



## A detailed post-IR IRSL dating study of the Niuyangzigou loess site in northeastern China

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1 **A detailed post-IR IRSL dating study of the Niuyangzigou loess site**  
2 **in northeastern China**

3

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6

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8 IRSL dating study of the Niuyangzigou loess site in northeastern China.

9

10 In this study, we report standard quartz SAR OSL and post-IR infrared (IR) stimulated  
11 luminescence (post-IR IRSL; pIRIR<sub>290</sub>) measurements made on sand-sized quartz  
12 and K-feldspar extracts from the loess-palaeosol sequence at Niuyangzigou in  
13 northeastern China. The quartz OSL characteristics are satisfactory. Extensive  
14 pIRIR<sub>50,290</sub> dose recovery tests were performed by adding doses on top of the natural  
15 dose. We found that dose recovery ratios improve significantly when the test dose  
16 ranges between ~15 and ~80% of the total dose, and good dose recovery (within ±5%  
17 of unity) can be obtained up to ~800 Gy. Otherwise, the dose recovery ratio deviates  
18 from unity. The D<sub>e</sub> values also depend on the test dose size so we conclude that the  
19 effect of test dose size should be routinely considered in pIRIR dating. First IR  
20 stimulation plateau pIRIR<sub>290</sub> results are compared with multiple elevated temperature  
21 -pIRIR (MET-pIRIR) data. It appears that the low temperature MET-pIRIR data are  
22 strongly affected by poor dose recovery, but this is not the case for the pIRIR<sub>290</sub>

23 results. Natural signal measurements at the highest (first IR) stimulation temperature  
24 on a sample expected to be in field saturation, suggest ~10% signal loss is present in  
25 pIRIR signals. Long term laboratory bleaching experiments (>80 days) show that a  
26 constant (or very difficult to bleach) residual pIRIR<sub>290</sub> signal is reached after ~300 h,  
27 corresponding to a dose of  $6.2 \pm 0.7$  Gy. Quartz OSL and feldspar pIRIR<sub>50,290</sub> ages are  
28 in good agreement at least back to ~70 ka. Beyond this the quartz ages begin to  
29 underestimate but the feldspar ages are in agreement with the expected Last  
30 Interglacial age palaeosol.

31

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41 Northeastern China is located in the East Asian monsoon region and lies near the  
42 present-day limit of the summer monsoon (Fig 1); as a result it is sensitive to the  
43 global climate systems of both the high and low latitudes. Since the 1980s this area

44 has been characterised by a pronounced regional temperature increase and a  
45 decrease in precipitation compared to the rest of China (Sun *et al.* 2007; Gao *et al.*  
46 2008). Because of these factors it is a very suitable place to study climate change and  
47 environmental evolution during the late Quaternary. The considerable area of loess  
48 deposits in north China forms one of the largest and most important aeolian records  
49 on Earth. Loess/palaeosol sequences contain detailed archives of terrestrial  
50 palaeoenvironmental changes and are highly sensitive to climatic changes,  
51 specifically to shifts in the Asian summer and winter monsoon and/or Northern  
52 Hemisphere westerly circulation (Liu & Ding, 1998). However, research into loess  
53 deposition and past climate change in northeastern China is limited due to the lack of  
54 independent age control (i.e. radiometric dating).

55

56 Luminescence dating has proved to be particularly successful for dating aeolian  
57 sequences (e.g. Stevens *et al.* 2006; Buylaert *et al.* 2008; Lai 2010; Lai & Fan 2014).  
58 These studies are all based on equivalent dose estimation using the single aliquot  
59 regenerative (SAR) dose protocol developed for fast-component dominated quartz  
60 OSL (Wintle & Murray 2006). However, its use is typically limited to samples with  
61 equivalent doses up to 150-200 Gy. This restricts the OSL dating of quartz to loess  
62 deposits (typical dose rate of between 3 and 4 Gy ka<sup>-1</sup>) from within the last 50-70 ka  
63 (Buylaert *et al.* 2007; Roberts 2008; Chapot *et al.* 2012; Timar-Gabor & Wintle 2013).  
64 The infrared stimulated luminescence (IRSL) signal from feldspar (Hütt *et al.* 1988)

65 has the potential to extend the datable age range because it saturates at much higher  
66 doses compared to quartz OSL (Huntley & Lamothe 2001). However, it is now widely  
67 accepted that IRSL measured at ambient temperature suffers from anomalous fading  
68 (e.g. Spooner 1994; Huntley & Lian 2006; Buylaert *et al.* 2011, 2012). Recent  
69 advances in the understanding of feldspar as a luminescence dosimeter (Thomsen *et al.*  
70 *al.* 2008, 2011; Murray *et al.* 2009; Jain & Ankjærgaard 2011) have led to new  
71 single-aliquot regenerative dose (SAR) dating protocols (so called post-IR IRSL  
72 protocols; e.g. Buylaert *et al.* 2009), in which a high temperature IRSL signal is  
73 measured at an increased temperature after a first IR stimulation at some lower  
74 temperature, usually close to ambient temperature. These post-IR IRSL (pIRIR)  
75 signals appear to be much less prone to fading than the conventional IRSL signal.  
76 Various single-aliquot based pIRIR dating protocols for feldspar have been developed;  
77 these include a two-step (e.g. Thomsen *et al.* 2008; Buylaert *et al.* 2009; Thiel *et al.*  
78 2011a) and a multiple elevated temperature (MET) (Li & Li 2011; 2012a) pIRIR  
79 stimulation procedure. These procedures have been shown to give accurate ages  
80 both for young (<10 ka, Fu & Li 2013; Reimann *et al.* 2011, 2012) and old (>100 ka,  
81 Buylaert *et al.* 2012; Li & Li 2011, 2012a; Kars *et al.* 2012; Zander & Hilgers 2013)  
82 samples. Even though pIRIR measurement protocols have become the preferred  
83 method to measure feldspar equivalent doses, there are still remaining issues  
84 concerning dose recovery results (Roberts 2012), the determination of a potentially  
85 unbleachable component (e.g. Buylaert *et al.* 2011, Stevens *et al.* 2011; Murray *et al.*

86 2012; Kars *et al.* 2014a) and signal (in)stability (Li & Li 2012b; Thomsen *et al.* 2011).

