Radiometric Zircon Ages of a Tuff Sample from the Baishantou Member of Wuyun Formation, Jiayin: A Contribution to the Search for the K-T Boundary in Heilongjian River Area, China

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Abstract: The existence of the Cretaceous-Tertiary (K/T) boundary in the non-marine succession is expected at Jiayin in the Heilongjiang River area, China. Zircons from a tuff sample from the Baishantou Member of Wuyun Formation in Jiayin were analyzed by the laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) U-Pb dating and fission-track dating methods. Ages of 64.1±0.7 Ma (U-Pb) and 61.7±1.8 Ma (fission-track dating) were obtained, which allow re-evaluation of a previously reported late Maastrichian age for the tuff layer that was in conflict with the paleontological evidence. These results confirm the Danian age of the section in agreement with the paleontological evidence.

Key words: U-Pb dating, fission-track dating, Baishantou section, K/T boundary, Danian, Heilongjiang River area

1 Introduction

The K/T boundary has been searched for in the Heilongjiang River area of China where Upper Cretaceous to Paleocene successions are distributed. These successions are composed of non-marine deposits, and fluvial plain to lacustrine paleoenvironment were reconstructed (Suzuki et al., 2004, 2010). There is no evidence for the influence of an impact of an asteroid (e. g., Alvarez et al., 1980) in Northeastern Asia and Northern Alaska (Sun et al., 2002, Herman et al., 2009) where the study of flora development during the Cretaceous-Paleogene transitional epoch has been carried out (Herman et al., 2009). Finding the precise K/T boundary is important for studies on the environmental change and evolution of life across the event. The Baishantou section is located on the right bank of the Heilongjiang River, about 7 km northwest of Wuyun Town, Jiayin County of Heilongjian Province, China. The K/T boundary is expected to exist in the section (Chen et al., 2004) and a sensitive high resolution ion micro-probe (SHRIMP) U-Pb zircon age of 66±1 Ma, i.e. an uppermost Cretaceous age was obtained for a tuff in the section (Li et al., 2004). However, recent paleontological research shows that the Baishantou Member is early Paleocene (Danian) in age (Sun et al., 2002, 2004, 2005; Markevich et al., 2006; Sun, 2009). The aim of this investigation is to obtain independent evidence for the age of the Baishantou Member using two radiometric dating methods, the laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) U-Pb-dating and the fission-track method.

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2 Geology of the Sampling Site

Late Cretaceous to early Paleocene successions are distributed in the Heilongjiang (Amur) River area on both sides of the Chinese-Russian border. On the Chinese side, the successions are subdivided into five formations; Yong'ancun-, Taipinglinchang-, Yuliangzi-, Furao- and

Table 1 Stratigraphy of the Upper Cretaceous to Paleocene of the Jiayin area, Heilongjiang

Chronological Stratigraphy			Jiayin of Heilongjiang, China	
P	Selandian		Wuyun Fm.	Coal-bearing Member
	Danian			Baishantou Member
K	Maastrichtian	U	Furao Fm.	
		M	Yuliangzi Fm.	
		L		
	Campanian Santonian		Taipinlinchang Fm.	
	Coniacian Turonian		Yong'ancun Fm.	

K: Cretaceous; P: Paleocene.

