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# Luminescence dating of Chinese loess beyond 130 ka using the non-fading signal from K-feldspar

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# Luminescence dating of Chinese loess beyond 130 ka using the non-fading signal from K-feldspar

## Abstract

A multi-elevated-temperature post-IR IRSL (MET-pIRIR) protocol, which utilizes the IRSL signals measured by progressively increasing the stimulation temperature from 50 to 300 °C in a step of 50 °C, was applied to date the potassium-rich feldspar (K-feldspar) extracts from loess samples at the Luochuan section of the Chinese Loess Plateau. It was observed that the MET-pIRIR ages obtained at elevated temperatures (250 and 300 °C) are consistent with independent chronological control for the samples from the first loess layer (L1) to the third paleosol layer (S3), which correspond to the marine isotope stages (MIS) 2e9. Our results indicate that the MET-pIRIR protocol can provide reliable ages for the Chinese loess up to ~300 ka. The results suggest that the MET-pIRIR signal measured at 250 and 300 °C gives the most reliable ages for older samples (>130 ka). For samples below L3, the natural MET-pIRIR signals measured at high temperatures reach the saturation level in dose response curves, suggesting a dating limit of ~300 ka for the Luochuan loess section.

## Keywords

luminescence, signal, fading, non, ka, 130, feldspar, beyond, k, loess, chinese, dating, CAS

## Disciplines

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# 1 Luminescence dating of Chinese Loess beyond 130 ka using the non-fading 2 signal from K-feldspar

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## 7 8 **Abstract**

9 A multi-elevated-temperature post-IR IRSL (MET-pIRIR) protocol, which utilizes the IRSL  
10 signals measured by progressively increasing the stimulation temperature from 50 to 300°C in a step  
11 of 50°C, has been applied to date the potassium-rich feldspar (K-feldspar) extracts from loess samples  
12 at the Luochuan section of the Chinese Loess Plateau. It was observed that the MET-pIRIR ages  
13 obtained at elevated temperatures (250 and 300 °C) are consistent with independent chronological  
14 control for the samples from the first loess layer (L1) to the third paleosol layer (S3), which  
15 correspond to the marine isotope stages (MIS) 2 to 8. Our results indicate that the MET-pIRIR  
16 protocol can provide reliable ages for the Chinese loess up to ~300 ka. The results suggest that, for  
17 young samples (<130 ka), it is preferable to use the MET-pIRIR signal at 200°C due to its low residual  
18 age. However, the MET-pIRIR signal measured at 250 and 300 °C gives the most reliable ages for  
19 older samples (>130 ka). For samples below L3, the natural MET-pIRIR signals measured at high  
20 temperatures reach the saturation level in dose response curves, suggesting a dating limit of ~300 ka  
21 for the Luochuan loess section.

22  
23 **Keywords:** K-feldspar, luminescence dating, post-IR IRSL, Luochuan loess.

## 24 **1. Introduction**

25 Chinese loess provides an ideal profile of past climatic change, due to its quasi-continuous  
26 nature of dust deposition (Liu, 1985). Luminescence dating is one of the main tools for dating loess,  
27 and it has been extensively applied to loess sections in China in the last three decades (Forman, 1991;  
28 Frechen, 1999; Lai et al., 2010; Lu et al., 1987; Wang et al., 2006; Zhou and Shackleton, 2001).  
29 However, most of these studies were limited to the upper most loess and paleosol sections (S0, L1 and  
30 S1) (Buylaert et al., 2007; Lai, 2010; Lu et al., 2007). Optical stimulated luminescence (OSL) dating  
31 of Chinese loess beyond ~130 ka has long been a challenge for luminescence researchers, mainly due  
32 to the early saturation of quartz OSL signal.

33 Feldspar is a promising candidate for extending the luminescence dating limit due to its much  
34 higher saturation dose. However, the anomalous fading of the infrared stimulated luminescence (IRSL)  
35 signal from feldspar has hampered the application of feldspar in optical dating (e.g. Huntley and  
36 Lamothe, 2001; Huntley and Lian, 2006; Lamothe and Auclair, 1999; Li et al., 2007). Recent  
37 progresses in understanding the anomalous fading of the IRSL from feldspar have allowed the  
38 possibility of isolation of a non-fading component in feldspar IRSL. It has been suggested that the  
39 initial part of the IRSL signal has a higher anomalous fading rate when compared to the later part  
40 (Thomsen et al., 2008). This observation has led to the development of a post-IR IRSL dating method,  
41 in which an IRSL bleaching at low temperature (~50°C) is applied before a high temperature (>200°C)  
42 IRSL measurement to reduce the fading rate of feldspar (Thomsen et al., 2008; Buylaert et al., 2009;  
43 Thiel et al., 2011). It was suggested that, when a high-temperature post-IR IRSL (pIRIR) was  
44 conducted at 290 °C, it is possible to obtain a non-fading signal (Thiel et al., 2011). However,  
45 detectable anomalous fading is still present in the elevated-temperature post-IR IRSL signals (Buylaert  
46 et al., 2009), although Thiel et al. (2011) suggested that fading correction is not necessary for their  
47 samples.

48 More recently, Li and Li (2011) found that a multiple IR stimulation procedure with increasing  
49 stimulation temperature is able to isolate the non-fading IRSL signal. Based on this observation, a

50 protocol for IRSL dating K-rich feldspar has been proposed, which utilizes the IRSL signals measured  
51 by progressively increasing the stimulation temperature from 50 to 250°C in a step of 50°C, and is  
52 called multi-elevated-temperature post-IR IRSL (MET-pIRIR) protocol (Li and Li, 2011). The  
53 laboratory fading test for a feldspar sample showed that there are negligible anomalous fading rates (g  
54 values) for the MET-pIRIR signals obtained at 200°C and 250°C. Hence, no fading correction is  
55 required. The reliability of this protocol has been tested using various sedimentary samples from  
56 China deposited in the last ~130 ka (Li and Li, 2011). It was observed that the MET-pIRIR ages  
57 obtained at 200 and 250 °C are consistent with independent or quartz OSL ages.