87

88 The present work focuses on the Niuyangzigou (NYZG) loess-palaeosol  
89 sequence in northeastern China (Fig. 1). The luminescence characteristics of quartz  
90 SAR OSL and K-feldspar pIRIR signals (pIRIR<sub>290</sub>) are documented in a SAR-based  
91 methodology. The pIRIR signal measured at 290°C is then investigated in detail to (i)  
92 test the dependence of dose recovery and D<sub>e</sub> on test dose size, (ii) determine the size  
93 of an (un)bleachable residual component and (iii) check the stability of the signal  
94 (including MET-pIRIR signals) using a sample expected to be of non-finite age. Finally,  
95 quartz OSL and pIRIR<sub>290</sub> ages are compared and a luminescence chronology for the  
96 upper part of the NYZG section is presented.

97

## 98 **Geological setting, stratigraphy and sampling**

99

100 Northeastern China extends from 40° to 59° N and 110 to 135° E, it includes the  
101 provinces of Heilongjiang, Jilin and Liaoning, and the Inner Mongolia Autonomous  
102 Region east of 110° E (Fig. 1). The major topographical features in this area are the  
103 extensive Northeast Plain surrounded by a series of mountains and hills. The  
104 Daxingan Mountains lie along the western side while the Xiaoxingan Range with a  
105 northwest-southeast orientation lies to the north.

106

107 The NYZG section (41°55' N, 118°43' E, 774 m.a.s.l) is situated in KaLaQin  
108 County, Chifeng city, in the northeastern part of the Inner Mongolia Autonomous  
109 Region (Fig. 1). The whole section is a 36.6 m thick series of loess intercalated by  
110 palaeosols; it is accessible through a natural exposure of the upper 26 m and a 10.6  
111 m deep exploratory well. The magnetic characteristics of the Matuyama–Brunhes  
112 palaeomagnetic boundary (0.78 Ma; Cande & Kent 1995) have been identified  
113 towards the bottom of the sequence and some evidence for the Jaramillo sub-chron is  
114 exposed in a basal complex of palaeosols (Zeng *et al.* 2011).

115

116 Fifteen luminescence samples were collected using light-tight steel cylinders  
117 (diameter 5 cm, length 20 cm) from the upper 3.2 m of a freshly excavated profile.  
118 Based on field observations, this section comprises the upper Holocene soil (S<sub>0</sub>), the  
119 Last Glacial loess (L<sub>1</sub>) and the Last Interglacial palaeosol (S<sub>1</sub>) (Fig.1). One additional  
120 sample (sample code 1535) was collected near the B/M boundary (Zeng *et al.* 2011)  
121 to provide a feldspar sample of non-finite luminescence age (expected burial  
122 dose >2000 Gy).

123

## 124 **Sample preparation and analytical facilities**

125

126 Samples were opened under subdued red light conditions and material from the outer  
127 ends of each tube was used for dose rate and water content measurement. The

128 non-light exposed material from the middle part of the tube was treated with HCl (30%)  
129 and H<sub>2</sub>O<sub>2</sub> (30%) to remove carbonates and organic matter, respectively. Grains in the  
130 range 63 to 90 µm were obtained by wet-sieving. Pure quartz grains (no significant  
131 IRSL signals) were obtained after a 40 min HF (40%) etch and 40 min 10% HCl rinse.  
132 For K-rich feldspar extraction, a portion of the initial 63-90 µm sieved fraction was  
133 cleaned with 10% HF for 20 min to remove coatings and the outer alpha irradiated  
134 layer, and then rinsed in 10% HCl acid for 20 min to remove any precipitated fluorides.  
135 K-rich feldspars were floated off using an aqueous heavy liquid (sodium  
136 heteropolytungstate 'LST Fastfloat'; density 2.58 g.cm<sup>-3</sup>).

137

138 Luminescence measurements employed Risø TL/OSL readers model DA-20  
139 (Bøtter-Jensen *et al.* 2003) equipped with blue LEDs (470 nm, ~80 mWcm<sup>-2</sup>) infrared  
140 (IR) LEDs (870 nm, ~135 mWcm<sup>-2</sup>); each reader was equipped with a calibrated <sup>90</sup>Sr  
141 /<sup>90</sup>Y beta source. Quartz OSL signals were collected through 7.5 mm of Schott U-340  
142 (UV) glass filter and feldspar (post-IR) IRSL through a combination of Corning 7-59  
143 and Schott BG-39 glass filters (blue-violet part of the spectrum). Quartz grains were  
144 mounted as large (8 mm) aliquots on stainless steel discs and K-rich feldspars as  
145 small (2 mm) aliquots on stainless steel cups; Silicone oil (Silkospray) was used as an  
146 adhesive. For quartz OSL, the signal was derived from the first 0.16 s of stimulation  
147 and an early background (0.16-0.32 s) to minimize the influence of slow and medium  
148 components (Ballarini *et al.* 2007; Cunningham & Wallinga 2010). Feldspar (post-IR)



149 IRSL signals were derived from the integral of the first 2 s of (post-IR) IRSL  
150 stimulation, less a background based on the last 50 s; for measurements made using  
151 an MET-pIRIR protocol these intervals are 1 and 25 s, respectively.

152

## 153 **Dosimetry**

154

155 The environmental dose rate was calculated from the uranium, thorium and  
156 potassium concentrations, measured by neutron activation analysis (NAA). The *in*  
157 *situ* water content (mass of moisture/dry mass) was determined by weighing the  
158 sample before and after drying, and was assigned an absolute uncertainty of  $\pm 5\%$   
159 (e.g. for a water content of 10% we have used  $(10 \pm 5)\%$ ). Using the revised dose rate  
160 conversion factors of Guérin *et al.* (2011) and water content attenuation factors  
161 (Aitken 1985), the elemental concentrations were converted into effective dose rate.  
162 Calculation of the cosmic dose rate is based on Prescott & Hutton (1994). For  
163 K-feldspar dose rates a K concentration of  $12.5 \pm 0.5\%$  and Rb concentration of  
164  $400 \pm 100$  ppm was assumed (Huntley & Baril 1997) consistent with earlier work on  
165 sand-sized K-feldspar from Chinese loess (Zhao & Li 2005; Li *et al.* 2008). A small  
166 internal dose rate contribution from U and Th of  $0.03 \pm 0.015$  Gy  $\text{ka}^{-1}$  and  $0.06 \pm 0.03$  Gy  
167  $\text{ka}^{-1}$  was included for quartz and K-feldspar respectively (Mejdahl 1987; Zhao & Li  
168 2005; Vandenberghe *et al.* 2008). Table 1 summarises the uranium, thorium and

