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1	A detailed quartz and feldspar luminescence chronology for the Khonako II loess		
2	section (South Tajikistan)		
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22			
23	Abstract		
24	Parts of Central Asia have been dominated by high dust flux since the start of the Quaternary.		
25	The resulting loess-palaeosol deposits are important archives for landscape and climate		
26	changes, but there is a lack of chronological data for the subaerial deposits of Southern		
27	Tajikistan. Our study presents a high-resolution luminescence chronology for the Khonako II		
28	section in the Khovaling region. Thirty-seven samples were collected from the first ~ 8 m of		
29	the section, corresponding to the Holocene soil and the upper part of the Loess 1 (L1) unit.		

- We applied quartz OSL dating to the coarse silt fraction (40-63 $\mu m)$ and polymineral 30
- $pIRIR_{200,290}$ dating to the very fine sand fraction (63-90 μm). The polymineral $pIRIR_{200,290}$ 31

32 signal has satisfactory characteristics. The quartz dose response curve displays a non-single 33 exponential growth above ~ 100 Gy, thus, the D_e values were estimated by fitting a single 34 exponential function plus linear component to the dose response data. To assess the pIRIR_{200,290} residual dose at deposition, we used the offset between our quartz and 35 36 polymineral chronologies from Khonako II and equivalent doses measured on modern dust 37 samples from Tajikistan and Uzbekistan. The resulting 9.2±0.4 Gy dose was then subtracted 38 from the pIRIR_{200,290} doses for final age calculation. The quartz OSL and pIRIR ages are then 39 generally consistent. The obtained high-resolution luminescence chronology shows that the 40 upper part of the L1 unit accumulated during the end of marine isotope stage (MIS) 3 and through MIS 2 (39 to 20 ka). The average dust accumulation rates (DARs) increased from 41 \sim 31 cm ka⁻¹ to 41 cm ka⁻¹ between MIS 3 and MIS 2. The DAR reached a peak of \sim 71 cm ka⁻¹ 42 at 21 ka. A hiatus of ~7-9 ka was then identified between the end of the last glacial maximum 43 44 (LGM) and the beginning of the Holocene, and the average DAR decreased during the Holocene. Our results indicate a continuous high dust flux in the Khovaling plateau over the 45 46 last 39 ka, but reworking processes and potential deflation events mark the LGM-Holocene 47 transition. High-resolution luminescence dating studies in Tajikistan are therefore important 48 to detect potential discontinuities in the sequences and provide an independent timescale for 49 interpreting the climate proxies and calculating dust accumulation rates.

Keywords: Southern Tajikistan, quartz OSL dating, polymineral pIRIR_{200,290} dating, high resolution dating, dust accumulation rates

52

53 1. Introduction

54 Loess deposits in Central Asia are well-known for providing exceptional palaeoclimate and 55 palaeoenvironmental archives. In the last ~2.7 Ma, dust from deflation processes under arid 56 and semi-arid conditions has been transported by westerly winds and is believed to be 57 deposited almost continuously (Zan et al., 2022). Most of the thickest loess sections are 58 located on the mountain footslopes of the Pamirs in the Afghan-Tajik depression (Southern 59 Tajikistan), with up to ~200 m of accumulated sediment. These loess deposits consist of 60 alternating thick loess units (L), resulting from high dust accumulation rates during glacial periods, and palaeosols forming pedocomplexes (PCs) developed during interglacial periods 61 62 under warmer and wetter climatic conditions. This alternating formation of loess and palaeosol units records climate stages and landscape evolution during the Quaternary (Ding et 63 64 al., 2002; Li et al., 2020).

