**The earliest evidence of hominid settlement in China:
combined electron spin resonance and uranium series (ESR/U-series) dating of
mammalian fossil teeth from Longgupo cave**

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Abstract

Longgupo cave, located in Chongqing Municipality, China, was discovered in 1984. Sixteen *Gigantopithecus* teeth and two hominid fossils accompanied with abundant mammalian fossils and stone artifacts were unearthed from the site. Previous dating result is questioned because of the complexity of deposition history. In this study, seventeen mammalian fossil teeth collected from different layers of unit C II and C III (C III') during the 2003-2006 Sino-Franco joint excavation were analyzed by combined ESR and U-series methods, and calculated with a US-ESR model. *In situ* dose rates were remeasured in detail in 2012, in order to refine the external dose rate determination. Uranium-series analyses indicate that no obvious uranium leaching has occurred, and all the teeth underwent a very recent uranium uptake history, except one from layer C III 3 of the south wall. The US-ESR results show that the ages of ten teeth from unit C III' of the north wall are consistent in general, about 2.35 Ma. The ages of seven samples from south wall are not in accordance with the stratigraphic order: three teeth are much younger than the other four, probably attributed to the relative higher uranium concentration and U/Th ratio in the dental tissues and significant higher *in situ* gamma dose rate. Nevertheless, the three teeth from C III of the south wall give an average age of ~ 2.48 Ma. The fossil ages obtained in this work combined with paleontological evidence and new paleomagnetic results place Longgupo at the very beginning of the Early Pleistocene. Compared with other early hominid settlements, Longgupo is one of the earliest evidences of hominid settlement in China and East Asia to date.

Keywords: Longgupo site, Hominid, China, ESR/U-series method, Fossil teeth, Early Pleistocene

1. Introduction

Longgupo cave, located in Chongqing Municipality, China (Figure 1), was discovered in 1984. A number of mammalian fossils and stone artifacts were unearthed from the site during several excavation campaigns (1984-1988, Huang and Fang, 1991; 1997-1998, Hou *et al.*, 1999; 2003-2006, Boëda *et al.*, 2011a). Sixteen *Gigantopithecus*

teeth and two hominid fossils, including one fragmental mandible with two teeth and an upper incisor, have attracted increasing attention since their discovery, and distinguished the site as among the earliest evidence of hominid settlement in China. The specimen from Longgupo was initially identified as a new subspecies of *Homo erectus* (*Homo erectus wushanensis*) (Huang and Fang, 1991), and later was described as having affinities with African *Homo habilis* and *Homo ergaster* (Huang *et al.*, 1995). However, this viewpoint is not widely accepted, and some researchers prefer to consider the specimen from Longgupo site as an ape instead of *Homo*, and probably the descendants of *Lufengpithecus* (Schwartz and Tattersall, 1996; Wu, 2000; Ciochon, 2009; Etler, 2009). Although the controversy continues, the evidence of *Gigantopithecus* teeth and stone artifacts on the site is still interesting, as it could provide the first indication of non-hominin tool-using apes in East Asia (Dennell, 2009). The stratigraphic sequence of Longgupo site was initially subdivided into twenty horizontal levels (1 m thickness for each level) (Huang and Fang, 1991). During the Sino-Franco joint excavation between 2003 and 2006 (Figure 2), the stratigraphy of Longgupo was reinvestigated and nomenclature defined in detail based on field observation and measurement (Boëda *et al.*, 2011b; Rasse *et al.*, 2011). The stratigraphy along the north and south walls were described by Rasse *et al.* (2011) and could be correlated with the sequence of Huang and Fang (1991) in general (Figure 3). The site was once a cave initially formed by fluvial cutting and erosion, and filled with fluvial deposits during the Plio-Pleistocene. The lowest layer, C III 10, is comprised of dark clays (levels 20-13). The overlying layers in complexes C III (south wall)/III' (north wall) mainly consist of coarse fluvial sediments (levels 12-6). The upper units C II (levels 5-2) and C I (level 1) are composed of sandy clay and fallen breccias, deposited until complete filling of the cave. The cave was then carpeted by collapsed coarse breccias along the slope during middle and late Pleistocene. As the bottom of the section was not exposed at the beginning of the field work, it was denominated as C III for the stratigraphic unit recovered from the south wall, and as C III' for the north wall. The multiple layers in the two units could be correlated in general, so they were considered as contemporary (Boëda *et al.*, 2011b).