58 In this study, we further tested the MET-pIRIR applicability to potassium-rich feldspar (K-  
59 feldspar) extracts from loess samples from the Luochuan section of the Chinese Loess Plateau. The  
60 method was applied to samples from the first loess layer (L1) to the third paleosol layer (S3),  
61 corresponding to marine isotope stages from MIS 2 to MIS 9. We aim to test the validity of the MET-  
62 pIRIR protocol for loess samples older than 130 ka by comparing with the well-established  
63 stratigraphic ages for Chinese loess (Ding et al., 2002). Finally we explore the MET-pIRIR age limit.

## 64 **2. Luochuan section and samples**

65 The Luochuan section has been extensively studied as one of the well-known sections in the  
66 Chinese Loess Plateau for palaeo-environmental studies (An et al., 1991; Liu, 1985; Porter and An,  
67 1995). The chronology of the upper most loess and paleosol (S0, L1 and S1) of this section has been  
68 established using the sensitivity-corrected multiple-aliquot regenerative-dose (MAR) OSL dating  
69 protocol for quartz (Lu et al., 2007). For the lower part of the section, independent ages can be  
70 obtained by climatic correlation to the well-established chronology in the marine sediments reported  
71 by Imbrie et al. (1984) (Liu, 1985; An et al., 1991; Porter and An, 1995). Based on orbital tuning of  
72 the grain-size records from several sections in Chinese Loess Plateau, Ding et al. (2002) established a  
73 stacked climate record of the Quaternary period from the Chinese loess, which can be correlated with  
74 the oxygen isotopic record in deep-sea sediments. Here we adopt the ages of the loess/paleosol

75 boundaries provided by Ding et al. (2002) as an independent age control for our samples, i.e. the age  
76 of individual sample can be obtained by linearly interpolating their stratigraphic positions (sampling  
77 depth) into the corresponding loess/paleosol boundary ages in which the samples were embedded. A  
78 total of 14 samples were collected between the top of first loess layer (L1) to the upper part of the  
79 forth loess layer (L4). Another was collected from the ninth loess layer (L9), at 63 m bellow the  
80 surface. According to Ding et al. (2002), the upper 14 samples were deposited from 20 to 350 ka ago.  
81 The sample taken from L9 was expected to have an age of ~900 ka, which is treated as a field-  
82 saturated sample, i.e. we expect that the IRSL signal is not growing any longer and has reached an  
83 equilibrium state with the dose rate. A summary of the stratigraphy of the section, sampling positions  
84 are shown in Table 1.

85

### 86 **3. Experimental procedures and analytical facilities**

87 The samples were routinely treated with HCl and H<sub>2</sub>O<sub>2</sub> to remove carbonate and organic  
88 matter and dried. After that, 63-90 μm grains were obtained by dry sieving. The K-feldspar grains  
89 were then separated using the heavy liquid with a density of 2.58 g/cm<sup>3</sup>. The K-feldspar grains were  
90 etched using 10% HF for 10 minutes to clean the surface of the grains and also reduce the alpha-  
91 irradiated layer around the grain surface. The K-feldspar IRSL measurements were made on an  
92 automated Risø TL-DA-15 reader equipped with IR diodes (870Δ40 nm) for stimulation (Bøtter-  
93 Jensen et al., 2003). The total IR power delivered to the sample position was ~135mW/cm<sup>2</sup> (Bøtter-  
94 Jensen et al., 2000). Irradiations were carried out within the reader using a <sup>90</sup>Sr/<sup>90</sup>Y beta source. The  
95 IRSL signals were detected using a photomultiplier tube with the stimulated luminescence passing  
96 through a filter pack containing Schott BG-39 and Corning 7-59 filters, which provides a blue  
97 transmission window (320-480 nm). Aliquots containing several hundred grains were prepared by  
98 mounting the grains in a monolayer on a 9.8 mm diameter aluminum disc with “Silkospay” silicone  
99 oil.

100 The environmental (or external) dose rates were measured using the thick-source alpha  
101 counting (TSAC) technique (Aitken, 1985) for determining the contribution from U and Th and X-ray  
102 fluorescence (XRF) for the K content. The water contents of  $15\pm 5\%$  and  $20\pm 5\%$ , similar to those used  
103 in previous publications (Lai, 2010; Lu et al., 2007), were used for the loess layers and paleosol layers,  
104 respectively. The contribution from cosmic ray was calculated from the burial depth and the latitude  
105 and altitude of the samples (Prescott and Hutton, 1994). The internal dose rate for K-feldspar used in  
106 age calculation was estimated by assuming  $K=13\pm 1\%$  and  $Rb=400\pm 100$  ppm (Huntley and Baril,  
107 1997; Huntley and Hancock, 2001; Zhao and Li, 2005; Li et al. 2008;). A summary of the dosimetry  
108 data for all samples is listed in Table 1.

109

### 110 **3. The MET-pIRIR protocol**

111 In the original MET-pIRIR protocol proposed by Li and Li (2011), the IRSL signals of both  
112 regenerative and test doses were measured for several times and each measurement was conducted by  
113 increasing the stimulation temperature in steps of  $50^\circ\text{C}$ , e.g. 50, 100, 150, 200 and  $250^\circ\text{C}$ . Based on  
114 this protocol, several equivalent doses and ages can be obtained for different MET-pIRIR signals at  
115 different stimulation temperatures. A basic feature of the MET-pIRIR protocol is that one can plot the  
116 MET-pIRIR ages with the corresponding IR stimulation temperatures, so-called Age\_Temperature (A-  
117 T) plot. It was observed that the MET-pIRIR ages were increasing with IR temperature, indicating a  
118 lower anomalous fading for the signals measured at higher temperatures. An age plateau was reached  
119 above  $200^\circ\text{C}$  (Li and Li, 2011). It was found that such age plateau is consistent with quartz OSL ages  
120 and independent ages for their samples, indicating that the non-fading component was depleted by  
121 previous IR stimulations at lower temperatures. Therefore, an age plateau in the A-T plot could be  
122 used as an indicator for whether a stable or non-fading component has been achieved using the MET-  
123 pIRIR procedure.