169 potassium concentrations and the resulting total dose rates to quartz and K-feldspar  
170 grains

171

## 172 **Quartz OSL characteristics and resulting ages**

173

174 The quartz equivalent doses ( $D_e$ ) were measured using a standard SAR protocol  
175 (Murray & Wintle 2000, 2003; Table 2). Typical dose response curves and OSL decay  
176 curves (inset) are shown for the upper (138101) and lower (138115) samples in Fig.  
177 2A and B, respectively. The blue-light stimulated OSL signals decrease very quickly  
178 during the first second of stimulation, indicating that the signal is dominated by the  
179 fast component (Jain *et al.* 2003; Singarayer & Bailey 2003). In order to select  
180 appropriate preheat conditions, the  $D_e$  dependence on preheat temperature was  
181 checked using a preheat plateau test. A plateau was observed for temperatures from  
182 180 °C to at least 260 °C (Fig. 2C). The suitability of our adopted SAR protocol was  
183 further checked with a dose recovery test (Murray & Wintle 2003). The ratios of the  
184 given doses to the measured doses were within 10% of unity over the entire  
185 temperature interval (Fig. 2D). Based on these preheat tests, a 260 °C preheat for 10  
186 s and 220 °C cut-heat was chosen for final  $D_e$  determination. For all samples,  
187 recuperation is low (average =  $0.06 \pm 0.01\%$  of natural,  $n = 245$ ) and the average  
188 recycling ratio is  $1.01 \pm 0.01$  ( $n = 233$ ) indicating that the adopted SAR protocol  
189 successfully corrects for laboratory sensitivity changes.

190

191 In order to ensure that only those aliquots that are capable of measuring the  
192 dose of interest have been included in the calculation of the mean  $D_e$ , aliquots with  
193  $2 \times D_0$  values that are smaller than the average dose (derived from all aliquots) are not  
194 used in the derivation of  $D_e$ , irrespective of the  $D_e$  value of the individual aliquot  
195 (based on Thomsen *et al.* 2016; note that for simplicity our data were approximated  
196 by a single saturating exponential function). For the upper four samples (<150 Gy) no  
197 aliquots were rejected based on this criterion. Below this level (1.20 m) the  
198 percentage of aliquots that are rejected ranges between 6 and 31%. Over all the 15  
199 samples the average  $D_e$  was decreased by ~3% because of this selection criterion;  
200 the biggest decrease was 16% and the largest increase was only 2%. Table 1  
201 summarises the resulting  $D_e$  values and quartz OSL ages. The calculated  $D_e$  values  
202 range from  $92 \pm 3$  Gy for the upper sample to  $270 \pm 13$  Gy for the lowest sample; the  
203 corresponding ages are  $30 \pm 2$  and  $99 \pm 7$  ka. We note that the bottom four samples all  
204 have  $D_e$  values  $\geq 200$  Gy. Several papers suggest that the upper limit of SAR based  
205 quartz OSL dating lies around ~200 Gy in loess and ages derived from these high  
206 doses should be interpreted with caution because they are likely to be  
207 underestimates (e.g. Buylaert *et al.* 2007, 2008; Zhou & Shackleton 2001; Lai 2010;  
208 Chapot *et al.* 2012, Timar-Gabor & Wintle 2013; Lai & Fan 2014).

209

## 210 **Feldspar luminescence characteristics**

## 211 ***Dose response curve and $L_x/T_x$ plots***

212 The SAR pIRIR<sub>290</sub> protocol proposed by Thiel *et al.* (2011a) and tested by Buylaert *et*  
213 *al.* (2012) was used to measure the K-feldspar dose in these samples (Table 2).  
214 Aliquots were preheated at 320 °C for 60 s followed by IR diode stimulation (90%  
215 power) at 50 °C for 200 s (the choice of this temperature is discussed in the next  
216 section) to recombine nearby electron-hole pairs (Jain & Ankjærgaard 2011).  
217 Subsequently, a more stable IRSL signal is measured at 290 °C for 200 s (referred to  
218 as pIRIR<sub>290</sub>); this is the dating signal of interest. The response to the test dose is  
219 measured in the same manner and is followed by an IR illumination at 325 °C for 200  
220 s at the end of each SAR cycle, to reduce recuperation. Representative K-feldspar  
221 pIRIR<sub>290</sub> and quartz OSL dose response curves (normalised to the fitted saturation  
222 values) are presented for the lowermost sample (138115) in Fig. 3A. It can be seen  
223 that the feldspar pIRIR<sub>290</sub> dose response curve has a much more extended dose  
224 range compared to quartz OSL, 86% of saturation is reached at 1250 and 310 Gy,  
225 respectively. This extended feldspar range indicates its usefulness for dating samples  
226 beyond the quartz OSL limit.

227

228 One of the main assumptions when using the SAR protocol to measure a dose is  
229 that the test dose luminescence sensitivity is directly proportional to the preceding  
230 regenerative dose; i.e. it can correct for sensitivity changes (Murray & Wintle 2000).  
231 We have constructed  $L_x$ - $T_x$  plots for the pIRIR<sub>290</sub> signal for five samples down our

232 section by repeating SAR cycles using a fixed regenerative dose and test dose (Fig.  
233 3B); there is clear proportionality between regenerative and test dose signals  
234 indicating that the test dose successfully corrects for sensitivity changes. This is also  
235 supported by the mean recycling ratio of  $1.031 \pm 0.002$  ( $n = 109$ , RSD = 2%; 15  
236 samples). If no test dose is used for sensitivity correction, the resulting recycling ratio  
237 is  $1.099 \pm 0.011$  with a much higher standard deviation of 10%. For all samples,  
238 recuperation is small (<3% of the natural signal) showing that our high temperature  
239 clean-out is sufficiently stringent. However, the most stringent test for any SAR  
240 protocol is the dose recovery test (Murray 1996; Wallinga *et al.* 2000; Murray & Wintle  
241 2003) and this is addressed in the next section.