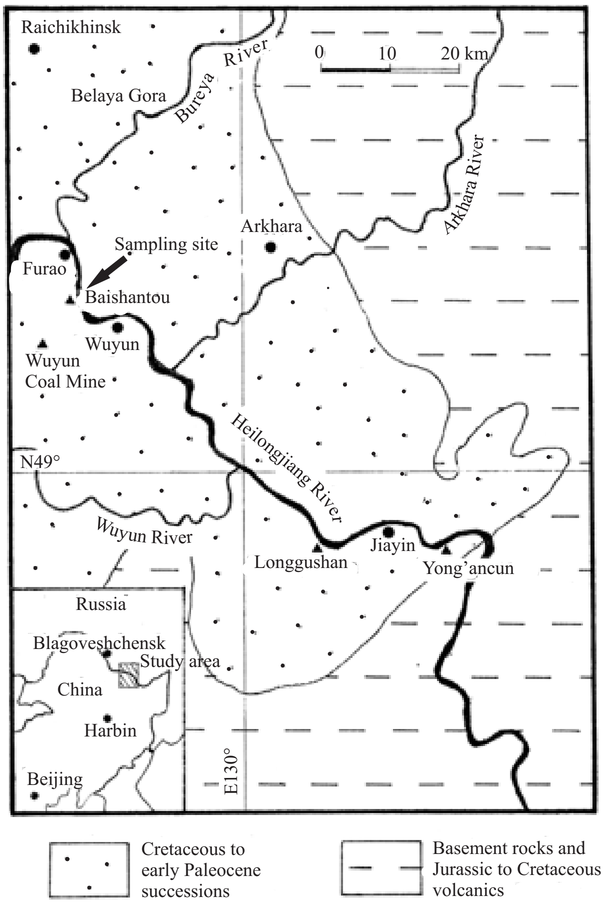


Fig. 1. Map showing the geographic locality of the sampling site.

Wuyun Formations in ascending order(Sun et al., 2007; Quan and Sun, 2008). The Yuliangzi Formation is characterized by abundant dinosaur remains and is dated to be of early to middle Maastrichtian age (Sun et al., 2002; Markevich et al., 2006). The Wuyun Formation yields Paleocene flora, and is subdivided into the Baishantou Member and the Coal-bearing Member. The Danian assemblage of the Wuyun flora, found from the Baishantou Member, is characterized by *Tiliaephyllum tsagajanicum* (Sun et al., 2002, 2005). Thus, the K/T boundary is expected to be located at the bottom of the Baishantou Member at the boundary between Furao and Wuyun Formations (Table 1).

The analyzed sample (BST05) was obtained from the lower part of the Baishantou section (49º17.2′N, 129º32.7′ E) (Fig. 1). The section comprises conglomerate, sandstone, mudstone, coal and rhyolitic tuff deposited in a fluvial environment (Sun et al., 2002, Suzuki et al. 2010). The succession of the section is subdivided into 10 beds from No. 1 to 10 based on their lithofacies (Sun et al., 2002) (Fig. 2). The Danian plants characterized by *Tiliaephyllum tsagajanicum* were collected from the No. 7 bed. The palynological samples indicating the Danian age were collected from beds 1, 3 and 4. Insect fossils are obtained from the No. 4 bed. The dated sample was collected from the rhyolitic tuff (bed No. 2) which is 90 cm thick.

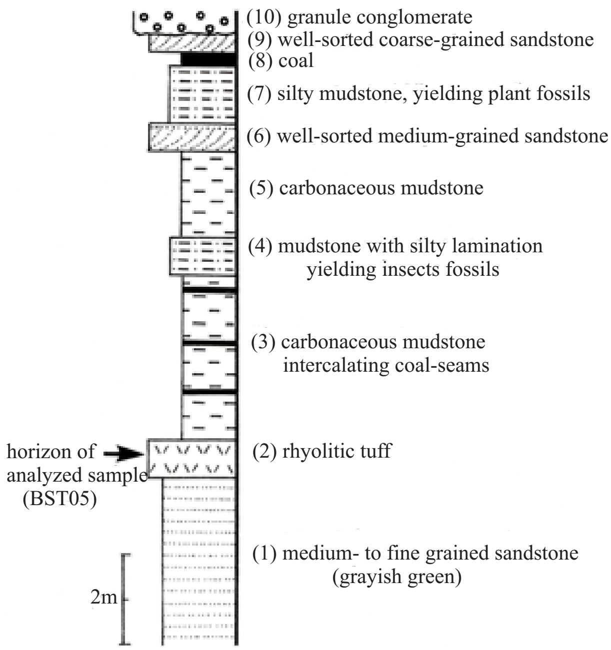


Fig. 2. Lithostratigraphic column of the Baishantou Member of the Wuyun-Formation at the Baishantou section (after Sun et al., 2002).

3 Analyzed Materials and Methods

The dated tuff is a fine-grained rhyolitic crystal tuff, which comprises quartz, feldspar and glass fragments within a very fine grained glassy matrix. Well preserved glasses of platy shards and cuspate shards (Fisher and Schmincke, 1984) are observed under the microscope (Fig. 3). Accessory minerals are zircon and opaque minerals.

Three hundred grams of the tuff contain about 2000 grains of zircon crystals, most of which are euhedral with few inclusions. The zircon assemblage is homogeneous in color and crystal habit, indicating that most crystals are essential. The zircons turned out to be just large enough for analysis with cross-sections measuring about 50 μm.