124 In this study, we applied a MET-pIRIR protocol introduced by Li and Li (2011) where we  
125 added stimulations at 300°C. following the 250°C measurement. We will show that a higher MET-  
126 pIRIR stimulation temperature above 250 °C was necessary to achieve an age plateau in the A-T plot  
127 for older samples (see next sections for details) and the MET-pIRIR measured at 200°C did not  
128 reached the stability observed at higher temperature for older samples. A detailed procedure of the  
129 protocol used in this study was shown in Table 2. A preheat at 320°C for 60 s (step 2 and 10) was  
130 applied after both regenerative and test doses to avoid significant influence from residual  
131 phosphorescence while recording the MET-pIRIR at 300°C (step 8 and 16). The IRSL signals of both  
132 regenerative and test doses are measured for several times and each measurement was obtained by  
133 increasing the stimulation temperature in steps of 50°C, i.e. 50, 100, 150, 200, 250 and 300 °C (Table  
134 2). At the end of the IRSL measurements for each test dose, a ‘hot’ IR bleaching at 325°C for 100 s  
135 (step 17) is conducted to minimize the residual signal preceding the next measurement cycle.

## 136 **4. Results**

### 137 **4.1 Laboratory dose response curves**

138 The dose response curves (DRCs) for the MET-pIRIR signals were obtained using the  
139 procedure of Table 2. It was found that different samples from this section have nearly identical DRCs.  
140 Fig. 1 shows the compiled sensitivity-corrected DRCs for different MET-pIRIR temperatures. These  
141 DRCs show a continuous growth of up to 1000 Gy, demonstrating a great potential of feldspars for  
142 dating old samples.

143 In Fig. 1, different DRC shapes were observed for various stimulation temperatures. The 50°C  
144 IRSL signal has the highest sensitivity-corrected intensity. The DRCs for the 100, 150 and 200 °C  
145 signals are similar, but the 250 and 300 °C signals have lower intensities. These DRCs can be fitted  
146 using single exponential saturation functions. The fitting results suggest that the 50 °C IRSL signal  
147 saturated at the highest dose level ( $D_0=402$  Gy, where  $D_0$  refers to the characteristic saturation dose).  
148 The 100, 150 and 200 °C signals saturated at a lower dose than the 50 °C IRSL did but have a similar



149 saturation dose level, i.e.  $D_0=338, 362$  and  $358$  Gy, respectively. Lower saturation doses of  $327$  and  
150  $250$  Gy were observed for the  $250$  and  $300$  °C signals, respectively.

151 Routine tests of recycling ratio and recuperation or the ratio between the signal responses of  
152 zero dose and natural dose can be conducted in construction of DRCs during  $D_e$  measurements (Wintle  
153 and Murray, 2006). The recycling ratios for all of the samples fall within the range of  $1.0\pm 0.1$ , and the  
154 recuperation values are generally less than  $5\%$ , all of which is considered acceptable.

155

## 156 **4.2 Age-Temperature (A-T) plots**

157 The MET-pIRIR ages were derived from the equivalent dose and the dosimetry data (Table 1).  
158 These feldspar ages, together with the expected ages, are summarized in Table 3. It is noted that no  
159 residual ages has been corrected for our samples, because it was observed that the residual dose is only  
160 a few Gy for the high-temperature MET-pIRIR signals for the Luochuan loess (Fu et al., submitted). A  
161 straightforward comparison of MET-pIRIR ages to expected ages can be illustrated in A-T plots.  
162 Typical A-T plots from 4 samples with different ages are shown in Fig. 2. The expected ages are  
163 shown as shaded bands in Fig. 2. It is observed that the IRSL ages obtained at  $50$  °C are the lowest  
164 ones, which significantly underestimate the expected chronology. This can be explained as a result of  
165 anomalous fading. The ages tend to increase with the stimulation temperature and an age plateau can  
166 be reached at higher temperatures. For the youngest sample in Fig. 2, LC-096, the MET-pIRIR ages  
167 measured at  $200$ °C and above are consistent with the expected ages (yellow shaded area). Similar  
168 results were also observed for the two younger samples, LC-019 and LC-054 (Table 3). However, for  
169 older samples in Fig. 2, LC-133, LC-170 and LC-230, the MET-pIRIR ages measured at  $200$ °C were  
170 underestimated, and only the  $250$  and  $300$  °C signals gave consistent ages with the expected ages (Fig.  
171 2). This result suggests that the natural luminescence of the MET-pIRIR measured at  $200$ °C probably  
172 suffered from anomalous fading, although in a small extent. This was not detectable for the relatively  
173 young samples ( $<130$  ka for our samples). Such a small anomalous fading rate became important for  
174 older samples, resulting in an age underestimation. Therefore, the results suggest that the MET-pIRIR

175 ages obtained using temperature at 200°C and above are more reliable for older samples (>130 ka)  
176 than the 200 °C MET-pIRIR signals. However, for younger samples, the 200 °C MET-pIRIR signals  
177 can still be used for age determination, given its lower residual ages compared to the signals at higher  
178 temperatures (Li and Li, 2011). Hence, the presence of an age plateau in the A-T plot is an important  
179 criterion for the selection of appropriate MET-pIRIR temperatures.

180

### 181 **4.3 Natural dose response curves**

182         Based on expected ages and the dose rates, one can calculate the expected paleodose for each  
183 sample (Table 3). It is thus possible to reconstruct the natural dose response curve (N-DRC) for the  
184 loess sample in Luochuan section. In Fig. 3, the sensitivity-corrected natural MET-pIRIR signals  
185 ( $L_n/T_n$ ) at different temperatures from different samples were plotted against the corresponding  
186 expected paleodoses. The smooth growth of the natural signal intensities for different samples at  
187 different depth and ages suggested that these samples have a similar dose response behaviour in nature.  
188 Different MET-pIRIR signals at different temperatures have different sensitivity-corrected intensities,  
189 but all the N-DRCs appear to be able to grow up to a dose level around ~1000 Gy. It is interesting to  
190 note that, if these natural DRCs are fitted with single exponential saturation functions (not shown in  
191 Fig. 3), the values of the natural saturation doses ( $D_0$ ) are 421, 338, 341, 324, 307 and 250 Gy for the  
192 MET-pIRIR signals at 50, 100, 150, 200, 250 and 300 °C, respectively. These values are identical to  
193 those obtained for laboratory DRCs shown in Fig. 1. The results indicate that the same origins of traps  
194 were involved for both the natural signals and laboratory signals. However, the different saturation  
195 dose levels for different MET-pIRIR signals at different temperatures indicate that these signals are  
196 probably involved from different groups of traps.