242

### 243 ***Dose recovery test***

244 Although several studies have presented good/acceptable dose recovery results for  
245 pIRIR<sub>290</sub> protocols on a variety of sediments (e.g. Buylaert *et al.* 2011, 2012, 2013;  
246 Nian *et al.* 2012; Thiel *et al.* 2012; Tsukamoto *et al.* 2014), there is also considerable  
247 evidence for poor dose recovery results (e.g. Stevens *et al.* 2011; Lowick *et al.* 2012;  
248 Roberts 2012; Thiel *et al.* 2011b, 2014 ; Murray *et al.* 2014). Usually, these poor dose  
249 recovery ratios were significantly greater than unity. Some authors report difficulties in  
250 bleaching natural pIRIR<sub>290</sub> signals (using SOL2 simulator or natural daylight); these  
251 can result in poor dose recovery because of incorrect residual dose estimation (e.g.  
252 Stevens *et al.* 2011; Alexanderson & Murray 2012). To avoid potential complications

253 related to bleaching natural samples, a dose recovery test can also be performed on  
254 modern/young samples by adding a beta dose on top of a relatively small natural  
255 dose (e.g. Buylaert *et al.* 2011); this is the approach taken in this study.

256

257 We investigated the dependence of dose recovery ratios on test dose size by  
258 adding beta doses (ranging from 99 to 1593 Gy) to aliquots of the uppermost sample  
259 (138101) which has a pIRIR<sub>290</sub> D<sub>e</sub> value of ~100 Gy. The dose recovery ratio was  
260 calculated as the measured dose divided by the sum of the natural and the given  
261 dose. Qin & Zhou (2012) suggested that the dose recovery ratio is dependent on the  
262 test dose size (their Fig. 3B) but their data are limited in test dose range and  
263 inconclusive (the data for different test dose sizes does not differ significantly). Fig 4A  
264 shows the dose recovery ratio as a function of test dose size over a wide test dose  
265 range (5-260% of the total (natural+added) dose). For small test doses (<15%), the  
266 dose recovery ratio is significantly greater than unity. In contrast, large test doses  
267 (>80%) yield ratios lower than unity. The best dose recovery ratios (within ±5% of  
268 unity) are found when the test dose ranges between ~15 and ~80% of the total dose.  
269 The data of Fig. 4A are also shown in Fig. 4B but now as a measured versus added  
270 dose plot; these are so-called Single Aliquot Regenerative Added dose (SARA;  
271 Mejdahl & Bøtter-Jensen 1994; Wallinga *et al.* 2001) dose response curves. As  
272 expected from the dose recovery ratios, the slope ( $1.01 \pm 0.02$ ) of the fitted line is  
273 indistinguishable from unity when test doses between 15 and 80% are used. It is

274 interesting to note that at least for added doses larger than about 600 Gy, the data  
275 measured with small (<15%) or large (>80%) test doses do not follow a simple  
276 straight line relationship between measured and added dose, rather the measured  
277 dose increasingly deviates from the known added dose; this occurs because of a  
278 systematic change in  $D_0$  with test dose.

279

280 We conclude from the data of Fig. 4 that, at least for these samples, an  
281 acceptable dose recovery ratio is best ensured by using a test dose in the range  
282 15-80% of the total (natural + added) dose.

283

#### 284 ***Equivalent dose***

285 ***Effect of test dose size.*** Fig. 5 shows the dependence of  $D_e$  on test dose size for the  
286 uppermost (138101), middle (138108) and lowermost (138115) samples from the  
287 section. Sample 138101 shows a clear  $D_e$  plateau for test doses ranging between 5  
288 and 80% of the  $D_e$  value; the  $D_e$  value of the older samples appears insensitive to test  
289 dose size over a shorter range, up to only ~60%. The shape of the dose recovery  
290 ratio versus test dose curve (Fig. 4A) broadly resembles the shape of the  $D_e$  versus  
291 test dose curve but it appears that especially for low test doses (<20%) the  $D_e$  value is  
292 less sensitive to test dose size than the dose recovery value. Nevertheless, for these  
293 samples, it appears to be inappropriate to use very small (<10%) or very large (>60%)  
294 test doses because of poor dose recovery ratios. For the remainder of this study we

295 adopt a test dose size of ~30% for  $D_e$  measurements; in this range the dose recovery  
296 ratio is within  $\pm 5\%$  of unity and the  $D_e$  values are consistent with the plateau regions  
297 in  $D_e$  versus test dose graphs.

298

299 **Comparison with MET-protocol.** Based on the model prediction by Jain &  
300 Ankjærgaard (2011) that the stability of the post-IR IRSL signal could be dependent  
301 on the first IR stimulation temperature and/or wavelength, Buylaert *et al.* (2012) have  
302 suggested the use of a first IR stimulation temperature plateau to investigate whether  
303 a stable signal was reached (interval over which  $D_e$  is insensitive to prior IR  
304 stimulation temperature). Fig. 6A,B shows  $D_e$  as a function of prior IR stimulation  
305 temperature for the top (138101) and bottom (138115) samples of the section,  
306 respectively; from these data it seems that for  $D_e$  values up to ~400 Gy the  $D_e$  is  
307 insensitive to first IR stimulation temperature and a stable signal is observed. This is  
308 in agreement with the data of Li & Li (2012b) who compared pIRIR<sub>50,290</sub>  $D_e$  values with  
309 pIRIR<sub>200,290</sub>  $D_e$  values and MET-pIRIR<sub>250</sub> results for Luochuan samples and showed  
310 that the three methods are indistinguishable back to ~400 Gy; they also showed that  
311 beyond this a low temperature IR cleaning at 50 °C is apparently not sufficient to  
312 recombine all nearby electron-hole pairs. Beyond ~400 Gy, pIRIR<sub>200,290</sub> is consistent  
313 with the MET-250 data. It is claimed by Li & Li (2011, 2012a, b) and Fu & Li (2013)  
314 that the MET-pIRIR protocols have an advantage over the two-step protocols  
315 (Buylaert *et al.* 2009, 2012; Thiel *et al.* 2011a) because of the possibility to construct