More than 1000 stone artifacts were recovered from 27 cultural layers during 2003-2006

(Boeda *et al.*, 2011c), and the stone artifacts unearthed from 1984 to 2012 which are termed the “Longgupo culture” were described in detail (Hou *et al.*, 1999; Wei *et al.*, 2014). Representative fauna in different geological units divided in Huang and Fang (1991) are listed in Figure 3. The co-occurrence of *Sinomastodon*, *Nestoritherium*, *Equus yunnanensis*, *Ailuropoda microta* and the rodent *Mimomys peii* suggest a time range of late Pliocene to early Pleistocene of the middle unit (Huang *et al.*, 1995). The first paleomagnetic and electron spin resonance (ESR) dating studies placed the site as early as 1.9 Ma. Using the LU (linear uptake)-ESR age of a bovid tooth from level 4, Huang *et al.* (1995) assigned the magnetically normal hominin-bearing levels 7-8 (approximately corresponding to new stratigraphic layers C III 2 and C III 3) to the Olduvai subchron. However, this result is questioned because of the complexity of the depositional history of the cave sedimentary sequence (Zhu *et al.*, 2003).

Preliminary ESR/U-series results were presented, based on seven tooth samples from different stratigraphic layers (Han *et al.*, 2012). Six of seven analyzed teeth collected from C III – C III’ unit give an age from 1.4 to 1.8 Ma, somewhat younger than the expected biostratigraphic age (e.g. Huang and Fang, 1991). In order to complete and refine the preliminary US-ESR age results of Longgupo site, a combined ESR/U-series method has been used to establish a detailed chronological framework of the Longgupo site. Ten additional tooth samples from different layers were analyzed in this study. This will better constraining the chronology of the sedimentary sequence of Longgupo.

2. Method and materials

Seventeen fossil teeth analyzed in this study were sampled during the Sino-Franco excavation between 2003 and 2006. Sixteen samples were collected from seven layers in unit C III (C III’), and one from unit C II 2 of the south wall (Figure 3). Most of the samples are herbivorous teeth, except LGP06S03 (Suid), LGP06N07 (Suid) and LGP06N08 (*Hyena*). Detailed information of the analyzed tooth samples in this study is shown in Table 1.

The combined electron spin resonance and uranium series (ESR/U-series) dating method is a numerical archaeological dating method which could date mammalian tooth fossils directly, throughout reconstruction of the uranium uptake processes in the dental

tissues, and more objectively than previous ESR models which were based on assumed U-uptake histories. This combined ESR/U-series dating technique has been successfully applied on several early hominid sites in Europe and Africa, including Early Pleistocene sites (Falguères *et al.*, 1999; Curnoe *et al.*, 2001; Bahain *et al.*, 2007; Duval *et al.*, 2012a). Dating fossil teeth by ESR is rather complex because of the open system behavior of uranium. In the early ESR studies, several uranium uptake models were proposed by different scholars. Early uranium uptake (EU) model (U-accumulation shortly after tooth burial) and linear uranium uptake (LU) model (continuous U-accumulation) were the two most commonly used U-uptake models (Bischoff and Rosenbauer, 1981; Ikeya, 1982). However, these two could not describe the U-uptake process faithfully, assuming the same kinetics for all the tissues of a tooth. In order to better describe the uranium uptake history into dental tissues and reconstruct the internal dose rate of a fossil tooth, U-series analysis must be combined with ESR measurement. Grün *et al.* (1988) proposed a function to describe the uranium uptake behavior by introducing an uptake parameter p (US-ESR model): $U(t) = U_0 (t/T)^{p+1}$, where $U(t)$ is the U-content at the time t , U_0 is the present U-content, and T is the sample age. The US-ESR model allows the calculation of p -values for each dental tissue from its specific isotopic data, but it is restricted to $p \geq -1$, i.e. U-loss could not be modeled (Grün, 2009). In the EU case, $p = -1$; in the LU one: $p = 0$, and $p > 0$ indicate RU (recent uptake).