197         For comparison, the laboratory DRCs for different MET-pIRIR signals shown in Fig. 1 are  
198 also shown (note that the laboratory DRCs shown in Fig. 3 are the best fitting curves from Fig. 1). It is  
199 shown from Fig. 3(a) that the N-DRC for the 50 °C IRSL signal is considerably below the laboratory  
200 DRC. If the natural intensity is projected onto the laboratory DRC, significant dose underestimation

201 will be resulted. In addition, older samples with larger paleodoses yield increasing underestimations,  
202 i.e. the dose (or age) underestimation increase from ~22% for the youngest sample LC-019 to ~55%  
203 for the sample LC-270. This result clearly supports the dose-dependent effect of the anomalous fading  
204 rate in nature (Li and Li, 2008). A similar pattern was observed for the 100 °C MET-pIRIR signal (Fig.  
205 3(b)), but a lower underestimation was observed, i.e. the N-DRC is more close to the laboratory DRC  
206 when compared to the 50 °C IRSL signal, suggesting that there is a lower anomalous fading for the  
207 100 °C MET-pIRIR signal. This is consistent with the anomalous fading rate test in Li and Li (2011)  
208 showing that there is a lower fading rate for the higher MET-pIRIR temperatures. A dose  
209 underestimation ranging from ~9% to ~50% from young samples to old samples were still observed  
210 for the 100 °C MET-pIRIR signal. For the 150 °C MET-pIRIR signal (Fig. 3(c)), the N-DRC is even  
211 more close to the laboratory DRC. It is interesting to note that the natural intensity from the youngest  
212 sample LC-019 is consistent with laboratory DRC, indicating a negligible underestimation and  
213 anomalous fading for this sample. For older samples, dose underestimation ranging from ~18% to ~40%  
214 was still observed.

215 For sediments younger than 130 ka it was previously suggested that MET-pIRIR stimulated at  
216 200°C was sufficient to yield accurate ages, mostly derived from quartz OSL ages, from different  
217 regions of China (Li and Li, 2011). This is consistent with the results in Fig. 3(d), which shows that  
218 the N-DRC is consistent with the laboratory DRC for samples with paleodose less than ~500 Gy  
219 (equivalent to ~140 ka). However, it is important to note that the 200 °C MET-pIRIR signal do get age  
220 underestimation for natural dose above ~500 Gy. For these older samples with paleodose larger than  
221 500 Gy, the natural luminescence intensities are slightly below the laboratory DRC. A small  
222 underestimation in the signal intensity can cause a significant underestimation in age (or dose),  
223 because the dose are located in the saturation part of the DRC. Therefore, we conclude that a  
224 considerable age underestimation may be caused for older samples with higher natural doses  
225 (e.g. >~500 Gy), even though a small anomalous fading rate (e.g. <1%/decade) was observed in

226 laboratory fading test. It is thus required to use higher MET-pIRIR temperatures to achieve non-fading  
227 components for these samples.

228 Despite of the underestimation observed for the MET-pIRIR signals at low temperatures  
229 (<200 °C), the N-DRCs for the MET-pIRIR signals at 250 and 300 °C show an excellent agreement  
230 with the laboratory DRCs for all samples investigated (Fig. 3(e) and Fig. 3(f)), indicating that a non-  
231 fading component can be achieved using a MET-pIRIR temperature at 250 °C and above. This can be  
232 further supported by measuring the natural intensities of the oldest sample from the ninth loess layer  
233 (L9), LC-626, which is expected to have received a natural dose of ~3000 Gy (equivalent to ~900 ka).  
234 Since the paleodose of LC-626 has far exceeded the natural saturation dose level (~1000 Gy), it can be  
235 treated as an infinite old sample whose luminescence attained an equilibrium state with the dose rate.  
236 The natural intensity of this sample should be consistent with the laboratory saturation level if there is  
237 no fading in nature and the signals are thermally stable. Our recent results (Li and Li, 2011; Li and Li,  
238 submitted) on the thermal stability of the MET-pIRIR signals indicated that these signals are thermally  
239 stable up to several million years. Therefore, anomalous fading would be expected to have occurred if  
240 the natural luminescence intensity from the field-saturated sample is lower than the saturation level of  
241 the laboratory-irradiated sample.

242 The natural MET-pIRIR intensities at different temperatures for LC-626 were shown as  
243 shaded area in Fig. 3. Note that the shaded area shows the  $1\sigma$  standard deviation. It is clearly shown  
244 that the natural intensities of LC-626 are consistent with those of samples with paleodose larger than  
245 ~1000 Gy, indicating that these samples have also reached at field saturation. The field saturation  
246 levels are below the laboratory saturation level for the MET-pIRIR signals at 50, 100, 150 and 200 °C,  
247 indicating that there were anomalous fading in nature for these signals. The difference between the  
248 field and laboratory saturation levels decreased as the stimulation temperature increased. This is  
249 expected because there is a lower anomalous fading rate for the signals at higher temperature. It is  
250 interesting to note that the field saturation levels agree very well with the laboratory saturation level

251 for the MET-pIRIR signals at 250 and 300 °C, confirming that the signals measured at 250 °C and  
252 above suffer negligible anomalous fading.

253

#### 254 **4.4 Dating results for Luochuan section**

255 The protocol shown in Table 2 has been applied to all samples and the MET-pIRIR ages were  
256 summarized in Table 3. All the ages were obtained from measurements of 4-6 aliquots. These ages,  
257 together with the stratigraphic column, are plotted against sampling depth in Fig. 4. The independent  
258 ages of the loess/paleosol (L/S) boundaries from Ding et al. (2002) are also shown. These age  
259 constrains were used for generating an age-depth curve (shown as full curve in Fig. 4), using linear  
260 interpolation of the sampling depth between the upper and lower boundaries of the loess or paleosol  
261 layers.