65 Loess-palaeosol sequences in the Afghan-Tajik depression are characterised by a considerable 66 thickness, reaching 200 m in several outcrops, a large number of well-defined palaeosols, 67 forming up to 40 PC's in the most complete sections, and a semi-continuous chronology, 68 covering the entire Quaternary. This makes the loess-palaeosol sequences of Southern 69 Tajikistan an important archive of Central Asian climate and landscape history. The most 70 complete loess-palaeosol sections have been described in the Khovaling loess plateau in the 71 piedmonts of the Pamirs (Dodonov, 2002; Ding et al, 2002; Dodonov and Zhou, 2008). The 72 current chronology of the Tajik loess sections is based mainly on magnetostratigraphy and 73 frequency-dependent magnetic susceptibility (Dodonov, 1991; Dodonov and Zhou, 2008; 74 Parviz et al., 2020; Zan et al., 2022), and numerous studies have suggested that the Central 75 Asian loess chronostratigraphy can be correlated with the Chinese Loess Plateau (CLP) and 76 δ^{18} O records (Dodonov and Baiguzina, 1995; Ding et al., 2002; Dodonov et al., 2006; 77 Dodonov and Zhou, 2008; Parviz et al., 2020; Tian et al., 2021). However, this assumption relies on the continuity and integrity of the sedimentary record, an assumption not yet tested 78 79 for the subaerial deposits of Southern Tajikistan, which could be affected by erosion and 80 slope post-depositional processes.

81 Determining high-resolution numerical chronologies for the Southern Tajik loess sequences is 82 important to ensure an independent timescale for interpreting climate proxies, such as grain 83 size and magnetic susceptibility, and accurate correlation of the palaeosols with the global 84 climate. High-resolution luminescence dating studies can also reveal discontinuities in the 85 sedimentary record. Frechen and Dodonov (1998) were the first to apply luminescence dating 86 to Tajik loess, and they coupled high-resolution (n=74) thermoluminescence (TL) and 87 infrared stimulated luminescence (IRSL) with an IR stimulation at 50°C (IR₅₀) dating in the 88 Darai Kalon section down to the Bruhnes/Matuyama boundary between PC9 and PC10. They 89 obtained a good agreement between their TL and fading uncorrected IRSL ages up to PC1 90 (~80 ka), confirming the correlation suggested from the palaeomagnetic record between PC1 91 and the last interglacial soil layer (S1) of the CLP record. Below PC1, the IRSL dose 92 underestimation and the scatter between the ages prevented the establishment of a reliable 93 chronology (Frechen and Dodonov, 1998).

94 The IR₅₀ signal is known to suffer from anomalous fading (e.g. Spooner, 1994) which usually

95 results in an underestimation of the true burial age. Since the recent methodological

96 developments in luminescence dating in terms of measurement procedure (single-aliquot

97 regenerative-dose [SAR] protocol, Murray and Wintle, 2000) and the discovery of a more

98 stable feldspar signal (post-IR IRSL [pIRIR], Thomsen et al, 2008; Thiel et al., 2011; Li and

99 Li, 2011;), most studies in loess are now based on quartz SAR optically stimulated

100 luminescence (OSL) (e.g., Buylaert et al., 2007, 2008; Lai et al., 2010) and/or K-rich feldspar

101 SAR post-IR IRSL ages (e.g., Buylaert et al., 2012, 2015; Li and Li, 2012a; Li et al., 2015, Yi

- et al., 2016, 2018; Stevens et al., 2018; Zhang et al., 2023). Because of the high dose rates in
- 103 loess (~ 3 Gy ka⁻¹), the OSL signal from quartz can only be used to date the last ~50 ka,
- 104 whereas feldspar has the potential to extend the chronological range to ~250 ka (Buylaert et

105 al., 2007; Li and Li, 2011).

In southern Central Asia, quartz OSL dating has been applied to obtain a numerical 106 107 chronology from the Holocene to the last glacial period on several loess sections in Southern 108 Tajikistan and the Tajik Basin (Wang et al., 2018; Yang et al., 2020; Song et al., 2021; Tian et 109 al., 2021). Luminescence sensitivity studies have also been carried out on the Karamaidan 110 loess section from MIS 9 to MIS 19 (Dave et al., 2022) and the Khonako III section from L1 111 to PC2 (Li and Zhou, 2021). These authors highlighted an increase in sensitivity of the quartz signal in the pedocomplexes units that might be linked to either a change in source or to 112 113 repeated irradiation and bleaching cycles during transportation processes. However, no 114 chronology was presented for the Khonako III section. Thus, luminescence chronologies are 115 limited for the Southern Tajik loess record, and the application of pIRIR protocols has not yet 116 been tested in this area. Li et al. (2020) successfully applied a pIRIR_{200,290} protocol to loess 117 sections in the northern margins of the Tianshan Mountains, China. These authors considered 118 their feldspar ages reliable up to ~ 250 ka, but that the quartz OSL ages start to underestimate 119 the feldspar ages beyond ~40 ka. These results highlight the potential of the method for

120 Central Asian loess-palaeosol sequences.

121 This study presents a detailed luminescence chronology for the Holocene soil and the upper 122 part of the L1 unit of the Khonako II section in Southern Tajikistan. We test the applicability 123 of quartz OSL and polymineral pIRIR_{200,290} measurement protocols and compare the offset 124 between the resulting ages with those estimated on modern dust samples to assess the residual 125 dose at deposition of the pIRIR_{200,290} signal.