Two different dating methods were used; the LA-ICP-MS U-Pb method and the fission-track method (FTD). For the LA-ICP-MS method, the zircons were dated using an Agilent 7500s quadrupole ICP-MS instrument equipped with a New Wave UP213 laser ablation system at the Department of Geosciences of the National Taiwan University. The laser beam had a diameter of about 30 μm (see pits in Fig. 4, grain 1). The system was calibrated using the GJ-1 zircon, whereas standard zircon 91500 and Australian Mud Tank Carbonatite zircon (MT) were used for data quality control. In addition, a ca 48 Ma zircon was used as a tertiary (in-house) standard for data quality

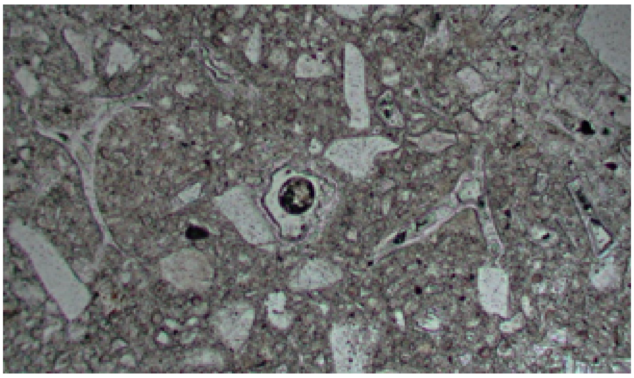


Fig. 3. Photomicrograph of rhyolitic bubble junction shards and bubble wall shards with crystal fragment (BST05). The photo is 1 mm in width.