262 In Fig. 4, it is shown that the IRSL ages obtained at 50 °C are all underestimated. The ages  
263 tend to increase as the stimulation temperature increased. There is a good agreement between the  
264 MET-pIRIR 200°C ages and the expected ages for the samples younger than ~130 ka (from L1 and  
265 S1). The MET-pIRIR 200°C underestimated the expected ages for older samples. For the 250 and 300  
266 °C signals, the obtained ages are consistent with expected ages up to ~300 ka (equivalent to the lower  
267 part of L3). This suggests that the MET-pIRIR signals at 250 and 300 °C suffers from negligible  
268 anomalous fading and can be used to evaluate the K-feldspar age beyond 130 ka without fading  
269 correction.

270 For the sample (LC-270) older than 300 ka, the uncertainty of the ages of the MET-pIRIR  
271 signals at 250 and 300 °C become extremely large, which is mainly due to the saturation of the MET-  
272 pIRIR signals at 250 and 300 °C (see Fig. 3). Therefore, we conclude that the MET-pIRIR protocol  
273 has an age limit of ~300 ka (equivalent to a dose limit of ~900 Gy) for the Chinese loess.

274 In summary, based on the dating results, we suggest to use the MET-pIRIR 250 °C ages as the  
275 best age estimates for samples above S1 (<130 ka), and to used the average of the 250 and 300 °C ages  
276 as the best age estimates for older samples (shown in the last column of Table 3).

## 277 **9. Conclusions**

278 The MET-pIRIR signals protocol of measuring the IRSL signal from K-feldspars by  
279 progressively increasing the stimulation temperature from 50 to 300°C in step of 50°C can be  
280 successfully applied to the loess/paleosol units from L1 to L3 at the Luochuan section. When  
281 compared to the expected ages based on stratigraphic correlation, the IRSL obtained at 50°C has the  
282 largest underestimation in age. The extent of age underestimation decreases as the stimulation  
283 temperature increases. The ages for the 200°C MET-pIRIR signal are consistent with expected age for  
284 young samples (<130 ka), but are underestimated for older samples (>130 ka). Negligible anomalous  
285 fading was observed for the MET-pIRIR signals obtained at 250 and 300°C, which is supported by the  
286 age-temperature (A-T) plots showing age plateau above 250°C. The 250 and 300 °C MET-pIRIR  
287 signals can give reliable age estimation for the Chinese loess up to ~300 ka.

288

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293

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## Figure captions

Figure 1: The dose response curves for the MET-pIRIR signals at different temperatures. The data points are the average of the data sets obtained from five samples, LC-096, LC-120, LC-150, LC-170 and LC-250. The test dose used is 54Gy.

Figure 2: Typical age-temperature (A-T) plots for samples LC-096, LC-133, LC-170 and LC-230 obtained using the protocol of Table 2. The yellow shaded area in each plot shows the expected age (see Table 3 for values).

Figure 3: The sensitivity-corrected natural MET-pIRIR signals ( $L_n/T_n$ ) at different temperatures from different samples plotted against the corresponding expected paleodoses (values from Table 3). The best fitting curves of the laboratory DRCs from Fig. 1 are also shown as full curves. The horizontal dashed line and shaded area shows the natural MET-pIRIR intensities for sample LC-626, which has reached field saturation level (see text). Note that the shaded area shows the  $1\sigma$  standard deviation.

Figure 4: The MET-pIRIR ages (data points) (values in Table 3) and expected ages (full curve) plotted against sampling depth. The paleosol/loess units are shown as stratigraphic column on the right. The independent ages of the loess/paleosol (L/S) boundaries from Ding et al. (2002) are also shown beside the stratigraphic column. The expected ages are based on linear interpolation of the sampling depth between the upper and lower boundaries of the loess or paleosol layer from which the sample was taken.