126 2. Site description and sampling

127 The Khonako site (N 38.35959° E 70.04552°) is located in the Tajik-Afghan depression

128 (Southern Tajikistan), about 10 km northeast of Khovaling (Fig. 1). As a result of a series of

129 landslides, three cirques have formed at this location: the western (geological section

130 Khonako-II), which is 1 km long and opens up to 16-18 PC's, central (palaeolithic site

131 Khonako-III), ~350 m long, and eastern (geological section Khonako-I), about 300 m long, all

- separated by small promontories. The Khonako III outcrop is well-known for its
- archaeological finds with Middle Palaeolithic industries in PC1 and PC2 and artefacts from
- the Lower Palaeolithic in PC3 and PC4 (Schäfer et al., 1998). The Khonako II section
- 135 (altitude 1890 m) is considered to be one of the best-preserved loess sections in the Khovaling

136 Plateau; up to 16 PCs have been identified, providing a sedimentary record presumably back 137 to ~MIS 31 (Dodonov, 2002). The Holocene cover in loess areas of Tajikistan is heavily 138 eroded due to modern agriculture (Murray et al, to be submitted) and is present in only a few locations in the Khovaling plateau. The Holocene soil at Khonako II is 2 m thick and is the 139 140 thickest preserved Holocene section among the loess records of the Khovaling region. The L1 141 unit is developed over 12.2 m. We excavated the top part of the Khonako-II outcrop in 2019; 142 two geological trenches were dug in the middle part of the outcrop and opened up 40.8 m of 143 in-situ sediments to the top of L3. The trenches were divided into forty-three sections of 144 between 0.5 and 1.5 m length. A field description of the loess and palaeosol layers was made 145 and samples for magnetic susceptibility measurements and sedimentological analyses 146 collected. Metal or plastic sampling tubes for luminescence dating were hammered into the 147 surface cleaned sections every ~ 10 to 30 cm.

In addition, modern dust samples were collected in 2022 and 2023 after mild dust storms,
which occur regularly in Tajikistan in summer and autumn. Dust was scraped off various
surfaces in areas remote from construction work. Our samples include only dust of natural
origin and long-distance transport material. The first sample was collected near Termez city
(Uzbekistan), 150 km southwest of Khovaling; the second and third samples were collected
near Dushanbe and Gissar cities (100 km northwest of Khovaling).

In this study, thirty-seven samples are used to develop a luminescence chronology for the top
8 metres, corresponding to the Holocene soil and upper part of the L1 unit (Figs. 1c,2, Table
S1).

157

Fig. 1.

- 158 3. Materials and methods
- 159 3.1. Sample preparation

160 The samples were prepared under dim orange light (Sohbati et al., 2021) using standard 161 procedures (e.g. Murray et al., 2021). The outer five cm of each tube end were removed and used for high-resolution gamma spectrometry measurements. Because of the relatively high 162 carbonate content of the sediment, a first 10 % HCl treatment was applied before wet sieving, 163 164 and the 63-90 and 40-63 µm fractions were kept for further analyses. To ensure the removal 165 of carbonates, a second 10 % HCl treatment was then applied, followed by a 10% H_2O_2 166 treatment to remove organic matter. Because of the fine-grained nature of the sediment and 167 thus limited fractions, heavy liquid separation was not carried out and pIRIR_{200,290} 168 measurements were performed on the polymineral 63-90 µm fraction. The coarse-silt quartz

169 fraction (40-63 μ m) was treated with 35% H₂SiF₆ for three to four days to remove the 170 feldspar grains.

