



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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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; pIRIR₂₉₀) 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_{50,290} 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_e 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 pIRIR₂₉₀ 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 pIRIR₂₉₀

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 pIRIR₂₉₀ signal is reached after ~300 h,
27 corresponding to a dose of 6.2 ± 0.7 Gy. Quartz OSL and feldspar pIRIR_{50,290} 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⁻¹) 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 (pIRIR₂₉₀) 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_e 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 pIRIR₂₉₀ 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 (S₀), the
119 Last Glacial loess (L₁) and the Last Interglacial palaeosol (S₁) (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 H₂O₂ (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⁻³).

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⁻²) infrared
140 (IR) LEDs (870 nm, ~135 mWcm⁻²); each reader was equipped with a calibrated ⁹⁰Sr
141 /⁹⁰Y 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 ± 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 ± 0.015 Gy ka⁻¹ and 0.06 ± 0.03 Gy
167 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 ± 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 ± 3 Gy for the upper sample to 270 ± 13 Gy for the lowest sample; the
203 corresponding ages are 30 ± 2 and 99 ± 7 ka. We note that the bottom four samples all
204 have D_e values ≥ 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 pIRIR₂₉₀ 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 pIRIR₂₉₀); 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 pIRIR₂₉₀ 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 pIRIR₂₉₀ 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 pIRIR₂₉₀ 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 ± 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 ± 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 pIRIR₂₉₀ 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 pIRIR₂₉₀ 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 pIRIR₂₉₀ D_e 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 ± 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_{50,290} D_e values with
309 pIRIR_{200,290} D_e values and MET-pIRIR₂₅₀ 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_{200,290} 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 pIRIR₂₉₀ 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 pIRIR₂₉₀ 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 pIRIR₂₉₀ 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 pIRIR₂₉₀ signal is a stable signal.
342 Other studies have made the same observation for pIRIR₂₉₀ (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 pIRIR₂₉₀
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 pIRIR₂₉₀ protocol, lower first-IR stimulation temperatures (50-140 °C) yield
360 fraction of saturation values of less than 0.90 (average = 0.87 ± 0.01) and higher
361 temperatures (170-260 °C) yield higher values (average = 0.92 ± 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 ± 0.01 and 0.965 ± 0.004 ,
365 respectively. Both for pIRIR₂₉₀ (measured with prior IR stimulation ≥ 170 °C) and the
366 MET-pIRIR protocols, the data are not consistent with unity (~4% below saturation
367 light level for MET and ~8% for pIRIR₂₉₀) which might suggest instability in the
368 pIRIR₂₉₀ signal and the high-temperature MET-pIRIR signals. For the pIRIR₂₉₀ signals
369 measured with low prior-IR stimulation temperatures this underestimation increases
370 to ~13%.

371

372 Fig. 7B shows the results when a small test dose of 50-60 Gy is used. For both
373 pIRIR₂₉₀ (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_{200,290} this value is 0.99 ± 0.02 , consistent with the MET-pIRIR signals
376 measured at 250 and 300 °C (0.96 ± 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_{50,290} and pIRIR_{200,290} respectively. It appears
393 that the pIRIR_{200,290} fading rate is lower than the pIRIR_{50,290} fading rate; this is
394 consistent with the saturation measurements on sample 1535 presented earlier. Li &
395 Li (2012b) have also argued that pIRIR_{200,290} is more stable than pIRIR_{50,290}.

396

397 ***Bleaching characteristics***

398 **Comparison of quartz OSL, feldspar IR₅₀ and pIRIR₂₉₀ bleaching rates.** Fig. 8A
399 summarises the quartz OSL, IR₅₀ and pIRIR₂₉₀ ages for all 15 samples. All the IR₅₀
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 IR₅₀ signal is significantly unstable
403 compared to pIRIR₂₉₀ (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 pIRIR₂₉₀ 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 pIRIR₂₉₀ dose, ~20 s for the IR₅₀ 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 (IR₅₀) and open stars (quartz OSL) in Fig. 8B, plotted against the residual pIRIR₂₉₀
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 IR₅₀ dose is ~55% of its initial

419 value and the pIRIR₂₉₀ 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 IR₅₀ and pIRIR₂₉₀ 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 pIRIR₂₉₀ signals (and thus the
429 IR₅₀ 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 pIRIR₂₉₀ 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 pIRIR₂₉₀ 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 pIRIR₂₉₀ 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 pIRIR₂₉₀
447 protocol. The results are summarized in Fig. 9A (pIRIR₂₉₀) and Fig. 9C (IR₅₀). 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 pIRIR₂₉₀ and IR₅₀ 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 pIRIR₂₉₀ residual dose of 6.2 ± 0.7
459 Gy has been subtracted from all measured D_e values used to calculate the pIRIR₂₉₀
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 pIRIR₂₉₀ 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⁻¹ during the
476 interglacial to $0.16^{+0.12}_{-0.05}$ m ka⁻¹ during the subsequent glacial period. The L₁ 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 pIRIR₂₉₀ 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 pIRIR₂₉₀ 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 IR₅₀ and pIRIR₂₉₀ feldspar signals and confirmed that quartz OSL bleaches
513 considerably more rapidly than feldspar, reaching <10% of its initial value when IR₅₀
514 has only lost <50% and pIRIR₂₉₀ <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 pIRIR₂₉₀ data are also consistent with an unbleachable
517 residual signal (equivalent to 6.2 ± 0.7 Gy) underlying an exponentially bleached
518 signal.

519

520 At this site, quartz OSL and pIRIR₂₉₀ 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⁻¹. 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 μm) 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 (2×100 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 pIRIR₂₉₀ 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 pIRIR₂₉₀. 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 pIRIR₂₉₀ 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 pIRIR₂₉₀. 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 pIRIR₂₉₀ 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 IR₅₀ and
772 pIRIR₂₉₀ signals. A. Quartz OSL and feldspar IR₅₀ ages plotted as a function of
773 pIRIR₂₉₀ age. The IR₅₀ and pIRIR₂₉₀ ages have a residual dose of 0.6 ± 0.1 Gy and
774 6.2 ± 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 IR₅₀ and pIRIR₂₉₀ doses for different Hönle SOL2 solar simulator bleaching
777 times for sample 138115. The solid line was derived from fitting the IR₅₀ to pIRIR₂₉₀ 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 pIRIR₂₉₀
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 pIRIR₂₉₀ and IR₅₀ 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 pIRIR₂₉₀ 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 pIRIR₂₉₀ 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 pIRIR₂₉₀ D_e values and luminescence ages. The absolute
801 uncertainty on the water content is $\pm 5\%$. The pIRIR₂₉₀ D_e values have a residual dose
802 of 6.2 ± 0.7 Gy subtracted from the measured value while the pIRIR₂₉₀ ages are not
803 corrected for fading. (n) denotes the number of aliquots contributing to the D_e.

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.