316 “age-temperature” plots from individual aliquots; the presence of an “age plateau” at  
317 higher stimulation temperatures is used to determine whether a stable signal was  
318 identified. We have also measured the MET-pIRIR signals (following Li & Li, 2012 a)  
319 for the top and bottom sample of our section and the data are also shown in Fig. 6A,B  
320 (open symbols). As expected the MET-pIRIR  $D_e$  values are consistent with the  
321 pIRIR<sub>290</sub>  $D_e$  measurements when stimulation temperatures of >200 °C are reached. It  
322 should be noted that the plateau identified by Li & Li (2011, 2012a) and Fu & Li (2013)  
323 is usually limited to two (or maximum three) datapoints over a limited temperature  
324 interval of 50-100 °C (e.g. Li & Li, 2011, 2012a). The same observation is made here.  
325 For sample 138101 we have carried out a dose recovery test as a function of prior IR  
326 stimulation temperature (Fig. 6C). It can be seen that dose recovery is satisfactory  
327 over a wide prior IR stimulation temperature interval between 50 and 260 °C for the  
328 pIRIR<sub>290</sub> data. This is in contrast with the MET-pIRIR dose recovery data which shows  
329 a pronounced increase in of dose recovery between 50 and 150 °C IR stimulation  
330 temperature; the shape is very similar to the MET-pIRIR  $D_e$  data shown in Fig. 6A for  
331 the same sample. We conclude that the MET-pIRIR  $D_e$  data are significantly affected  
332 by dose recovery problems at low IR stimulation temperatures (a suggestion to this  
333 effect was made in Li & Li 2011). This is consistent with the observations of Kars *et al.*  
334 (2014b) who showed that high preheats (>300 °C) should not be used with low  
335 temperature IR stimulations. Because of this, we suggest using first IR stimulation

336 temperature plateaus instead of the MET-method to determine whether a more stable  
337 signal has been reached.

338

339 **Stability.** Thiel *et al.* (2011a) were the first to show a natural pIRIR<sub>290</sub> signal in  
340 saturation on the laboratory dose response curve for a sample collected below the  
341 B/M boundary; this led them to conclude that the pIRIR<sub>290</sub> signal is a stable signal.  
342 Other studies have made the same observation for pIRIR<sub>290</sub> (Buylaert *et al.* 2011;  
343 Thomsen *et al.* 2011) and the high-temperature MET-pIRIR signals (Li & Li 2011,  
344 2012a). In order to investigate this for our material, we collected a sample (code 1535,  
345 27.30 m) below the B/M boundary defined using palaeomagnetic measurements  
346 (Zeng *et al.* 2011). The expected burial dose is >2000 Gy and all natural signals are  
347 expected to be in field saturation.

348

349 Natural signals and dose response curves were measured for both the pIRIR<sub>290</sub>  
350 signals (using different prior-IR stimulation temperatures) and the MET-pIRIR signals.  
351 The natural signals are shown in Fig. 7, expressed as a fraction of the saturation level  
352 of the laboratory dose response curves. Note that for simplicity a single saturating  
353 exponential was fitted through the data; Guralnik *et al.* (2015) have shown that for  
354 their sample a single exponential fit gives a  $D_0$  indistinguishable from that obtained  
355 from their theoretically based generalised growth curve. Because most earlier work

356 tended to use relatively small test doses, in this experiment we use both a large (500  
357 Gy, approximately equal to  $D_0$ ) and a small (50-60 Gy) test dose.

358 The results obtained using a 500 Gy test dose are discussed first (Fig. 7A). For  
359 the pIRIR<sub>290</sub> protocol, lower first-IR stimulation temperatures (50-140 °C) yield  
360 fraction of saturation values of less than 0.90 (average =  $0.87 \pm 0.01$ ) and higher  
361 temperatures (170-260 °C) yield higher values (average =  $0.92 \pm 0.01$ ). The  
362 MET-pIRIR data measured with a 500 Gy test dose does not seem to show a clear  
363 plateau at higher temperature (previously reported at 250-300 °C; see Fig. 3 in Li & Li  
364 2012a). At 250 and 300 °C the fraction of saturation is  $0.93 \pm 0.01$  and  $0.965 \pm 0.004$ ,  
365 respectively. Both for pIRIR<sub>290</sub> (measured with prior IR stimulation  $\geq 170$  °C) and the  
366 MET-pIRIR protocols, the data are not consistent with unity ( $\sim 4\%$  below saturation  
367 light level for MET and  $\sim 8\%$  for pIRIR<sub>290</sub>) which might suggest instability in the  
368 pIRIR<sub>290</sub> signal and the high-temperature MET-pIRIR signals. For the pIRIR<sub>290</sub> signals  
369 measured with low prior-IR stimulation temperatures this underestimation increases  
370 to  $\sim 13\%$ .

371

372 Fig. 7B shows the results when a small test dose of 50-60 Gy is used. For both  
373 pIRIR<sub>290</sub> (only measured at 50 and 200°C prior-IR stimulation temperature) and the  
374 MET-pIRIR signals the fraction of saturation appears to have increased significantly.  
375 For the pIRIR<sub>200,290</sub> this value is  $0.99 \pm 0.02$ , consistent with the MET-pIRIR signals  
376 measured at 250 and 300 °C ( $0.96 \pm 0.02$ ); this could be interpreted as suggesting

377 negligible instability. However, given the test dose dependence of the dose recovery  
378 and  $D_e$  results, it is not surprising that the fraction of saturation reached by a natural  
379 signal is also a function of test dose size. One must be critical of saturation  
380 measurements made with a test dose of only 60 Gy ( $\ll 10\%$  of the natural dose). The  
381 measurements made with a 500 Gy test dose ( $\sim D_0$ ) are more likely to yield accurate  
382  $D_e$  measurements and so more accurate measurements of saturation level. We  
383 conclude that the apparent stability of signals (e.g. Fig. 1 in Buylaert *et al.* 2012 -  
384 pIRIR; Li & Li 2012a – MET-pIRIR) measured with a small test dose is likely to be an  
385 artefact of the dose recovery dependence on test dose size, and that the small but  
386 finite apparent instability (up to  $\sim 8\%$  below saturation) observed at saturation using a  
387 large test dose is probably real.

388

389 To further investigate the stability of pIRIR signals we have measured fading  
390 rates ( $g_{2\text{days}}$  values) on sample 138106 using a 50 and 200 °C prior-IR stimulation  
391 temperature. The resulting  $g_{2\text{days}}$  values are  $0.75 \pm 0.10\%$ /decade ( $n = 6$ ) and  
392  $0.37 \pm 0.13\%$ /decade ( $n = 6$ ) for pIRIR<sub>50,290</sub> and pIRIR<sub>200,290</sub> respectively. It appears  
393 that the pIRIR<sub>200,290</sub> fading rate is lower than the pIRIR<sub>50,290</sub> fading rate; this is  
394 consistent with the saturation measurements on sample 1535 presented earlier. Li &  
395 Li (2012b) have also argued that pIRIR<sub>200,290</sub> is more stable than pIRIR<sub>50,290</sub>.