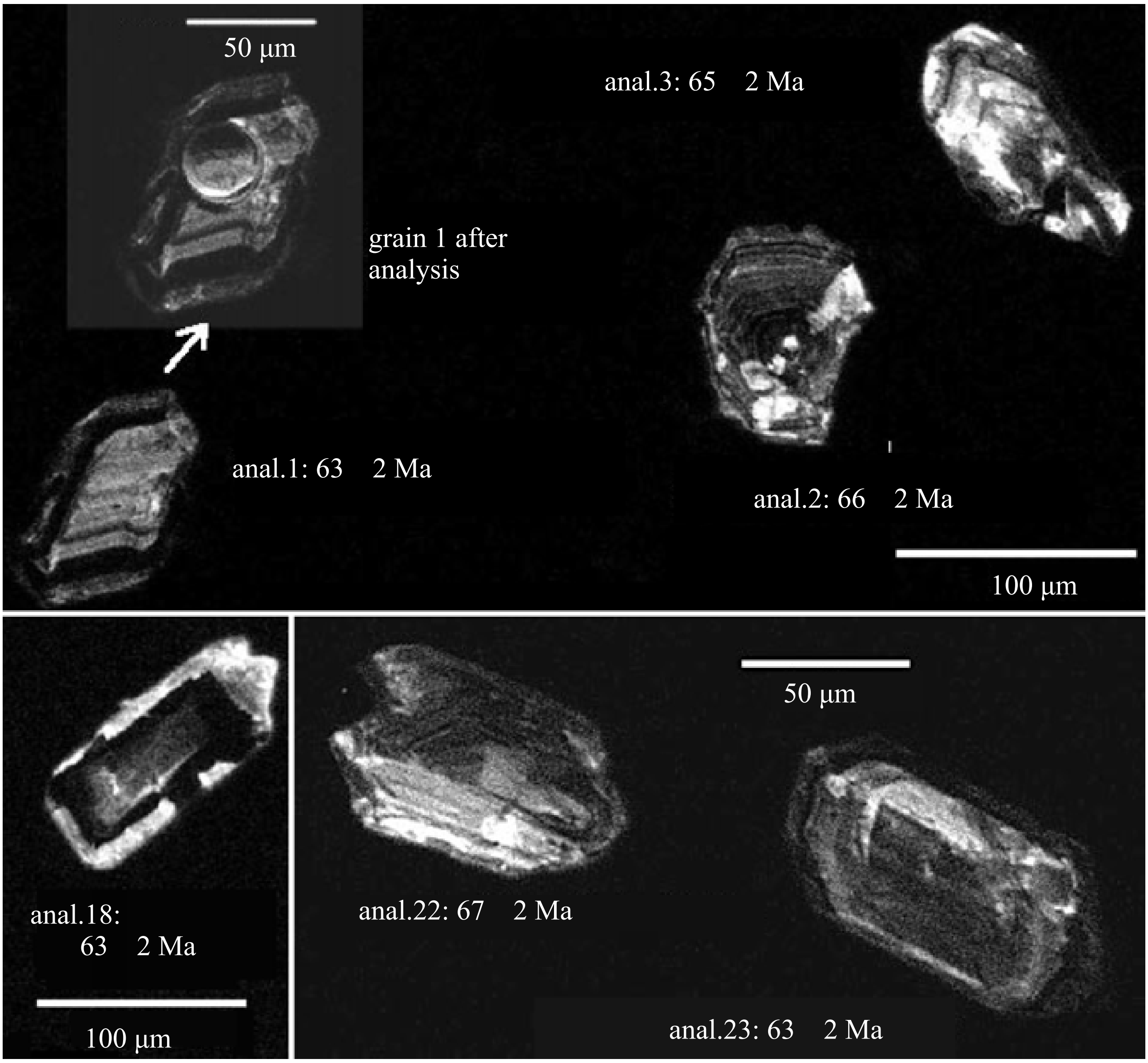


Fig. 4. Cathodoluminiscence (CL) pictures of ca. 64 Ma zircons in sample BST 05. All grains exhibit zoning patterns typical for igneous zircons. For grain 1, pictures are shown before and after analysis to illustrate the size of the pit excavated by the laser beam.

control. Additional details of the analytical procedures are given in Chiu et al. (2009). All U-Th-Pb isotope ratios were calculated using the GLITTER 4.0 (GEMOC) software, and common lead was corrected using the common lead correction function proposed by Anderson (2002). The weighted mean U-Pb ages were calculated and concordia plots drawn using Isoplot v. 3.0 (Ludwig, 2003).

The fission-track dating was performed using the conventional external detector method that applies to an internal surface of zircon (ED1, Danhara et al., 1991, 2003). The fission-track age was calibrated based on the zeta age calibration approach (Hurford, 1990), and examined to see if any additional uncertainty, other than that allowed by Poisson variation in track counts using the χ^2-test (Galbraith, 1981). To estimate potential thermal annealing effects on the fission-track age, the confined-fission-track lengths in the dated zircons were measured and compared with a standard sample.

4 Results

4.1 U-Pb dating using the LA-ICP-MS method

Sixteen out of 23 grains analyzed for BST05 gave ages in the range of 61–70 Ma (Table 2). Excluding the extreme values (61±1 and 70±2 Ma), the remaining 14 analyses define an average of 64.1±0.7 Ma with a mean standard weighted deviation (MSWD) of 0.71 (Fig. 4 and 5), which indeed suggests that the data defines the age of a single event. The average age calculated for all 16 61–70 Ma zircons (63.9±1.0 Ma) does not differ significantly from that value, but the the MSWD increases to 1.9 casting doubt on the significance of this figure. In cathodoluminiscence (CL) pictures, these grains exhibit various oscillatory zoning patterns, typical for igneous zircons (Fig. 4). No obvious evidence for old, inherited cores was seen.

Four analyses were discarded due to the fact that the data were inhomogeneous, i.e., the isotopic ratios and hence, also the calculated ages, varied during the run, probably because the laser beam excavated core/rim zones. Ages obtained for these grains were 64±1, 70±2, 72±2 and 90±2 Ma suggesting that some of these grains contain perhaps older components, though faint and equivocal evidence for a core is seen only in the CL picture of grain 12.

Three grains yielded nearly concordant ages of 105±2,

Table 2 U-Pb isotope data and calculated ages for sample MIN-20B. Only $^{206}Pb/^{238}U$ ages are reported here due to the large errors attached to $^{207}Pb/^{206}Pb$ ages for zircons younger than ca. 1 Ga.