171

3.2. Luminescence measurements

All measurements were made using Risø TL/OSL readers equipped with calibrated ⁹⁰Sr/⁹⁰Y
beta sources (Bøtter-Jensen et al., 2010; Hansen et al., 2018; Autzen et al., 2022). Polymineral
samples were optically stimulated using 870 nm IR LEDs (between 135 and 350 mW cm⁻²),
and the IRSL signal was detected using a blue filter combination (4 mm of Corning 7–59 in
combination with 2 mm Schott BG 39). Quartz stimulation was achieved using 470 nm blue
LEDs (between 40 and 100 mW cm⁻²), and the OSL signal was detected through a 7.5 mm
Hoya U-340 filter.

We used a pIRIR_{200,290} SAR protocol (Li and Li, 2012b; Buylaert et al., 2012, 2015; Yi et al.,
2015; Stevens et al., 2018) with a preheat of 320 °C for 60 s to measure the polymineral
samples (Ø=2 mm for aliquots, hundreds of grains per aliquot). The first infrared stimulation

182 was carried out at 200 °C for 200 s, followed by a second IR stimulation at 290 °C of the

same duration. The size of the test dose was equivalent to $\sim 100\%$ of the expected dose. At the

184 end of each SAR cycle, the polymineral aliquots were bleached using an elevated-temperature

185 IR stimulation (325 °C for 200 s). The first 2 s of the pIRIR₂₉₀ signal was used for dose

estimation. The last 40 s was used to estimate background.

187 The coarse-silt quartz samples were measured as multi-grain aliquots (\emptyset =2 or 8 mm for 188 aliquots, hundreds to thousands of grains per aliquot) using a standard SAR quartz protocol 189 (Murray and Wintle, 2000, 2003) with a preheat of 240 °C (10 s), a cutheat of 200 °C, and a 190 high-temperature bleach at 280 °C for 100 s between SAR cycles to minimise potential 191 recuperation (Murray and Wintle, 2003). The quartz OSL signals were measured using the 192 blue LEDs at 125 °C for 40 s. In the analysis, we summed the initial 0.48 s of stimulation and 193 estimated the background from the subsequent 0.64 s (early background subtraction; Ballarini 194 et al., 2007; Cunningham and Wallinga, 2010).

195 The quartz purity was assessed using an OSL IR depletion ratio test (Duller, 2003). Seven

samples (228850, 228856, 228860, 228861, 228862, 228864, 228866) displayed

unsatisfactory average OSL IR depletion ratios (average of 0.82±0.04) and were subsequently

198 discarded from the OSL dataset. For the accepted samples, a comparison between the average

sensitivity corrected OSL IR depletion ratio (0.970±0.010) and the blue-stimulated recycling

- ratio $(0.971\pm0.007, n = 156, 30 \text{ samples})$ confirms the absence of a significant IRSL signal
- and the ability of the protocol to adequately correct for sensitivity changes.

202 3.3. Dose rates

203 Given the fine-grained nature of the sediment and the risk of losing the feldspar grains during 204 the etching process, we decided not to etch our very fine sand fraction (63-90 μ m) and instead 205 take the alpha component into account. We use an a-value of 0.09±0.02 (Schmidt et al., 2018) 206 and an alpha attenuation factor of 0.30±0.05 (Martin et al., 2014). External beta dose rate 207 attenuation factor was calculated following Mejdahl (1979). The internal beta dose rate 208 contribution is based on an assumed 12.5±0.5 % K content (Huntley and Baril, 1997). For the 209 quartz (40-63 µm) alpha component, we use an a-value of 0.035±0.003 (Lai et al., 2008) and 210 an alpha attenuation factor of 0.40±0.05, consistent with Martin et al. (2014).

211 For radionuclide concentration measurements, the sediment from the outer part of the tubes

212 was dried at 110 °C for 24 hours, ground, mixed with wax, and cast into cup- or disc-shaped

213 moulds. After a minimum of three weeks of storage, radionuclide concentrations were

214 measured using high-resolution gamma spectrometry (Murray et al., 1987, 2018) and

converted into infinite matrix dry dose rates using the conversion factors of Cresswell et al.

216 (2018). The cosmic ray contribution was calculated based on Prescott and Hutton (1994).