396

397 ***Bleaching characteristics***

398 **Comparison of quartz OSL, feldspar IR<sub>50</sub> and pIRIR<sub>290</sub> bleaching rates.** Fig. 8A  
399 summarises the quartz OSL, IR<sub>50</sub> and pIRIR<sub>290</sub> ages for all 15 samples. All the IR<sub>50</sub>  
400 data are consistent with a smooth curve passing through the origin and lying below  
401 the 1:1 line; this is what would be expected from samples for which both signals are  
402 well-bleached at deposition but for which the IR<sub>50</sub> signal is significantly unstable  
403 compared to pIRIR<sub>290</sub> (Buylaert *et al.* 2013). This result is not surprising given the  
404 prolonged and almost ideal light-exposure received by wind-blown dust before final  
405 deposition. Note that this relationship does not arise because of a dose-dependent  
406 change in initial sensitivity of the IRSL signals (see Fig. S1). Although the scatter in  
407 the quartz and pIRIR age relationship is larger, the data are consistent with the fitted  
408 curve and there is again no evidence for pIRIR<sub>290</sub> outliers at larger ages.

409  
410 To illustrate the likely initial conditions of this material had it not been well-bleached,  
411 we exposed quartz and feldspar aliquots of the oldest sample (138115) to a SOL2  
412 spectrum at a lamp-sample distance of ~80 cm for various lengths of time. It took  
413 ~130 s for the pIRIR<sub>290</sub> dose, ~20 s for the IR<sub>50</sub> dose and <2 s for the quartz OSL  
414 dose to be reduced by 50%. The resulting residual dose are shown as open circles  
415 (IR<sub>50</sub>) and open stars (quartz OSL) in Fig. 8B, plotted against the residual pIRIR<sub>290</sub>  
416 doses. The differential bleaching rates of the three signals are obvious and each  
417 dataset has been fitted with a single and double exponential; when the residual  
418 quartz OSL dose is less than 10% of its initial value the IR<sub>50</sub> dose is ~55% of its initial

419 value and the pIRIR<sub>290</sub> is >80%, comparable to the results described by Murray *et al.*  
420 (2012). As expected both curves point at the origin – sufficient light exposure will  
421 bleach all three signals so that the apparent residual doses are close to zero. Had our  
422 loess samples been incompletely bleached in nature at deposition then the observed  
423 IR<sub>50</sub> and pIRIR<sub>290</sub> doses would have grown along a line parallel to the solid line but  
424 starting from some point on the dashed line passing through the open circles,  
425 depending on the degree of incomplete bleaching. Given the inevitable variation in  
426 the degree of incomplete bleaching with time this would have resulted in scattered  
427 points lying below the observed solid line (as was seen by Buylaert *et al.* 2013). Since  
428 our data do not show such scatter we conclude that the pIRIR<sub>290</sub> signals (and thus the  
429 IR<sub>50</sub> and quartz OSL signals) from these samples were all well-bleached at  
430 deposition.

431

432 **Residual component.** Thomsen *et al.* (2008) and Buylaert *et al.* (2009) have shown  
433 that some pIRIR signals can be readily bleached (with residual doses <2 Gy); these  
434 levels are insignificant when older sediments are dated. However, more recent work  
435 has found a much wider ranges in residual doses (2 to >20Gy) after natural and  
436 laboratory bleaching of the pIRIR<sub>290</sub> signal (Thiel *et al.* 2011a, b; Buylaert *et al.* 2011,  
437 2012; Stevens *et al.* 2011; Lowick *et al.* 2012; Murray *et al.* 2012, 2014; Sohbaty *et al.*  
438 2012; Kars *et al.* 2014a). The curve fitted through the open circles in Fig. 8B suggests  
439 that it is possible to bleach the pIRIR<sub>290</sub> signals to relatively small doses but it is not

440 clear whether there may be an unbleachable component. To investigate the degree to  
441 which our pIRIR<sub>290</sub> signal is bleachable, we have repeated the experimental  
442 procedures described by Sohbati *et al.* (2012). A set of 225 aliquots was prepared for  
443 each of three samples from the top, middle and bottom of the of the loess sequence  
444 (138102, 138108, 138115). These were exposed in groups of three to twelve aliquots  
445 per sample for various lengths of time (3.7 to 4171 h) in a Hönle SOL2 simulator at a  
446 lamp-sample distance of 80 cm. The apparent dose was measured using the pIRIR<sub>290</sub>  
447 protocol. The results are summarized in Fig. 9A (pIRIR<sub>290</sub>) and Fig. 9C (IR<sub>50</sub>). There is  
448 a clear tendency for the residual doses to increase with equivalent dose ( $D_e$ ).  
449 Extrapolation of these residual doses to a  $D_e$  of zero Gy can be used to predict the  
450 average residual dose that would have been present in these samples before burial  
451 had they been exposed to these light levels (Buylaert *et al.* 2012; Sohbati *et al.* 2012;  
452 Kars *et al.* 2014a). The inset to Fig. 9A shows this relationship for all 15 samples for a  
453 15 h Sol2 bleach. The intercepts of the three samples bleached for various lengths of  
454 time are plotted against exposure time in Fig. 9B,D for pIRIR<sub>290</sub> and IR<sub>50</sub> signals,  
455 respectively. In both cases this residual appears to be consistent with a constant (or  
456 very difficult to bleach) dose after a bleaching time of ~300 h. In our view it is likely  
457 that these apparently constant difficult-to-bleach signals were present in these  
458 samples at the time of deposition and accordingly a pIRIR<sub>290</sub> residual dose of  $6.2 \pm 0.7$   
459 Gy has been subtracted from all measured  $D_e$  values used to calculate the pIRIR<sub>290</sub>  
460 ages given in Table 1.