The fossil teeth collected from the site present only enamel and dentine tissues, and thus “sediment-enamel-dentine” geometry was applied for age calculation. The two dental tissues were separated mechanically with dentist tools. The enamel was ground into fine powder, sieved (100-200 μm) and prepared into several aliquots for gamma irradiation. The uranium concentration and isotopic ratios of each dental tissue were analyzed by alpha spectrometer. Radon loss was calculated from the combined gamma and alpha-spectrometer measurement (Bahain *et al.*, 1992).

The ESR intensity was measured by peak-to-peak amplitudes (T1-B2) of the ESR signal of enamel (Grün, 2000). The equivalent doses (D_E) were determined by double saturation exponential (DSE) fitting function using Origin 8.0 software, with weighting by $1/I^2$ (Figure 4). Previous study shows that DSE function often better describes the analytical data points than does the conventional single saturation exponential (SSE)

function, especially for early Pleistocene samples (Duval *et al.*, 2009, Han *et al.*, 2011), and it could indicate the presence in tooth enamel of two distinct CO_2^- species with different thermal stabilities and saturation behaviors (Grün *et al.*, 2008). In this study, the error of D_E values in average determined by DSE function is about 5%.

As *in situ* dose rate measurements were only available for a few layers of the site in Han *et al.* (2012), the external dose rate for some layers were obtained by laboratory gamma spectrometry measurement of the sediments associated with tooth samples. However, Longgupo is a typical “lumpy site” where sediments are composed of clay and pebble mixtures, and *in situ* gamma dose rate measurement is highly advisable. The first *in situ* gamma dose rate measurement was carried out in 2003-2006, but only three gamma dose values were available. Revisiting the site in 2012 allowed us to access five different layers (C III-2, 5, 6, 7, 8) in C III unit and make a detailed gamma survey of the site (Figure 2). The measurement was carried out using a Canberra-1000 gamma spectrometer with NaI detector using the threshold technique (Mercier and Falguères, 2007). We carried out supplementary *in situ* gamma dose rate measurements using the same technique in 2012 for all the samples.

3. Results and discussion

The analyzed $^{230}\text{Th}/^{234}\text{U}$ activity ratios of Longgupo samples are shown in Table 2 and Figure 5. The uranium concentration ranges from 0.32 to 9.14 ppm for enamel and from 21.08 to 105.48 ppm for dentine tissue. The $^{230}\text{Th}/^{234}\text{U}$ activity ratios of the samples are far from equilibrium (Figure 5), ranging from 0.42 to 0.90, except for LGP06S04 for which $^{230}\text{Th}/^{234}\text{U}$ ratios are close to 1 in both two tissues and the enamel tissue of LGP06S07 (~0.97). Accordingly, the apparent U-series ages of Longgupo dental tissues range from 58 ka to 208 ka, indicate a very recent uranium uptake, except for enamel and dentine tissue of LGP06S04 and enamel tissue of LGP06S07 which provide values higher than 250 ka. The relative consistent apparent U-series ages probably reflect a dramatic hydrological variation at the Longgupo site during the late Middle Pleistocene and Late Pleistocene. Although the uranium-series data did not give meaningful ages of the samples, it allows dating of Longgupo teeth with the US-ESR model using the combined ESR and U-series method, which is not common for early

Pleistocene open-air sites, as recently illustrated by Duval *et al.* (2012b) and Han *et al.* (submitted).