217 To obtain reliable in situ water contents for the Holocene part of the section we dug two pits

218 (2 m by 1 m) on top of the plateau, ~35 m from the edge of the cliff where our KH-II section

219 was dug. One pit was dug after the summer (dry) season in October 2002 and the second one

after the winter (wet) season in May 2023. In each pit, seven samples were collected during

digging the pit until a depth ~2 m. Samples were double-packed in plastic zip bags water

contents (weight water/dry weight x 100, %) measured upon arrival in the laboratory in

223 Denmark (Table S3). For samples 228848 to H28815, water content measurements were done

- on the inside material that was most wet when opening the tubes.
- **225 4.** Results

4.1. Stratigraphy of the upper part of the Khonako II section

Unit 1 (0.0 to 2.0 m) can be divided into two layers: the Holocene soil (layer 1) and a weakly
reworked loess (layer 2). Layer 1 (0.0–0.8 m) is subdivided into four horizons: A (0-0.3 m):

Light loam dark, gray, dry, highly porous, organic rich; B (0.3-0.5 m): Medium loam, dark

230 gray, dense, porous, blocky-prismatic structure; Bm (0.5-0.6 m): Medium loam, brown,

porous, prismatic structure, krotovinas with light sediments from lower layers; BCcaz (0.6-0.8

m): Medium loam, light beige, dense, highly porous, blocky, considerable CaCO₃, krotovinas

233 with dark material from upper soil. Layer 2 (0.8-2.0 m) is loam, light beige, dry, dense,

234 coarse-medium-highly porous, blocky, with considerable CaCO₃ and containing krotovinas

with material from the upper soil. Unit 2 (2.0 to 12.25 m) corresponds to most of loess unit L1

and is divided into two layers (layers 3 and 4). Layer 3 (2.0-10.2 m) is loam, light beige,

porous, massive, homogeneous, with rare carbonate veins. It is typical loess. Layer 4 (10.20-

12.25 m is loam yellow and brown with rare bioliths, CaCO₃ in small pores, and carbonate

nodules up to 5 cm in diameter. It is identified as an interstadial palaeosol.

240

Fig. 2.

241 4.2. Dose rates

The total dose rates are presented in Table S1, and the radioelement concentrations, dry alpha, 242 243 dry beta, and dry gamma dose rates in Table S2. The dose rate values are typical for loess, with an average of 3.50 ± 0.07 Gy ka⁻¹ (n=37) for quartz. A clear trend can be seen with the 244 245 highest dose rate values for the four samples in the upper part of the Holocene soil and a 246 decrease towards the L1 unit. Quartz dose rates range from 3.0 ± 0.2 (sample 228849) to 4.7 ± 0.2 (sample 228842) Gy ka⁻¹ in the Holocene soil, and feldspar dose rates range from 247 3.5 ± 0.2 to 5.3 ± 0.3 Gy ka⁻¹. The L1 dose rates are homogenous with average values of 248 3.38 ± 0.02 Gy ka⁻¹ (n = 30) for quartz and 3.96 ± 0.03 Gy ka⁻¹ for feldspar. The ²¹⁰Pb to ²²⁶Ra 249 ratio of 0.98±0.03 (n=20) indicates no radon loss over the last few decades (De Corte et al., 250 2006). Individual ²³⁸U analyses are insufficiently precise to allow us to discuss the degree of 251 possible disequilibrium in individual samples. However, the unweighted average ²²⁶Ra to ²³⁸U 252 ratio is 1.30 ± 0.05 (n=30), omitting ratios with an uncertainty of >50%. We conclude that, on 253 254 average, there may be evidence for a weak disequilibrium in the first part of the ²³⁸U decay 255 series.

The average water content of the samples collected from the pit after the dry season is $9\pm1\%$ and increases to $17\pm2\%$ after the wet season. To best account for the seasonal variability, an average value of $13\pm5\%$ was chosen for the wet dose rate calculations of the upper Holocene

259 part of the section (samples 228842 to 228847, see also discussion below).

For samples 228848 to H28815 (L1 unit), the average measured water content is 20±2%, but
sample 228860 gave a maximum value of 29%. We consider this value to be the most
representative of the present water content, as we suspect a dry out from the trenches before
sampling and from inside the tubes.