461

462 **A chronology for the NYZG section: comparison of quartz and**  
463 **feldspar ages**

464

465 Fig. 10A presents the quartz and pIRIR<sub>290</sub> feldspar ages on a simplified section of the  
466 NYZG site. All of the quartz and feldspar ages are consistent with each other down to  
467 a depth of 2.60 m corresponding to an age of ~70 ka. The quartz dose in the 71±5 ka  
468 sample (138112 at 2.60 m) is 212±10 Gy, consistent with the ~200 Gy limit suggested  
469 by Buylaert *et al.* (2007, 2008). This agreement is clearer in Fig. 8A which shows that  
470 there is no easily detectable systematic deviation between quartz and feldspar below  
471 ~80 ka. Only the oldest three samples show significant deviation.

472

473 The data suggest that the clearly defined soil at the bottom of this section below  
474 2.8 m is consistent with deposition during the MIS 5 (130-75 ka). The sedimentation  
475 rate during this period increases dramatically from  $0.0063^{+0.0034}_{-0.0017}$  m ka<sup>-1</sup> during the  
476 interglacial to  $0.16^{+0.12}_{-0.05}$  m ka<sup>-1</sup> during the subsequent glacial period. The L<sub>1</sub> loess  
477 from ~2.7 up to 1.2 m accumulated over a time period that is small compared to our  
478 uncertainties. There may be a discontinuity above 1.2 m where the feldspar age  
479 drops from 59±3 to 44±3 ka, and another between 0.8 m (43±2 ka) and 0.6 m (32±2  
480 ka). However, the spatial resolution of the data is insufficient to be confident of these  
481 breaks and we confine ourselves to calculating the average sedimentation rate for the



482 top 1 m of  $0.031 \pm 0.005 \text{ m ka}^{-1}$ . The OSL ages confirm that the Holocene soil is  
483 missing but the weak soil developed during the warming period of the Last Glacial, i.e.  
484 MIS 3, so we cannot tell whether recent deposition rates are comparable with the  
485 previous interglacial.

486

487 It appears that sedimentation rate was low during MIS 5 and accelerated at the  
488 onset of MIS 4. At MIS 4 there was a period of very high dust deposition followed by  
489 slow deposition throughout MIS 3.

490

## 491 **Conclusions**

492

493 We have examined both quartz and feldspar from the NYZG loess deposit in  
494 northeastern China in detail. The quartz is sensitive, fast component dominated and  
495 not surprisingly has excellent luminescence characteristics

496

497 Based on studies of dose recovery and equivalent dose as a function of test dose  
498 size measured using the pIRIR<sub>290</sub> signal, we deduce that the test dose employed  
499 should be related to the equivalent dose under investigation. Typically it would appear  
500 that a test dose of 15 to 80% of the  $D_e$  is most likely to give a satisfactory dose  
501 estimate. Furthermore, we question the interpretation of MET-pIRIR data in terms of  
502 increasing stability with increasing stimulation temperature. Even in a sample of

503 non-finite feldspar age, the signal resulting from a low first-IR stimulation temperature  
504 in a pIRIR<sub>290</sub> protocol only results in a sensitivity corrected luminescence signal ~5%  
505 lower than that resulting from a high first-IR stimulation temperature; contrast this with  
506 the ~21% underestimate at low MET-temperatures. It appears that this underestimate  
507 is at least in part an artefact of variation in dose recovery with stimulation temperature.  
508 Nevertheless, both methods suggest the presence of some instability (~10% signal  
509 loss) at field saturation even at the highest (first IR) stimulation temperature (Fig. 7).

510

511 Finally, we have investigated the relative bleaching characteristics of quartz OSL  
512 and the IR<sub>50</sub> and pIRIR<sub>290</sub> feldspar signals and confirmed that quartz OSL bleaches  
513 considerably more rapidly than feldspar, reaching <10% of its initial value when IR<sub>50</sub>  
514 has only lost <50% and pIRIR<sub>290</sub> <20%. These data support the suggestion that the  
515 ratios of the three signals can be used as indicators of the degree of bleaching of the  
516 more sensitive signals. Our pIRIR<sub>290</sub> data are also consistent with an unbleachable  
517 residual signal (equivalent to  $6.2 \pm 0.7$  Gy) underlying an exponentially bleached  
518 signal.

519

520 At this site, quartz OSL and pIRIR<sub>290</sub> ages are in agreement back to ~70 ka. For  
521 older sediments we deduce that quartz increasingly underestimates the deposition  
522 age. Based on our preferred ages we conclude that sedimentation rates increased  
523 rapidly during the Last Glacial, peaking at MIS 4 (~60 ka) at  $0.16^{+0.12}_{-0.05}$  m ka<sup>-1</sup>. During

524 MIS 3 this sedimentation rate was considerably lower but remained high at  
525  $0.031 \pm 0.005 \text{ m ka}^{-1}$  Unfortunately, MIS 2 and 1 are missing at this site.

526

527 This study has demonstrated the consistency of quartz and feldspar ages (and  
528 probably their reliability) over the last glacial period. Beyond that, quartz is  
529 increasingly unreliable and it appears that feldspar ages are consistently more  
530 accurate.

531

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539

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710

## 711 **Figure captions**

712

713 Fig.1. Map of loess distribution in northeastern China showing location of the study  
714 site (Niuyangzigou, NYZG) and its stratigraphy. L and S represent loess and  
715 palaeosol units, respectively. From the top to the bottom, the loess–soil sequences

716 are named as  $L_1$  and  $S_1$  followed the designation of Liu (1985). Based on the OSL  
717 data the Holocene soil ( $S_0$ ) appears to be missing.

718

719 Fig.2. Luminescence characteristics for coarse-grained (63-90  $\mu\text{m}$ ) quartz. A. and B.  
720 Dose–response curves for aliquots of samples 138101 and 138115 respectively (inset  
721 shows the natural decay curves measured at 125 °C and at 90% blue LED power). C.  
722 Preheat plateau tests of samples 138101 and 138115. Three aliquots were measured  
723 at each temperature and error bars represent 1 standard error. The dash-dot line is  
724 drawn at the average  $D_e$  over the 180-280 °C interval. D. Dose recovery ratio as a  
725 function of preheat temperature for samples 138101 and 138107. Aliquots were first  
726 bleached with blue light at room temperature (2 $\times$ 100 s separated by a 10 ks pause)  
727 and then given a dose of 78.5 Gy and 156 Gy for 138101 and 138107, respectively.  
728 The solid line is drawn at unity and the dashed lines at  $\pm 10\%$ . Three aliquots were  
729 measured per preheat temperature and error bars represent one standard error. The  
730 inset shows a histogram of recycling ratios for all samples (measured as part of  $D_e$   
731 determination).