Table 2 shows US-ESR age results including the different dose rate components calculated with USESR program with MATLAB software (Shao *et al.*, 2014). For all the Longgupo teeth, the external dose rates contributed by sediments and cosmic rays makes a significant contribution to the total dose rates (49% - 90%), except for LGP06S04 (27%), and *in situ* dose rates (gamma and cosmic dose rates) accounted for the major part of external dose rates (58% - 95%). During the revisiting of Longgupo site, we made more detailed gamma dose rate measurement based on the new stratigraphy. The renewed *in situ* gamma dose rate measurement was pursued not only on the new layers, but also some previously measured layers. The result of the new measurement shows systematically much lower values than in 2006. This is probably because during the 2006 excavation the NaI detector could not be inserted deeply into the surrounding deposits in order to avoid the potential destruction of masked fossil remains. In the revisit, a detector with smaller diameter was used, and the measurements were pursued carefully on each available layer. We have preferentially used the *in situ* gamma dose rate measured during 2012 in these layers for age calculation of the corresponding teeth.

The calculated p-values of the dental tissues in all the samples are beyond zero, which represent a recent uranium uptake history, except for sample LGP06S04 from layer C III 3 of the south wall. For the north wall, ten teeth from unit C III' show consistent ages in general, about 2.35 Ma on average, and there is not much difference from the mean age (~ 2.43Ma) if we exclude sample LGP06N07, for which the younger age is probably caused by the high uranium content in the dental tissues and attached sediments.

For the south wall, the sample age distribution is not in accordance with the stratigraphic order. Three teeth (LGP06S02, LGP06S04 and LGP06S05) are much younger than the other four, probably because of the relative higher uranium concentration and U/Th ratio in the dental tissues (LGP06S04), and significant higher *in situ* gamma dose rate, especially for LGP06S02 from layer C III H1 (corresponding to layer C III 1 α), whose *in situ* dose value is almost three times higher than adjacent layers. In this layer, coarse and fine grained sediments alternate, indicating flood recurrence and dissipation of the

nearby river (Boëda *et al.*, 2011b). Nevertheless, excluding the three teeth with unexpected young ages, the three teeth from C III give an average age of ~ 2.48 Ma, corresponding with unit C III' of the north wall in general. Study of layer C II 2 shows that the bone accumulated with stone artifacts, and the presence of traces on some bones may indicate secondary butchery work (Wei *et al.*, 2014). This could explain the older age of sample LGP06S01 from layer C II 2a than the mean age of teeth samples of unit C III, as it was probably not deposited originally in that layer. The age results of Longgupo site in this study is older than our preliminary estimates (Han *et al.*, 2012) as *in situ* dose rate measurement was not available for some layers in the previous study. As shown in Figure 6, the external dose rate of Longgupo fossil teeth, which is comprised of beta and gamma dose rate from sediment and cosmic dose rate, makes a great contribution to the total dose rate for most of the samples. This indicates the external dose rate determination is crucial to the age calculation of Longgupo samples. This is the main difficulty of fossil dating at Longgupo, as the deposition is rather complex and non-homogenous. In addition, as the fossil teeth in some layers may have a unique uranium migration history at a certain period of time due to hydrological or geochemical variation of the surrounding sediment, as shown in Han *et al.* (2010) and this study, analyzing several teeth from a single layer is highly recommended in order to identify the conditions causing age underestimation or overestimation. In spite of the above difficulties in this study, the updated results based on detailed *in situ* dose measurement in 2012 agree with the paleontological evidence of Longgupo, which suggest a time range of late Pliocene to early Pleistocene (Huang *et al.*, 1995). In addition, our preliminary paleomagnetic results show that the Longgupo sedimentary sequence is dominantly of reverse polarity, which most possibly corresponds to the Matuyama reverse chron between the Gauss-Matuyama geomagnetic reversal (2.581 Ma) and the onset of the Olduvai normal subchron (1.945 Ma), if biochronology of the Longgupo mammalian fossils and ESR chronology of this study are considered (Figure 7). Recent chronological studies of *Gigantopithecus* faunas in Guangxi Province show that they could be dated to approximately 1.8–2.0 Ma, within the Olduvai subchron for the Chuifeng cave in the Bubing Basin (Shao *et al.*, 2014) and the Juyuan cave in the Chongzuo area (Jin *et al.*, 2014; Sun *et al.*, 2014), or within the pre-Olduvai Matuyama

chron for the Baikong cave in the Chongzuo area (Jin *et al.*, 2014; Sun *et al.*, 2014). According to the present work, the age of the *Gigantopithecus* fauna in the Longgupo site was slightly older than the studied cave sites in Guangxi Province.