The long-term burial water content for the L1 samples was then calculating followingequation:

266 $\frac{duration_{Hol}*w.c._{1} + (age_{sample} - duration_{Hol})*w.c._{2}}{age_{sample}}$

267 Where $w. c_{.1}$ corresponds to the measured water content in the Holocene soil, and $w. c_{.2}$ to 268 the maximum observed water content value from layer 3. The Holocene duration 269 $(duration_{Hol})$ is assumed to be 11 ka. Since a hiatus was identified between 1.3 and 1.4 270 metres depth (see discussion), we expanded the $duration_{Hol}$ to 13 ka for samples between 1.41 and 2.45 m depth to reflect the successive burial, exposure, and re-burial events of the 271 272 sediment. Our model follows an iterative process, where the initial water content used in the 273 calculation of the quartz OSL age (age_{sample}) is updated until convergence with the 274 modelled water content. The resulting modelled water content values are displayed in Table 275 S4, and an uncertainty of 5% was assumed.

276

4.3. Quartz OSL characteristics

277 The inset in Fig. 3a shows a typical quartz OSL stimulation curve (sample 288881). It is very 278 similar in shape to that from a known fast-component dominated sample, also shown (Risø 279 calibration quartz, Hansen et al., 2015). Fig. 3a shows a typical quartz dose response curve 280 (DRC) from sample 228881. Also shown is a single exponential function (EXP, dashed line), 281 which clearly does not fit the data well. However, an exponential function with a linear 282 component (EXPLIN), where the linear component becomes dominant at ~ 100 Gy, fits the 283 data adequately. The natural signal is then interpolated on the dose response curve up to ~ 120 284 Gy, and the EXPLIN fit was chosen for all the samples. For doses below 60 Gy, a single saturating exponential function fit of the dose response curve was also satisfactory. Fig. 3b 285 286 shows that the estimated dose is independent of the chosen preheat temperature ranging 287 between 200 and 300 °C. The cutheat temperature is 40 °C lower than the preheat temperature. Therefore, a preheat of 240 °C and a cut-heat of 200 °C were chosen for further 288 289 measurements.

A series of dose recovery tests (Murray, 1996) was performed for samples 228842, 228844,

228855, and 228870, using 24 aliquots per sample except for sample 228842 (n=36), with

given doses close to the estimated natural doses. The samples were bleached twice using the

blue LEDs at room temperature for 100 s (with an intervening pause of 10 ks) to remove the

natural signal prior to giving a beta dose ranging between 12 and 76 Gy, followed by

295 measurement. The resulting ratios are shown in Fig. S1. The average dose recovery ratio is

1.06±0.01 (n=99), and we conclude that our SAR protocol is well-suited to determine a dose

given to the sample before any heat treatment.

The arithmetic average D_e (Guérin et al., 2017) estimates are presented in Table S1 with the
 corresponding quartz ages.

300

Fig. 3.

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4.4. Polymineral pIRIR_{200,290} characteristics

302	The pIRIR ₂₉₀ dose response curve is well-represented by a single saturating exponential
303	function (Fig. 4a). The estimated D_e seems to be independent of the first IR stimulation
304	temperature for first IR temperature above 50°C (Fig. 4b). The first IR stimulation
305	temperature was chosen to be 200°C to ensure the stability of the signal even at high doses (Li
306	and Li, 2012b; Stevens et al., 2018). A sample from the Holocene soil and one from the L1
307	unit (228842 and 228871) were bleached in a solar simulator (Hönle SOL2 at a distance of 80
308	cm) for eight days for a dose recovery test. First, residuals of 9±2 Gy (n=5, 228842) and 23±1
309	Gy (n=5, 228871) were measured. Secondly, 36 solar-bleached aliquots were given beta doses
310	of 27 Gy (228842) or 115 Gy (228871). These doses were then measured using test doses
311	(TDs) of 30%, 50%, and 100% of the given dose (six aliquots per treatment). For a given dose
312	of 27 Gy, the average dose recovery ratios (after subtraction of the measured residual dose)
313	are 1.07 ± 0.10 (TD 30%), 1.20 ± 0.13 (TD 50%), and 1.23 ± 0.10 (TD 100%), and the
314	corresponding ratios for a given dose of 115 Gy are 1.17 ± 0.05 , 1.18 ± 0.04 and 1.01 ± 0.02 (Fig
315	4c.), respectively. The dose recovery ratios in the low dose experiment are all consistent with
316	each other, but it should be noted that the ratios are poorly known because of the large
317	uncertainty associated with the residual measurements. In the high dose experiment, the dose
318	recovery ratio is only acceptable when the test dose is equal to the given dose.