732

733 Fig.3. A. Natural signals and dose–response curves for coarse-grained quartz and  
734 K-feldspar of sample 138115. Data were fitted with a sum of two saturating  
735 exponential functions of the form  $y = ax(1 - \exp(-bx)) + cx(1 - \exp(-dx))$ . The  
736 sensitivity-corrected luminescence ( $L_x/T_x$  values) was normalized to the fitted

737 saturation values ('a+c'). B.  $L_x/T_x$  plots for the pIRIR<sub>290</sub> signal for a set of samples  
738 down the section. Three aliquots were measured for each sample but only one  
739 representative aliquot is shown. Sample 138101 ( $L_x = 100$  Gy;  $T_x = 30$  Gy); 138102 ( $L_x$   
740 = 999 Gy;  $T_x = 300$  Gy); 138108 ( $L_x = 216$  Gy;  $T_x = 65$  Gy); 138115 ( $L_x = 362$  Gy;  $T_x =$   
741 108 Gy) and the B/M boundary sample 1535 ( $L_x = 999$  Gy;  $T_x = 300$  Gy).

742

743 Fig.4. Result of the dose recovery test for pIRIR<sub>290</sub>. A. Plot of the dose recovery ratio  
744 versus test dose size. The test dose size varies from 5 to 260% of the total  
745 (natural+added) dose. The dose recovery ratio was calculated as the measured dose  
746 divided by the sum of the natural and the given dose. The solid line is drawn at unity  
747 and the dashed-dot lines at  $\pm 5\%$ . B. dose recovery results show as the measured  
748 dose versus added dose.

749

750 Fig.5. Dependence of  $D_e$  on test dose size for the uppermost (138101), middle  
751 (138108) and lowermost (138115) samples from the upper 3.2 m section. The  
752 dash-dot line is the average of the  $D_e$  value for test doses ranging between 15 and 80%  
753 of the total (natural + added) dose.

754

755 Fig.6. Comparison of pIRIR<sub>290</sub> and MET-pIRIR. A. and B. Dependence of  $D_e$  on prior  
756 IR stimulation temperature for the uppermost (138101) and lowermost (138115)  
757 samples from the upper 3.2m section. Three aliquots were measured at each

758 temperature and error bars represent one standard error. The dash-dot line is drawn  
759 at the average  $D_e$  over the 50-260 °C interval for the pIRIR<sub>290</sub>. C. Dependence of  
760 dose recovery ratio on prior IR stimulation temperature for the uppermost sample  
761 (138101). Three aliquots were measured at each temperature and error bars  
762 represent one standard error. The dash-dot line is drawn at unity on the vertical axis.

763

764 Fig.7. Fraction of saturation for both the pIRIR<sub>290</sub> and MET-pIRIR signals for a sample  
765 from below to the B/M boundary (burial dose >2000 Gy). A. Fraction of saturation  
766 measured using the large test dose. B. Fraction of saturation measured using the  
767 small test dose. All the data were fitted with a single saturating exponential function.  
768 Each data point is an average of three aliquots and error bars represent one standard  
769 error. The solid line is drawn at unity and the dashed line at 0.9.

770

771 Fig.8. Comparison of age and bleaching rate for the quartz OSL, feldspar IR<sub>50</sub> and  
772 pIRIR<sub>290</sub> signals. A. Quartz OSL and feldspar IR<sub>50</sub> ages plotted as a function of  
773 pIRIR<sub>290</sub> age. The IR<sub>50</sub> and pIRIR<sub>290</sub> ages have a residual dose of  $0.6 \pm 0.1$  Gy and  
774  $6.2 \pm 0.7$  Gy subtracted from the  $D_e$  and are not corrected for fading. The vertical  
775 dashed line shows the upper quartz OSL dating limit. B. Residual quartz OSL,  
776 feldspar IR<sub>50</sub> and pIRIR<sub>290</sub> doses for different Hönle SOL2 solar simulator bleaching  
777 times for sample 138115. The solid line was derived from fitting the IR<sub>50</sub> to pIRIR<sub>290</sub>  $D_e$   
778 values for all 15 samples. Each data point is an average of three aliquots and error

779 bars represent one standard error.

780

781 Fig.9. The relationship between the residual dose and  $D_e$  obtained with the pIRIR<sub>290</sub>  
782 protocol on three samples (138102,-08,-15). A. and C. Average residual dose  
783 obtained after different Hönle SOL2 solar simulator bleaching times as a function of  
784 the pIRIR<sub>290</sub> and IR<sub>50</sub>  $D_e$ , respectively. Each point is the average residual dose of  
785 three to twelve aliquots per sample obtained after the given exposure times and error  
786 bars represent 1 standard error. The relationship between residual dose and  
787 equivalent dose is described by a linear fit for each exposure time. Inset in A. is the  
788 same relationship for all 15 samples for a single bleaching time of 15 h. B. and D.  
789 Intercept of the linear fits with the y-axis from A. and C. as a function of exposure  
790 time.

791

792 Fig.10. Stratigraphy, quartz OSL and feldspar pIRIR<sub>290</sub> ages for NYZG. The ages are  
793 plotted against the section depth. Open circles represent the quartz OSL ages and  
794 closed circles represent the pIRIR<sub>290</sub> ages. Sedimentation rates were derived from  
795 linear regression.

796

### 797 **Table captions**

798

799 Table 1. Summary of sample code and depth, radionuclide concentrations, calculated

800 dose rates, OSL and pIRIR<sub>290</sub> D<sub>e</sub> values and luminescence ages. The absolute  
801 uncertainty on the water content is  $\pm 5\%$ . The pIRIR<sub>290</sub> D<sub>e</sub> values have a residual dose  
802 of  $6.2 \pm 0.7$  Gy subtracted from the measured value while the pIRIR<sub>290</sub> ages are not  
803 corrected for fading. (n) denotes the number of aliquots contributing to the D<sub>e</sub>.

804

805 Table 2. Outline of dose measurement protocols used in this study. SAR protocol  
806 after Murray & Wintle (2000, 2003), post-IR IRSL protocol after Thiel *et al.* (2011a),  
807 MET-post-IR IRSL after Li & Li (2012a). For the 'natural' sample, the give dose = 0. T  
808 varies from 50 to 260 °C. The whole sequence is repeated for several regenerative  
809 doses including a zero dose and a repeat dose.