4. Conclusions

This study dated the fossil teeth systematically from different layers of the south and north walls of Longgupo site according to the new stratigraphic division and updated *in situ* gamma dose rate measurement. The age results of fossil teeth from the top of unit C III to the layer C III 8 range between ~2.2 and 2.5 Ma. The sedimentary layer bearing hominid and artifacts, which is correlated to C III 2, is dated to ~2.2 Ma. This finding places the Longgupo site at the very beginning of the Early Pleistocene. Although the creators of the “Longgupo culture” are still a mystery (Wei *et al.*, 2014), compared with other convincing evidence of early hominid settlements in China, such as Renzidong Cave, Anhui Province (2- 2.4 Ma) (Jin and Liu, 2009; Wang *et al.*, 2012), Majuangou III Paleolithic site in the Nihewan Basin, northern China at ~1.7 Ma (Zhu *et al.*, 2004; Liu *et al.*, 2014), *Homo* in the Yuanmou Basin, southern China at ~1.7 Ma (Zhu *et al.*, 2008) and *Homo erectus* at Gongwangling, Lantian, southern Chinese Loess Plateau at ~1.6 Ma (Zhu *et al.*, 2014), the Longgupo site is undoubtedly one of the earliest evidences of hominid settlement in China and East Asia to date.

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Table captions:

Table 1 Information of Longgupo fossil teeth and ESR analyzed data

Table 2 U-series analysis data of Longgupo fossil teeth

Table 3 The dose rate components of US-ESR model by combined ESR/U-series analysis

Figure captions:

Figure 1 Location of Longgupo site

Figure 2 Sino-Franco joint excavation of Longgupo during 2003-2006

(a) Excavation along south wall (b) north wall

(c) South wall section and stratigraphic well; (d) *in situ* dose rate measurement in the well of south wall in 2012

Figure 3 Stratigraphic sequence of Longgupo site based on two periods of excavation (1984-1988, [Huang and Fang, 1991](#); 2003-2006, [Boëda *et al.*, 2011b](#)). A, B-stratigraphic sequence of [Huang and Fang \(1991\)](#); C, D-stratigraphic division in 2003 and 2006 ([Rasse *et al.*, 2011](#)); E-representative fauna in different geological units of Longgupo ([Huang and Fang, 1991](#)).

Figure 4 ESR measured dose points fitted by double exponential saturation (DES) function

Figure 5 U/Th isochron of analyzed fossil teeth

Figure 6 Alpha, beta, gamma and cosmic dose rate contribution of total dose rate in Longgupo fossil teeth

Figure 7 Calculated US-ESR ages of all Longgupo fossil teeth samples and their correlations with the Astronomically Tuned Neogene Time Scale of Hilgen *et al.* (2012) (ATNTS2012). J, Jaramillo; O, Olduvai; K, Kaena; M, Mammoth. Grey area is expected ages based on magnetobiochronology.

1- Clay; 2- Stalagmitic floor (Pl. St); 3- Coarse alluvial deposits; 4- Very large cobbles; 5- Breccified area; 6- Archaeological layer; 7- Hominin fossil layers (levels 7-8); 8- *Gigantopithecus* fossil layers (levels 5-8); 9- ESR dating samples