Figure 1

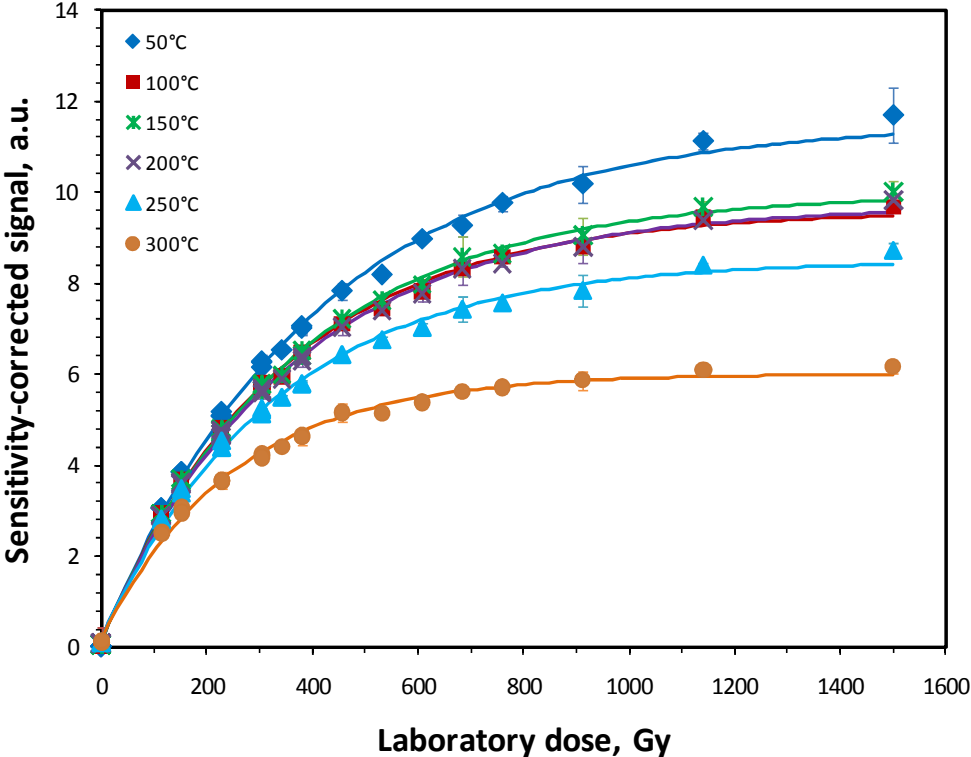


Figure 2

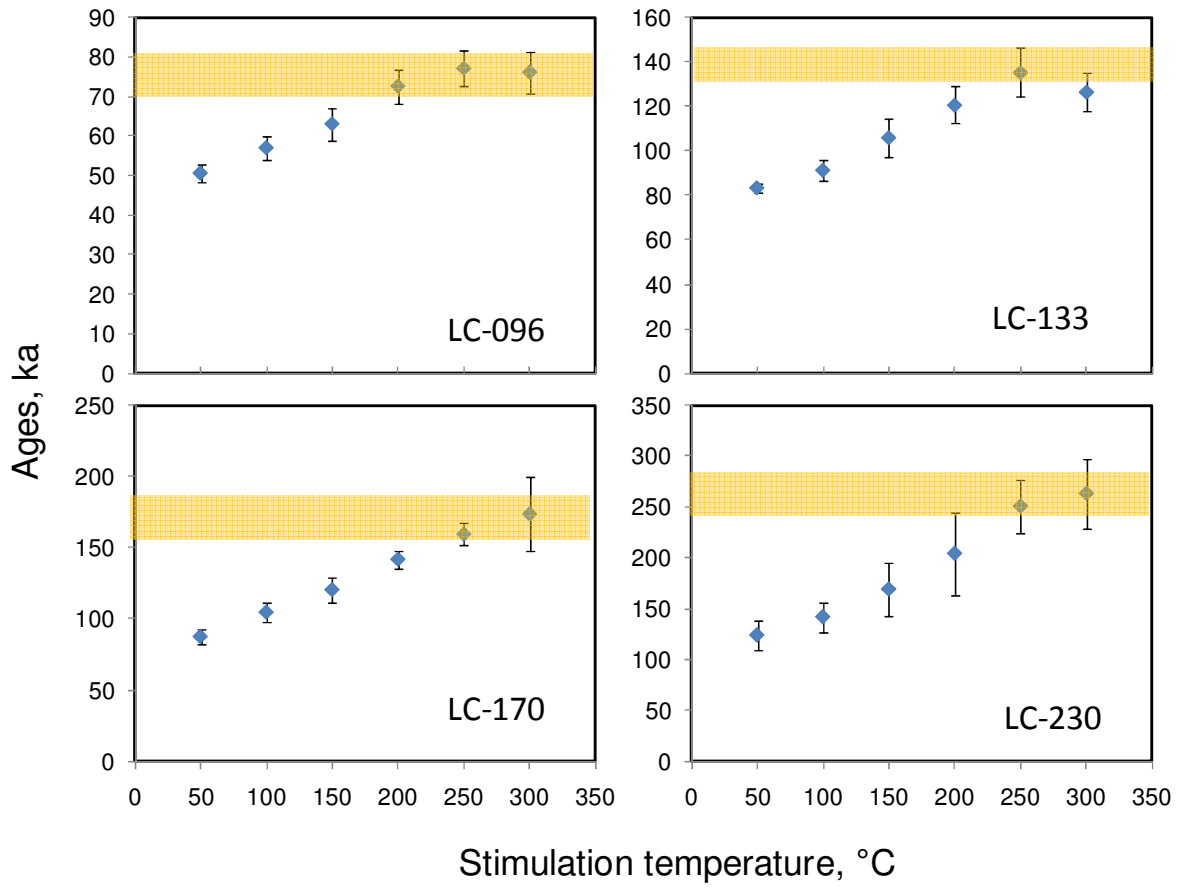


Figure 3

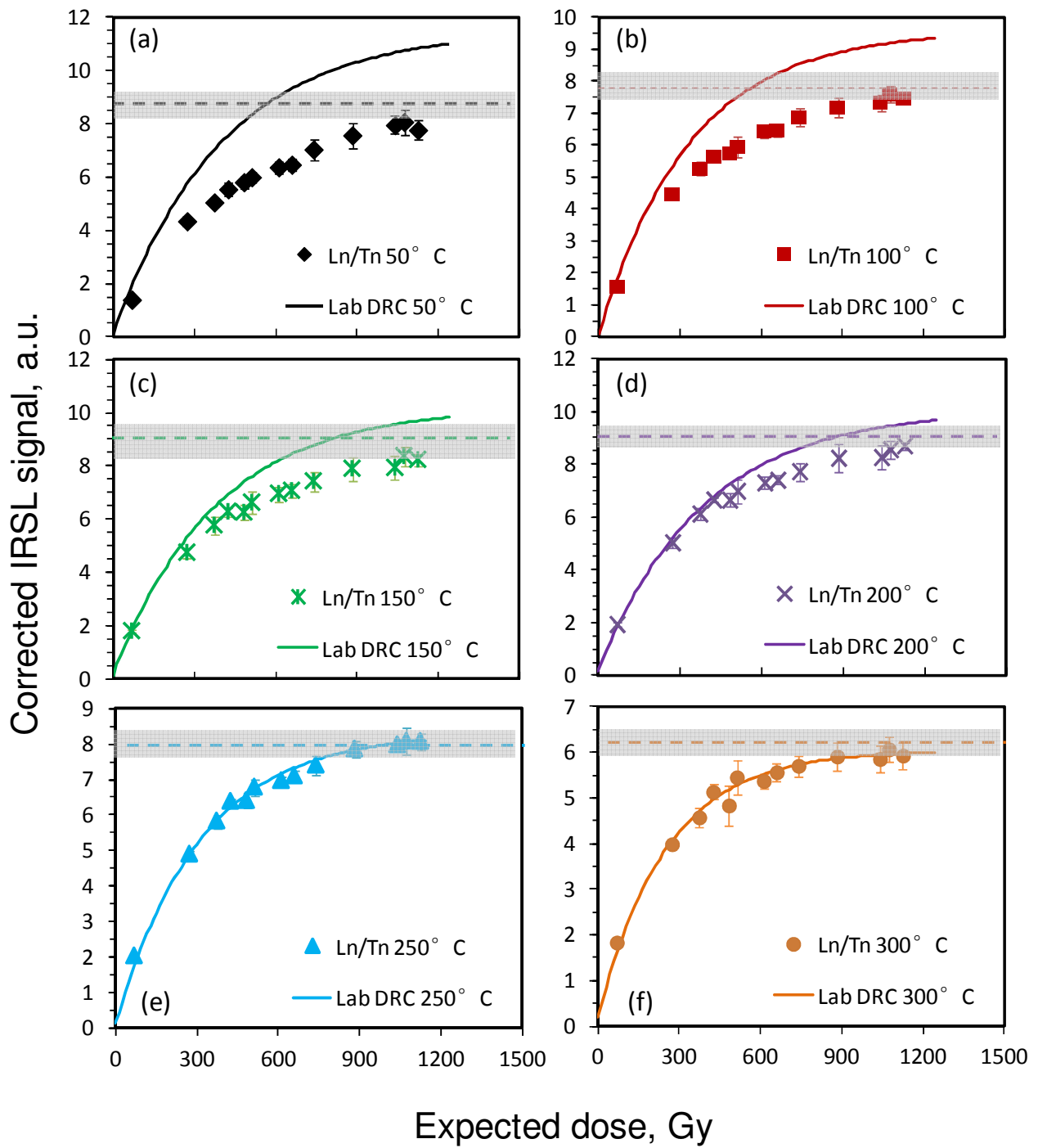


Figure 4

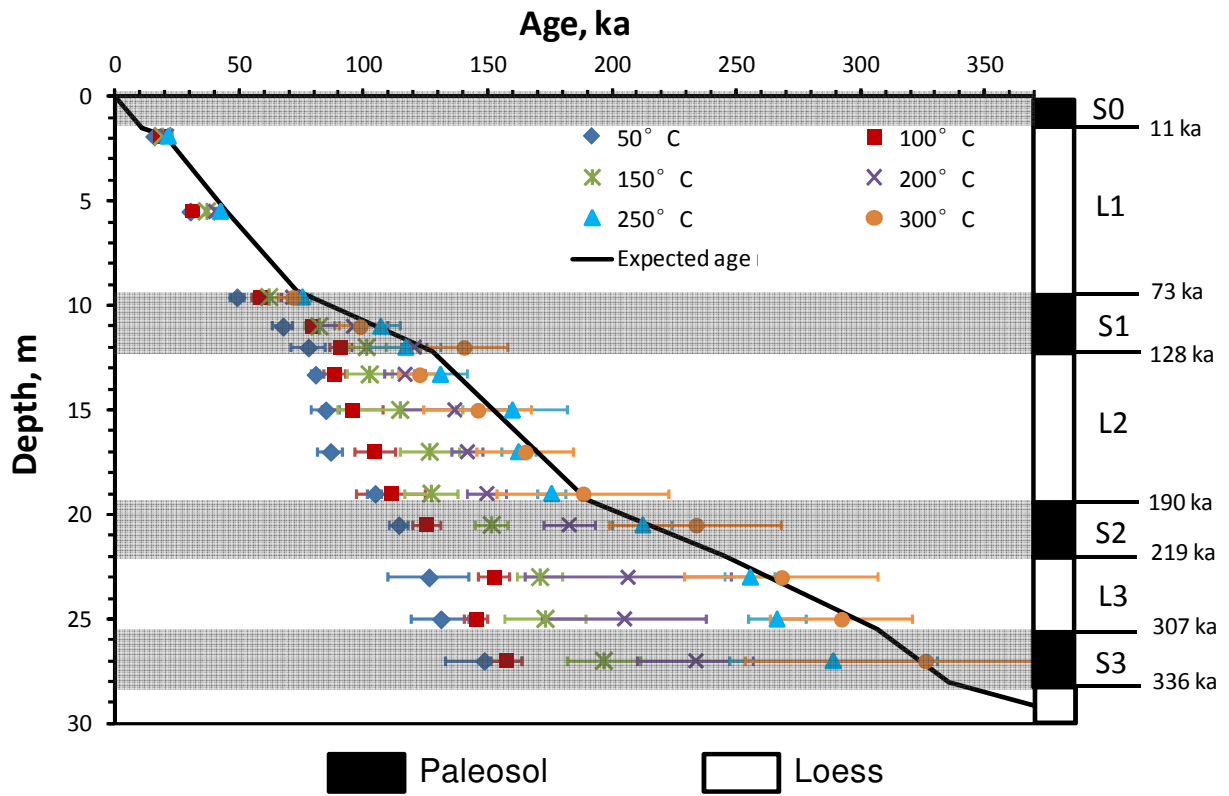


Table 1: Summary of sampling depth, stratigraphic unit and the dosimetry data for the samples.

Sample	Depth (m)	Unit	Grain size ( $\mu\text{m}$ )	K content (%)	Alpha count rate (cts/ks)	Water content (%)	Cosmic rays (Gy/ka)	Ext. dose rate (Gy/ka)	Int. dose rate <sup>a</sup> (Gy/ka)
LC-019	1.9	L1	63-90	1.70	11.38 $\pm$ 0.22	15 $\pm$ 5	0.17	3.1 $\pm$ 0.1	0.36
LC-054	5.4		63-90	1.97	12.33 $\pm$ 0.19	15 $\pm$ 5	0.09	3.3 $\pm$ 0.1	0.36
LC-096	9.6	S1	63-90	2.06	12.86 $\pm$ 0.20	20 $\pm$ 5	0.06	3.3 $\pm$ 0.1	0.36
LC-110	11.0		63-90	1.98	12.66 $\pm$ 0.19	20 $\pm$ 5	0.05	3.2 $\pm$ 0.1	0.36
