◇, plagioclase; ⬡, biotite.



the $^{40}Ar-^{39}Ar$ results and taking into account differences in published absolute time scales for the Aptian, a range from 121 to 114 Ma yields the best estimate for the classic Jehol sedimentation.

Our analyses place important refinements on the ages of fossil groups in the overlying Jiufotang Formation, particularly, the recently described avian *Sinornis santensis* (Sereno and Rao 1992). Compared to the Late Jurassic *Archaeoptrix, Sinornis* shows much more developed skeletal adaptations for sustained flight characteristic of modern birds. Based on the analysis of spore–pollen assemblages, *Sinornis* was suggested to have lived in Valanginian time (Sereno and Rao 1992). However, the $^{40}Ar-^{39}Ar$ age for the Upper Yixian now constrains the form to be no older than 121 Ma, which, using the time scale of Harland et al. (1989), places the fossil three stages younger in Aptian time. Precise quantification of the time interval separating *Archaeoptrix* and *Sinornis* is not possible, because currently Mesozoic biostratigraphies are tied to the absolute scales only at the stage level and the errors on the boundaries of stages are several million years. This underlines the need for more precise ages to calibrate the time scale in order to quantify and assess phylogenetic relationships between taxa widely dispersed in time and space. However, this study shows that the time frame for fossil groups occurring in lake basins can be defined to <1 Ma using $^{40}Ar-^{39}Ar$.

Finally, we note that the very promising results on the disseminated glauconies bring us a step closer to the possibility of obtaining precise direct ages of these sediments. Lacustrine sediments frequently show cyclicity in the Milankovitch frequency band (10^4-10^6 years), which offers the potential to resolve time at least an order of magnitude greater than the minimum uncertainty of about 0.2–0.3 Ma in the $^{40}Ar-^{39}Ar$ ages typical for a Cretaceous rock. However, the assessment of the significance of cycles, particularly in older sediments, currently relies on poorly constrained estimates of extrapolated absolute ages. On the other hand, if successful, a series of $^{40}Ar-^{39}Ar$ glaucony dates may allow the direct calibration of the longer period Milankovitch cycles in such sediments.

Conclusions

The $^{40}Ar-^{40}Ar$ ages obtained in this study provide a considerable improvement in the accuracy of the emplacement of the Yixian Formation. The integrated ages alone, which show only a range of ±4 Ma, represent at least a five-times refinement in dating uncertainty relative to the biochronological estimates of ± 20–30 Ma. The age range of 121.1–122.9 Ma for the Yixian Formation, established more precisely by using plateaux and isochrons, corresponds to Aptian (125–112 Ma) using the time scale of Harland et al. (1989) but may be as early as Hauterivian (122–116 Ma) using the scale of Odin and Odin (1990). Using either time scale the base of the Jehol Group is well into the Cretaceous Period.

Acknowledgments

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