	Corrected isotopic ratios					
	$^{207}Pb/^{235}U$	$\pm\sigma$	$^{206}Pb/^{238}U$	$\pm\sigma$	$^{206}Pb/^{238}U$ age (Ma)	σ
BST05-01	0,06280	0,00466	0,00988	0,00026	63	2
BST05-02	0,06555	0,00245	0,01032	0,00024	66	2
BST05-03	0,06585	0,00571	0,01014	0,00027	65	2
BST05-04	0,06687	0,00445	0,01009	0,00025	65	2
BST05-05	0,07604	0,00970	0,01038	0,00032	67	2
BST05-06**	0,65401	0,01487	0,08120	0,00177	503	11
BST05-07**	0,10429	0,00545	0,01641	0,00036	105	2
BST05-08	0,06067	0,00232	0,00955	0,00020	61	1
BST05-09*	0,14548	0,00318	0,01124	0,00024	72	2
BST05-10	0,06426	0,00440	0,00996	0,00023	64	1
BST05-11	0,07069	0,00204	0,00998	0,00022	64	1
BST05-12*	0,09599	0,00214	0,01095	0,00024	70	2
BST05-14*	0,09669	0,00298	0,00998	0,00022	64	1
BST05-15	0,06287	0,00293	0,00990	0,00023	63	1
BST05-16	0,06686	0,00209	0,00982	0,00022	63	1
BST05-17**	0,30422	0,00741	0,04361	0,00094	275	6
BST05-18	0,06923	0,00664	0,00989	0,00024	63	2
BST05-19*	0,50658	0,01153	0,01404	0,00031	90	2
BST05-20	0,06495	0,00431	0,01022	0,00026	66	2
BST05-21	0,07132	0,00526	0,01098	0,00028	70	2
BST05-22	0,06909	0,00319	0,01051	0,00024	67	2
BST05-23	0,06210	0,00522	0,00977	0,00024	63	2
BST05-24	0,07154	0,00351	0,01013	0,00024	65	2

* unstable run; ** xenocryst

Table 3 Analytical results of fission-track dating on zircon samples

			Spontaneous		Induced		Dosimeter					
Sample	method	n	ρ_s ($10^6/cm^2$)	*Ns*	ρ_i ($10^6/cm^2$)	*Ni*	ρ_d ($10^4/cm^2$)	*Nd*	r	$Pr(x^2)$ (%)	U content (ppm)	Age (±1σ) (Ma)
BST05	ED1	80	8.55	9761	2.09	2388	7.987	4089	0.164	15	250	61.7±1.8

Note: n is number of grains analyzed. ρ and *N* are the density and the total number of fissiontrack tracks counted, respectively. Analyses were made by th external detector method that applied to internal surfaces of zircon (ED1). Ages were calculated using a dosimeter glass NIST-SRM612 and a zeta calibratio factor of 380±3 for HI (Danhara et al., 2003). Thermal neutrons were irradiated in the pneumatic tube of JRR-4 reactor unit of the Japan Atomic Energ Agency, Japan. r is correlation coefficient between ρs and ρi. $Pr(x^2)$ is probability of obtaining the x^2 value for ν degrees of freedom (ν = number of cristals - 1 Galbraith, 1981). Uranium content was calculated based on the induced fission track densities (Iwano et al., 2000).