LC-120	12.0		63-90	2.10	11.37 $\pm$ 0.18	20 $\pm$ 5	0.05	3.1 $\pm$ 0.1	0.36
LC-133	13.3	L2	63-90	1.90	11.65 $\pm$ 0.22	15 $\pm$ 5	0.04	3.2 $\pm$ 0.1	0.36
LC-150	15.0		63-90	1.85	10.97 $\pm$ 0.18	15 $\pm$ 5	0.04	3.0 $\pm$ 0.1	0.36
LC-170	17.0		63-90	2.00	11.77 $\pm$ 0.25	15 $\pm$ 5	0.03	3.2 $\pm$ 0.1	0.36
LC-190	19.0		63-90	1.96	11.43 $\pm$ 0.25	15 $\pm$ 5	0.03	3.2 $\pm$ 0.1	0.36
LC-205	20.5	S2	63-90	1.97	12.19 $\pm$ 0.23	20 $\pm$ 5	0.03	3.1 $\pm$ 0.1	0.36
LC-230	23.0	L3	63-90	1.83	11.17 $\pm$ 0.24	15 $\pm$ 5	0.03	3.0 $\pm$ 0.1	0.36
LC-250	25.0		63-90	1.93	11.46 $\pm$ 0.20	15 $\pm$ 5	0.02	3.1 $\pm$ 0.1	0.36
LC-270	27.0	S3	63-90	1.90	11.54 $\pm$ 0.20	20 $\pm$ 5	0.02	3.0 $\pm$ 0.1	0.36
LC-290	29.0	L4	63-90	1.89	11.77 $\pm$ 0.20	15 $\pm$ 5	0.01	3.1 $\pm$ 0.1	0.36
LC-626	62.6	L9	63-90	-	-	-	-	-	-