Table 1 Information of Longgupo fossil tooth ESR analyzed data

Sample No.	Layer	Depth	Species	Thickness enamel (μm)	Removed enamel (S1/S2*) (μm)	D _E (Gy)
<i>South wall</i>						
LGP06S01	C II 2a	2	Cervid	1206 $\pm$ 121	73 / 46	1778.2 $\pm$ 74.7
LGP06S02	C III H1	6	Bovinae	1457 $\pm$ 146	40 / 58	2574.2 $\pm$ 125.7
LGP06S03	C III 2	7	Suid	2498 $\pm$ 250	179 / 80	1672.2 $\pm$ 38.6
LGP06S04	C III 3	8	Rhinoceros	1153 $\pm$ 115	26 / 33	3221.4 $\pm$ 129.5
LGP06S05	C III 7a	12	Cervid	1227 $\pm$ 123	49 / 60	2406.6 $\pm$ 126.9
LGP06S06	C III 8a	13	Cervid	1791 $\pm$ 179	101 / 47	2147.6 $\pm$ 91.4
LGP06S07	C III 8b	14	Cervid	1215 $\pm$ 122	78 / 49	3012.7 $\pm$ 162.1
<i>North wall</i>						
LGP06N01	C III' 2	8	Megaovis	854 $\pm$ 85	27 / 53	1982.3 $\pm$ 214.1
LGP06N02	C III' 5	10	Rhinoceros	2010 $\pm$ 201	90 / 68	1931.9 $\pm$ 117.6
LGP06N03	C III' 6	11	Tapirus sinensis	2256 $\pm$ 226	101 / 80	1698.1 $\pm$ 75.4
LGP06N04	C III' 6	11	Cervid	1186 $\pm$ 119	81 / 60	2122.7 $\pm$ 96.0
LGP06N05	C III' 6	11	Bovinae	1105 $\pm$ 110	51 / 40	2216.7 $\pm$ 119.4
LGP06N06	C III' 6	11	Bovinae	1391 $\pm$ 139	37 / 114	2444.9 $\pm$ 226.5
LGP06N07	C III' 6 base	12	Suid	2111 $\pm$ 211	140 / 89	2374.9 $\pm$ 107.6
LGP06N08	C III' 6 base	12	Hyena	831 $\pm$ 83	67 / 40	2662.7 $\pm$ 123.0
LGP06N09	C III' 6 base	12	Tapirus sinensis	2640 $\pm$ 264	62 / 72	1785.9 $\pm$ 45.9
LGP06N10	C III' 6 base	12	Bovinae	1331 $\pm$ 133	65 / 278	2727.7 $\pm$ 126.2

* S1 and S2 represent the dentine side and sediment side of enamel

samples respectively.

Table 2 U-series analysis data of Longgupo fossil tooth

Sample No.	Layer	Tissue	U-content (ppm)	$^{234}\text{U}/^{238}\text{U}$	$^{230}\text{Th}/^{234}\text{U}$	$^{226}\text{Ra}/^{230}\text{Th}$	$^{222}\text{Rn}/^{230}\text{Th}$	U-series age (ka)
LGP06S01	C II 2a	Enamel	1.04 ± 0.04	1.203 ± 0.060	0.416 ± 0.058	1.000	0.390	$57.6 + 11.3/-10.1$
		Dentine	44.10 ± 1.10	1.252 ± 0.029	0.786 ± 0.047	0.576	0.379	$154.5 + 22.5/-18.6$
LGP06S02	C III H1	Enamel	3.02 ± 0.08	1.211 ± 0.035	0.697 ± 0.025	0.603	0.602	$123.8 + 9.1/-8.3$
		Dentine	105.48 ± 1.71	1.149 ± 0.016	0.716 ± 0.026	0.562	0.336	$131.8 + 9.9/-9.0$
LGP06S03	C III 2	Enamel	2.87 ± 0.07	1.339 ± 0.035	0.768 ± 0.044	0.793	0.866	$144.8 + 18.5/-15.7$
		Dentine	36.59 ± 0.64	1.454 ± 0.023	0.827 ± 0.044	0.637	0.503	$164.3 + 20.8/-17.7$
LGP06S04	C III 3	Enamel	3.08 ± 0.09	1.352 ± 0.040	0.992 ± 0.043	0.674	0.543	$276.1 + 68.3/-42.4$
		Dentine	79.31 ± 2.00	1.419 ± 0.031	1.007 ± 0.033	0.511	0.282	$282.2 + 48.9/-34.3$
LGP06S05	C III 7a	Enamel	6.73 ± 0.10	1.247 ± 0.018	0.777 ± 0.019	0.696	0.621	$150.8 + 8.5/-7.9$
		Dentine	41.94 ± 0.59	1.112 ± 0.014	0.757 ± 0.019	0.536	0.328	$148.2 + 8.7/-8.0$
LGP06S06	C III 8a	Enamel	0.91 ± 0.04	1.305 ± 0.074	0.757 ± 0.058	1.000	0.734	$141.8 + 25.9/-20.4$
		Dentine	51.62 ± 0.91	1.147 ± 0.019	0.861 ± 0.024	0.410	0.273	$197.5 + 17.6/-14.9$
LGP06S07	C III 8b	Enamel	9.14 ± 0.20	1.304 ± 0.021	0.965 ± 0.033	0.763	0.540	$256.7 + 39.6/-29.3$
		Dentine	79.16 ± 2.02	1.291 ± 0.029	0.807 ± 0.052	0.641	0.377	$161.6 + 26.3/-21.2$
LGP06N01	C III' 2	Enamel	1.31 ± 0.06	1.279 ± 0.062	0.866 ± 0.053	0.891	0.756	$190.4 + 39.4/-28.2$
		Dentine	68.70 ± 1.83	1.300 ± 0.031	0.899 ± 0.033	0.634	0.379	$208.1 + 25.9/-20.8$
LGP06N02	C III' 5	Enamel	5.37 ± 0.10	1.315 ± 0.024	0.699 ± 0.024	0.588	0.745	$122.3 + 8.0/-7.4$
		Dentine	40.57 ± 0.54	1.213 ± 0.015	0.769 ± 0.016	0.497	0.310	$148.9 + 7.1/-6.6$
LGP06N03	C III' 6	Enamel	0.43 ± 0.04	1.259 ± 0.168	0.693 ± 0.106	1.000	0.947	$121.4 + 46.5/-30.2$
		Dentine	53.32 ± 2.10	1.219 ± 0.044	0.754 ± 0.072	0.365	0.281	$143.3 + 32.7/-24.9$
LGP06N04	C III' 6	Enamel	1.71 ± 0.06	1.236 ± 0.043	0.834 ± 0.045	0.505	0.685	$176.6 + 27.9/-21.8$