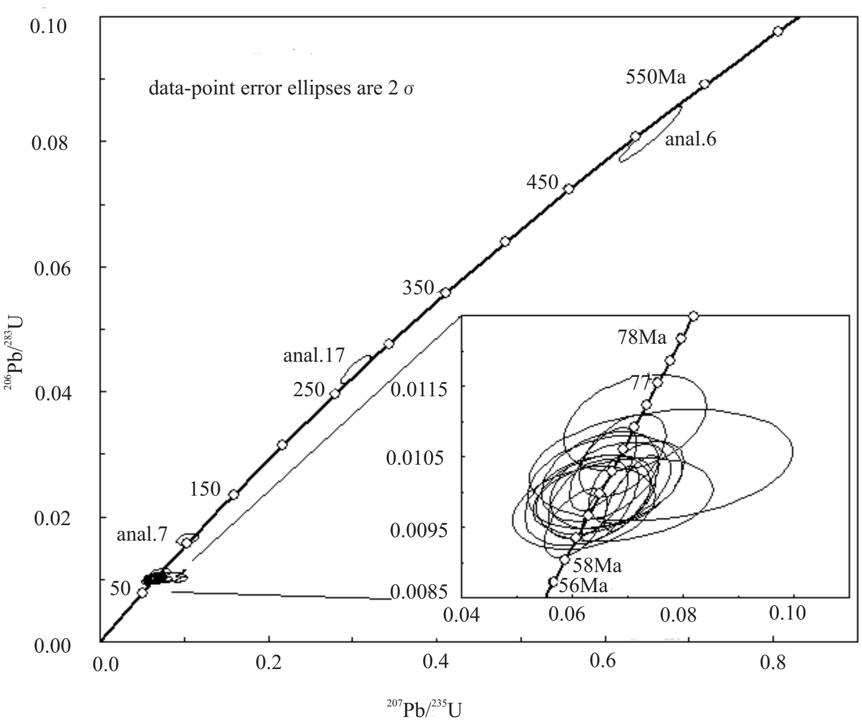


Fig. 5. Concordia diagram for zircons of sample BST05. Inset shows data for ca. 64 Ma zircons. Error ellipses for old zircons are labeled.

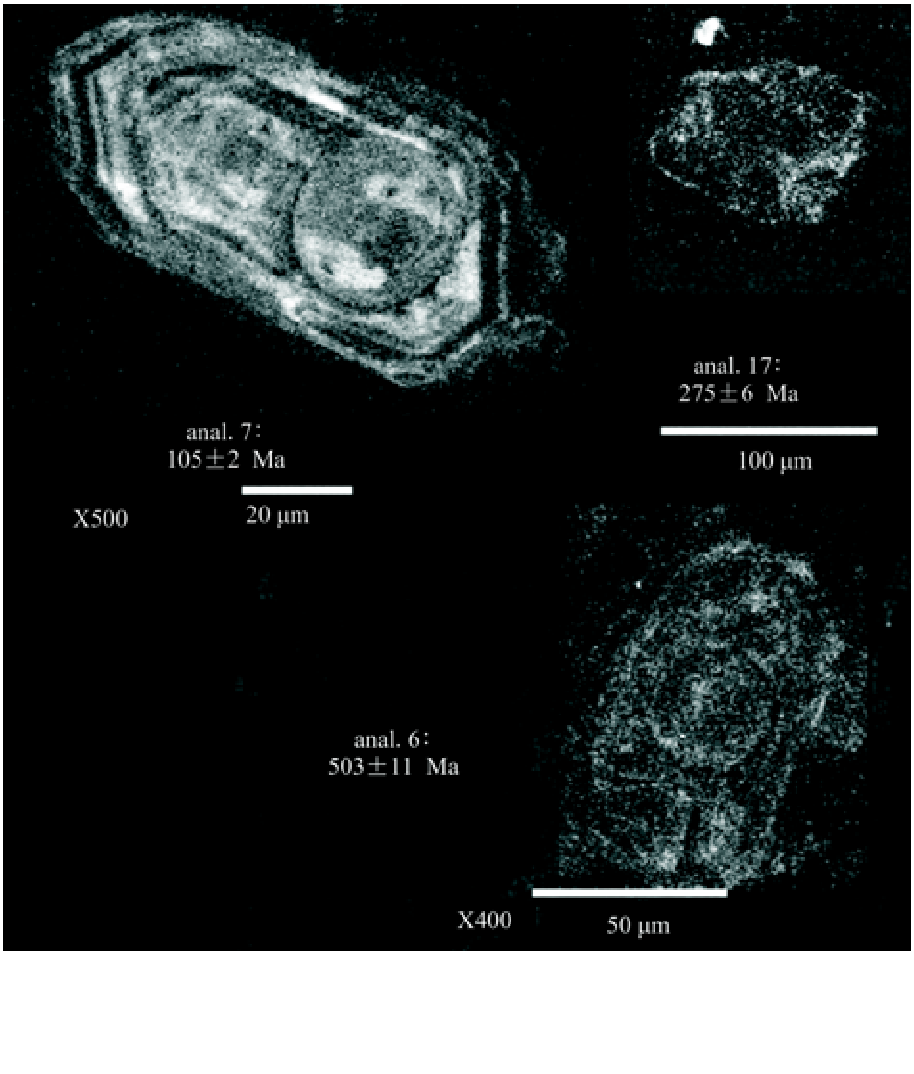


Fig. 6. Cathodoluminiscence (CL) pictures of old zircons. Pictures for zircons 6 and 7 were taken after analysis. The CL was so low for all grains that extreme contrast enhancement was required to show internal features, hence the 'coarse' appearance of the photos.

275±6, and 503±11 Ma and obviously represent xenocrysts (Fig. 5). These zircons appear very dark in CL pictures. Photo processing reveals igneous zoning in grain 7 and perhaps also in grain 6, whereas grain 17 is featureless (Fig. 6).