319 A dose recovery test was undertaken using a single-aliquot regeneration and added dose (SARA) protocol (Mejdahl and Bøtter-Jensen, 1994) on sample 228842 (youngest sample 320 321 with a polymineral dose of 29.3 ± 3.4 Gy) to investigate further the influence of the test dose 322 size. Doses in the range of interest (40, 80, 160 Gy) were added to the natural dose. One set of 323 12 aliquots was measured with a TD of \sim 30% of the expected dose, and a second set of 12 324 aliquots was measured with a TD of 80-100% of the expected dose. In Fig. 4d, the measured 325 doses are fitted with a linear function, from which the slope estimates the dose recovery ratio. Both a TD of ~30% and a TD of 80-100% give satisfactory results (slopes of 1.057±0.006 and 326 327 0.932±0.013, respectively).

328 In the SOL2 experiment, the dose recovery ratio is only acceptable when the test dose is equal

to the given dose for a dose >100 Gy, but our SARA dose recovery experiment does not

indicate a clear dependence of the dose recovery on the test dose size. Therefore, we conclude

that our pIRIR_{200,290} protocol seems to adequately correct for sensitivity change in a 30-100%

332 TD range. All the Khonako II D_e measurements were made using a TD of ~100% of the

estimated dose for consistency.

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Fig. 4.

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4.5. Quartz OSL and pIRIR_{200,290} age comparison

336 It is known that pIRIR signals bleach significantly more slowly compared to quartz and 337 usually include a hard-to-bleach component, which can lead to a significant dose overestimation for young samples (e.g., Buylaert et al., 2011, 2012; Zhang et al., 2023). One 338 339 way to assess how well the pIRIR signal was reset at deposition is to compare the quartz OSL 340 ages and the pIRIR ages, assuming that in loess the quartz was well-bleached at burial (e.g., Murray et al., 2012; Stevens et al., 2018; Kurbanov et al., 2022; Taratunina et al., 2022; 341 342 Volvakh et al., 2022). In this study, we examine the size of this residual by comparing the 343 quartz OSL and pIRIR_{200,290} ages and by measuring quartz OSL and pIRIR_{200,290} polymineral 344 D_e values on three modern dust samples (229101,03,04). The quartz OSL D_e values for these 345 samples show they are modern ($D_e < 0.5$ Gy, Table S5). We used the average quartz and 346 feldspar dose rates from our Khonako II data set to derive apparent ages for the modern dust, and plotted these pIRIR_{200,290} and quartz ages with our Khonako II dataset (Fig. 5). Three 347 samples from Khonako II (228846,-47,-48) were not taken into account in the comparison; 348 349 they display an unusual high age discrepancy of \sim 7-5 ka and are located in layer 2, where evidence of bioturbation and reworking processes have been detected (see discussion). 350 The data are well-represented by a linear fit (of slope unity). The intercept is 2.25 ± 0.09 ka on 351 352 the pIRIR_{200,290} age axis. From this intercept and the average feldspar dose rate in Khonako II, 353 we deduce that the residual offset is 9.2±0.4 Gy. This value has been subtracted from all our 354 pIRIR_{200,290} doses. It is interesting to note that our residual pIRIR_{200,290} offset is very similar to 355 other measured offsets in loess, i.e., 5 ± 2 Gy (Stevens et al., 2018) and 8 ± 3 Gy (Taratunina et 356 al., 2022) as well as the pIRIR_{50,290} 10 ± 2 Gy measured on polymineral fine-grains from 357 modern/young Chinese dust samples (Buylaert et al., 2011). Table S1 summarises the quartz 358 OSL and pIRIR_{200,290} ages (with and without residual dose subtraction). 80% of our 359 pIRIR_{200,290} ages are statistically consistent (at 95% confidence, random uncertainties only)

- with the quartz OSL ages (Table S1, Figure 2). Omitting the three samples mentioned above
- 361 (228846,-47,-48), 87% are consistent with each other (96% if total uncertainties are used).
- 362

Fig. 5.

363 4.6. Age-depth modelling

A Bayesian model was run using the Bacon software (Blaauw and Christen, 2011) on the 30
quartz OSL and pIRIR_{200,290} ages, using only the random component of the age uncertainty.
The following settings were used to run the model: a prior accumulation rate of 50 cm ka⁻¹

(acc.mean=(50)) and thick = 10. Ages estimates were produced every 5 cm. Fig. 2 compares
magnetic susceptibility (Fig. 2b), the luminescence ages with the Bayesian model (at 95%
confidence) (Fig. 2c), and the dust accumulation rates calculated from the median Bayesian
age estimates (Fig. 2d).