LGP06N05	C III' 6	Dentine	43.26 ± 1.33	1.235 ± 0.034	0.880 ± 0.047	0.449	0.295	$201.7 + 36.9/-27.3$
		Enamel	2.19 ± 0.07	1.357 ± 0.037	0.836 ± 0.030	0.659	0.633	$171.8 + 16.3/-14.1$
		Dentine	28.37 ± 0.61	1.296 ± 0.027	0.819 ± 0.027	0.440	0.333	$166.4 + 13.9/-12.3$
LGP06N06	C III' 6	Enamel	4.60 ± 0.09	1.273 ± 0.023	0.769 ± 0.022	0.767	0.663	$147.1 + 9.4/-8.6$
		Dentine	21.08 ± 0.36	1.193 ± 0.019	0.745 ± 0.019	0.569	0.393	$140.4 + 7.8/-7.2$
LGP06N07	C III' 6 base	Enamel	6.65 ± 0.30	1.261 ± 0.045	0.847 ± 0.040	0.446	0.595	$181.5 + 26.0/-20.6$
		Dentine	53.77 ± 1.64	1.053 ± 0.030	0.797 ± 0.046	0.518	0.335	$169.8 + 29.3/-22.4$
LGP06N08	C III' 6 base	Enamel	4.74 ± 0.18	1.294 ± 0.044	0.710 ± 0.057	1.000	0.662	$126.2 + 20.9/-17.4$
		Dentine	30.74 ± 1.24	1.159 ± 0.044	0.863 ± 0.051	0.452	0.316	$197.3 + 42.7/-29.7$
LGP06N09	C III' 6 base	Enamel	0.32 ± 0.01	1.311 ± 0.043	0.716 ± 0.033	1.000	0.545	$127.8 + 12.1/-10.8$
		Dentine	36.32 ± 0.55	1.181 ± 0.016	0.829 ± 0.017	0.482	0.270	$176.8 + 10.2/-9.2$
LGP06N10	C III' 6 base	Enamel	7.72 ± 0.17	1.348 ± 0.027	0.788 ± 0.049	0.687	0.684	$151.9 + 21.7/-18.1$
		Dentine	21.3 ± 0.68	1.214 ± 0.036	0.830 ± 0.042	0.498	0.349	$175.8 + 25.5/-20.3$