4.2 Fission-track method

Eighty zircon grains from the same mineral separate as used for ICP-MS U-Pb analysis were analyzed. Though individual grain data scatter from 35 Ma to 100 Ma because of larger errors of grain age, they passed the χ^2-test at the 5% significant confidence level (Fig. 7). Therefore, these belong to a single age population, and give a pooled age of 61.7±1.8 (1 sigma) Ma (Table 3). The average track length is as long as in the standard (Fish Canyon Tuff), which is not affected by thermal disturbance (Fig. 8). This suggests that the sample BST05 has not been disturbed by thermal events after the deposition and hence the age obtained should reflect the age of magmatism. The fission-track ages of the xenocrysts with older U-Pb ages were reset by the final magmatic event.

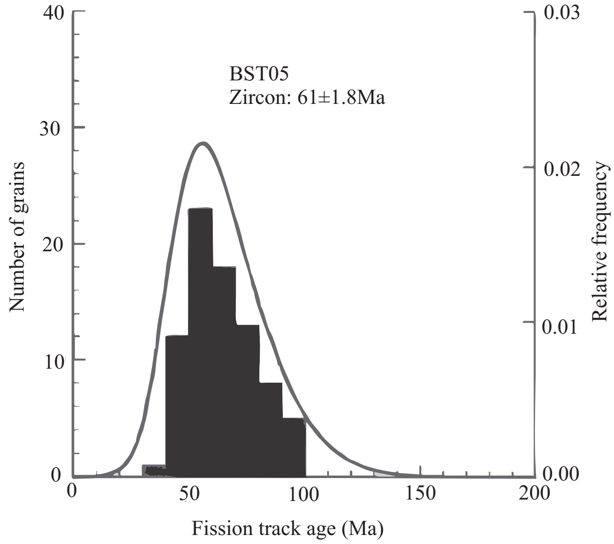
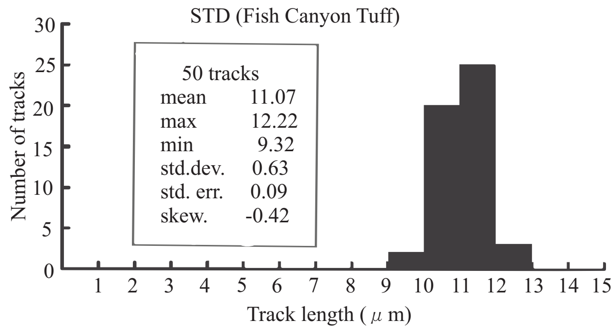


Fig. 7. Composite probability density plot and grain histogram of the single-grain age result of the tuff dated by fission track method.



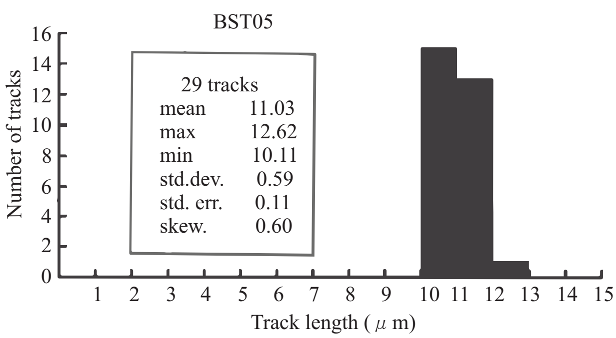


Fig. 8. Comparison of confined track length between BST05 and a standard sample (Fish Canyon Tuff).

5 Discussion and Conclusion

Chen et al. (2004) proposed that the K/T boundary is located in the upper part of the Beishantou section (at the bottom of bed 10 of Fig. 2). This opinion was based mainly upon the U-Pb zircon age of 66±1 Ma obtained with the SHRIMP for the rhyolite tiff in the lower part of the section (bed 2 of Fig. 2) (Li et al., 2004).