Note: <sup>a</sup> The internal dose rate for K-feldspar used in age calculation was estimated by assuming K=13 $\pm$ 1% and Rb=400 $\pm$ 100 ppm (Li et al. 2008; Huntley and Baril, 1997; Zhao and Li, 2005; Huntley and Hancock, 2001).

Table 2: The single-aliquot regenerative-dose (SAR) protocol for multi-elevated-temperatures post-IR IRSL.

MET-pIRIR protocol		
Step	Treatment	Observed
1	Give regenerative dose, $D_i^a$	
2	Preheat at 320°C for 60 s	
3	IRSL measurement at 50°C for 100 s	$L_{x(50)}$
4	IRSL measurement at 100°C for 100 s	$L_{x(100)}$
5	IRSL measurement at 150°C for 100 s	$L_{x(150)}$
6	IRSL measurement at 200°C for 100 s	$L_{x(200)}$
7	IRSL measurement at 250°C for 100 s	$L_{x(250)}$
8	IRSL measurement at 300°C for 100 s	$L_{x(300)}$
9	Give test dose, $D_t$	
10	Preheat at 320°C for 60 s	
11	IRSL measurement at 50°C for 100 s	$T_{x(50)}$
12	IRSL measurement at 100°C for 100 s	$T_{x(100)}$
13	IRSL measurement at 150°C for 100 s	$T_{x(150)}$
14	IRSL measurement at 200°C for 100 s	$T_{x(200)}$
15	IRSL measurement at 250°C for 100 s	$T_{x(250)}$
16	IRSL measurement at 300°C for 100 s	$T_{x(300)}$
17	IR bleaching at 325°C for 100 s	
18	Return to step 1	

<sup>a</sup> For the ‘natural’ sample,  $i = 0$  and  $D_0 = 0$ . The whole sequence is repeated for several regenerative doses including a zero dose and a repeated dose.

Table 3. Summary of the samples used and their corresponding ages obtained using different signals.

Sample	Depth (m)	Unit	Expected age <sup>a</sup> (ka)	Paleodose <sup>b</sup> (ka)	K-feldspar MET-pIRIR ages (ka) <sup>c</sup>						KF ages (ka) <sup>d</sup>
					50°C	100°C	150°C	200°C	250°C	300°C	
LC-019	1.9	L1	20±3	69±10	15.7±0.4	18.4±0.4	19.2±0.4	20.8±0.6	21.5±1.8	-	21.5±1.8
LC-054	5.4		46±4	273±13	30.2±0.7	31.4±1.4	37.0±1	40.5±0.7	42.4±2.8	-	42.4±2.8
LC-096	9.6		75±5	374±18	49.0±2.8	58.6±3.7	62.1±3.9	71.5±4.3	75.5±3.7	71.4±5.5	75.5±3.7
LC-110	11.0	S1	105±5	425±18	67.5±3.9	79.2±1.5	82.3±6.0	96.0±13.5	107.0±7.9	98.6±8.5	107.0±7.9
LC-120	12.0		124±5	483±17	77.7±6.8	90.9±4.4	101.4±7.6	120.5±5.1	117.1±14.3	140.4±17.5	117.1±14.
LC-133	13.3		138±5	512±34	80.6±1.8	88.3±4.5	102.6±8.9	116.8±8.0	131.1±10.8	122.5±8.5	127±7
LC-150	15.0	L2	152±10	612±35	84.8±5.8	95.6±12.2	114.9±25.0	136.8±22.2	160.0±22.4	146.0±21.5	153±16
LC-170	17.0		170±10	660±36	86.7±5.1	104.6±8.2	126.7±11.9	141.9±6.2	162.5±6.8	165.0±19.3	164±10
LC-190	19.0		187±10	740±35	104.6±2.9	111.3±13.8	127.4±10.7	149.7±7.6	175.8±5.5	188.3±34.6	182±18
LC-205	20.5	S2	214±10	883±35	114.1±3.7	125.4±5.8	151.6±6.4	182.8±10.4	212.5±11.9	233.8±34.6	223±18
LC-230	23.0	L3	263±10	1041±34	126.3±16.5	152.7±6.2	171.1±9.0	206.5±41.3	255.7±10.0	268.2±38.8	262±20
LC-250	25.0		298±15	1074±35	131.1±11.6	145.4±4.7	173.3±16.1	205.1±32.9	266.5±11.6	292.3±28.7	279±15
LC-270	27.0	S3	324±15	1125±50	148.5±15.2	157.8±6.4	196.8±14.8	233.8±23.3	289.1±41.7	326.1±72.5	308±42
LC-290	29.0	L4	340±15	1190±33	-	-	-	-	-	-	-
LC-626	62.6	L9	900	3000	-	-	-	-	-	-	-

Note: <sup>a</sup> The expected age of sample LC-054 is based on OSL dating fine-grain quartz (see Fu et al., this issue). For other samples, all expected ages are based on stratigraphic ages (see text for detailed description of the expected age model).

<sup>b</sup> The expected paleodoses are calculated using the expected ages and the dose rates provided in Table 2.

<sup>c</sup> The MET-pIRIR ages for LC-019 and LC-054 are based on the original protocol of Li and Li (2011), in which the stimulation temperatures of 50-250°C were used. The MET-pIRIR ages for LC-290 and LC-626 cannot be obtained because the saturation of the natural signals.

<sup>d</sup> The KF ages in the last column are the best age estimates for the samples. For samples above S1 (<130 ka), the MET-pIRIR 250 °C ages were used, and for older samples the average of the 250 and 300 °C ages were used.