Table 3 The dose rate components of US-ESR model by combined ESR/U-series analysis

Sample No.	Layer	Level (Huang et al., 1995)	γ sediment +cosmic ($\mu\text{Gy/a}$)	Internal dose ($\alpha+\beta$) enamel ($\mu\text{Gy/a}$)	β dentine ($\mu\text{Gy/a}$)	Total dose rate ($\mu\text{Gy/a}$)	p-enamel	p-dentine	US-ESR age (ka)
LGP06S01	C II 2a	2	494 ± 37	11 ± 9	50 ± 41	640 ± 56	34.15 ± 2.75	5.75 ± 0.50	$2711 \pm 289/151$
LGP06S02	C III H1	6-7	1473 ± 140	143 ± 120	123 ± 103	1818 ± 211	4.41 ± 0.60	3.92 ± 0.55	$1364 \pm 199/96$
LGP06S03	C III 2	7-8	405 ± 34	162 ± 56	27 ± 9	656 ± 66	5.91 ± 0.66	3.85 ± 0.44	$2460 \pm 409/131$
LGP06S04	C III 3	8-9	622 ± 51	1440 ± 561	833 ± 324	3112 ± 650	-0.58 ± 0.06	-0.71 ± 0.07	$1133 \pm 124/341$
LGP06S05	C III 7a	12	776 ± 72	441 ± 151	59 ± 20	1427 ± 169	3.32 ± 0.47	3.77 ± 0.53	$1642 \pm 224/136$
LGP06S06	C III 8a	13-14	623 ± 55	41 ± 38	55 ± 50	829 ± 84	6.44 ± 0.67	2.73 ± 0.32	$2540 \pm 299/181$
LGP06S07	C III 8b	13-14	619 ± 55	385 ± 151	95 ± 37	1200 ± 165	6.22 ± 0.90	2.62 ± 0.43	$2449 \pm 474/226$
LGP06N01	C III' 2	7-8	405 ± 34	143 ± 83	307 ± 178	997 ± 199	1.85 ± 0.48	1.14 ± 0.35	$1946 \pm 399/306$
LGP06N02	C III' 5	10-11	541 ± 50	226 ± 100	29 ± 13	881 ± 117	7.46 ± 1.02	4.90 ± 0.71	$2133 \pm 324/196$
LGP06N03	C III' 6	11-12	513 ± 50	15 ± 23	26 ± 39	646 ± 68	9.44 ± 1.01	6.70 ± 0.75	$2638 \pm 249/261$
LGP06N04	C III' 6	11-12	513 ± 50	124 ± 64	93 ± 48	894 ± 95	3.29 ± 0.42	2.00 ± 0.28	$2341 \pm 279/196$
LGP06N05	C III' 6	11-12	513 ± 50	155 ± 79	44 ± 22	859 ± 97	3.65 ± 0.46	4.19 ± 0.52	$2552 \pm 294/231$
LGP06N06	C III' 6	11-12	513 ± 50	220 ± 116	21 ± 11	829 ± 127	6.89 ± 0.92	7.97 ± 1.07	$2862 \pm 484/251$
LGP06N07	C III' 6 base	11-12	508 ± 50	671 ± 166	49 ± 12	1407 ± 175	1.79 ± 0.32	2.77 ± 0.46	$1564 \pm 334/76$
LGP06N08	C III' 6 base	11-12	508 ± 50	174 ± 89	85 ± 43	1138 ± 113	7.54 ± 0.75	2.34 ± 0.28	$2290 \pm 264/156$
LGP06N09	C III' 6 base	11-12	508 ± 50	10 ± 14	21 ± 30	670 ± 61	8.45 ± 0.82	3.90 ± 0.41	$2628 \pm 284/191$
LGP06N10	C III' 6 base	11-12	508 ± 50	435 ± 121	36 ± 10	1085 ± 132	5.08 ± 0.66	3.61 ± 0.50	$2541 \pm 284/316$