However, detailed paleobotanical study of the rich plant fossils from the section (bed 7 of Fig. 2) indicates the plants are Danian in age. In particular the discovery of the typical Danian taxon *Tiliaephyllum tsagajanicum* supports this conclusion (Sun et al., 2002, 2004, 2005). The palynological analysis (beds 1, 3 and 4 of Fig. 2) shows a *Triatriopollenites confuses – Aquilapollenites spinulosus* assemblage, consisting of *Aquilapollenites spinulosus, A. subtilis, Triatriopollenites confuses, T. plictosus*, *Myricapollenites* sp., *Alnipollenites* sp., etc., which are clear evidence for the Danian age of the section. This palynomorphic assemblage is comparable to those from the upper part of Tsagayan stratotype section of the Belaya Hill, and the lower part of the Arkhara-Boguchan section on the left bank of Amur (Heilongjiang) River; to the upper part of the Rarytkin Formation from the Anadyr' River Basin, and the upper part of the Chukotka Formation from the Ugol'naya River Basin (NE Russia), which are all of Danian age (Markevich et al., 2006).

The ages we determined for the rhyolite tuff (bed 2 in Fig. 2), namely 64.1±0.7 Ma (U-Pb zircon age) and 61.7±1.8 Ma (FTD zircon age) are compatible with the paleontological evidence. The statistical error attached to the fission-track age is larger than that of the U-Pb age. Both errors overlap at 63.4–63.5 Ma and this age might be the best age estimate at present (Fig. 9). The previously reported SHRIMP zircon U-Pb-age of 66 ± 1 Ma (Li et al., 2004) is significantly older. However, these authors already considered the possibility that some of their

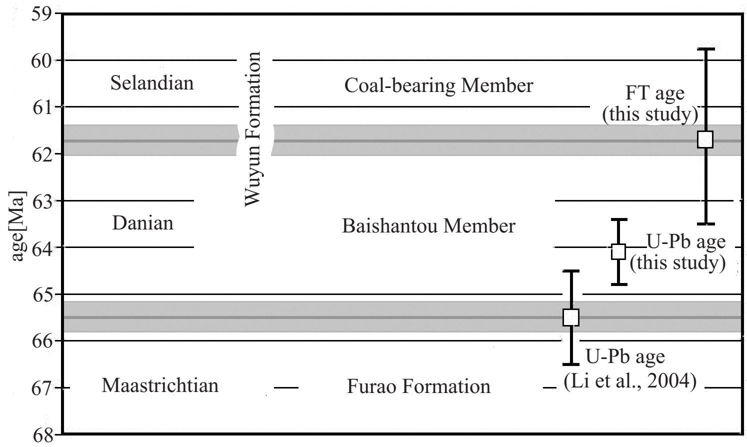


Fig. 9. Comparison among the results from sensitive high resolution ion micro-probe (SHRIMP) U-Pb method, inductively coupled plasma mass spectrometry (ICP-MS) method and the fission-track method.

analyses are biased by some older, inherited components. This is supported by the high MSWD value of 1.4 attached to their 66.2±1.2 Ma age, which already suggests a slightly disturbed system. Omitting three analyses (71.3±2.7, 72.9±2.3, and 71.8±3.9 Ma), two of which are distinctly discordant and the third one having a very large statistical error (Li et al., 2004, Fig. 2), Li et al. (2004) calculate an age of 65.5±1.0 Ma with a MSWD value of 0.63. While Li et al. (2004) considered the fact, that the ages they calculated including or excluding these analyses are identical within uncertainties to suggest a preferred age of 66±1 Ma, we prefer their younger age because of the MSWD<1, the observation that we obtained similar ages >70 Ma in unstable runs and the fact that our study confirmed the presence of old inherited zircon cores, which justifies the exclusion of slightly older ages. This 65.5 ± 1.0 Ma age is virtually identical to our U-Pb age (64.1±0.7 Ma). Both datasets may be slightly biased towards older ages due to the presence of inherited zircon cores, which may not be visible in CL pictures, but the low MSWD values indicate that such a shift, if indeed present, must be very small. Nevertheless, such a small shift could explain the somewhat younger FTD age of 61.7±1.8 Ma. Hence our best estimate for the age of the dated tuff bed of the Baishantou section is 63–64 Ma, which agrees with the latest paleontological and palynological results obtained by Sun et al. (2005) and Markevich et al. (2006).

It might be of interest to note that a similar case occurred in the horizons just above the K-T boundary in the Denver Basin of the United States where at least three samples were dated to be older than 65.5 Ma (personal communications by Dr. K. Johnson in 2008). Thus, it appears that great care needs to be exercised even in the interpretation of U-Pb zircon ages, even where these were obtained by spot analyses.

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