371 5. Discussion

372 Chapot et al. (2012) and Timar-Gabor and Wintle (2013) showed that the quartz laboratory 373 dose response curve for Chinese and Romanian loess is best described using a sum of two saturating exponential functions while the natural signal follows a single saturating 374 375 exponential function, which saturates significantly faster than the laboratory curve. The 376 divergence between the two curves occurs at 100-150 Gy. The laboratory dose response 377 curves for quartz OSL from Tajik loess also display non-single exponential growth above 378 ~100 Gy. The largest measured quartz dose in this study is ~120 Gy and because of the good 379 agreement between quartz OSL ages and pIRIR_{200,290} feldspar ages, we deem our overall 380 quartz chronology reliable over this age range. However, there is a need for more studies with 381 independent age control and reliable natural dose response curves to fully examine the 382 reliability of quartz OSL in the high dose range, where laboratory dose response curves are 383 not well-represented by a single saturating exponential function.

According to the Bayesian chronological model of the Khonako II section, the upper part of 384 385 L1 (2.0-7.9 m in unit 2) was deposited between ~39 and ~21 ka, during the end of MIS 3 and 386 first part of MIS 2. However, the Bayesian approach shows methodological limits for the age 387 estimate of layer 2 (0.8-2 m). Based on the individual luminescence ages, we identified a 388 hiatus between 1.41 and 1.31 m in layer 2. Its duration varies depending on whether the 389 quartz OSL chronology or the polymineral pIRIR_{200,290} chronology is examined. When 390 considering the individual pIRIR_{200,290} ages, the hiatus would be between ~19 ka and 10 ka, 391 and between ~15 and 7 ka when looking at the individual quartz OSL ages. The Bayesian 392 model gives a shorter estimate of the hiatus duration (3 ka, compared to 9 and 7 ka). This can 393 be explained by the mathematical construction of the model regarding the changes in 394 accumulation rates.

The Bacon model (Blaauw and Christen, 2011) is an iterative model that includes a memory factor, w: the accumulation rate at a given depth is a weighted average of the accumulation rate of the previous depth and an independent noise, with prior density functions for both the memory factor and the independent noise. In their paper, Blaauw and Christen (2011) point to the resulting issue regarding hiatuses and any other sharp variation in the accumulation rate; their model is not adapted to such discontinuities in the accumulation rate. Therefore, they

401 recommend that any profile is cut into segments and the model run on each part separately;

402 this segmentation should be based on external information (e.g., sedimentological

403 characteristics). However, since it is the purpose of our geochronological study to identify

404 such discontinuities, we are unable to define the different accumulation regimes before age

405 measurements. As a consequence, we expect that Bacon will not describe discontinuities

406 satisfactorily simply because it is, by its nature, not adapted to such features.

407 The Bayesian model is, therefore, not suitable to describe the sharp changes in sedimentation

408 rates occurring in layer 2. Nonetheless, we consider the Bayesian chronology reliable for the

samples of the upper part of unit 1, which show no inversion or significant discrepancy

between the quartz OSL and the $pIRIR_{200,290}$ ages (0-1 m); the sedimentation then appears to

411 have been continuous during ~ 8 and ~ 3 ka.

412 The hiatus was not identified in the field, but the rapid increase of the magnetic susceptibility 413 at this depth confirms that it occurred at the transition period between the Holocene and the LGM (Constantin et al, 2019). Therefore, the LGM-Holocene boundary does not correspond 414 415 to the bottom of Unit 1 (2 m). We propose the following model to explain the hiatus 416 formation and the discrepancy between the quartz and polymineral ages: (1) At the end of the LGM, dust continued to accumulate until the Late Glacial time (~12 ka). Then, (2) an erosion 417 event possibly linked to climate change deflated the surface until sediments which were 418 419 deposited between 17-18 ka were exposed. (3) Soil processes reworked the exposed erosional 420 boundary (layer 2, unit 1), and the resulting short sunlight re-exposure of the surface led to 421 heterogeneous bleaching of the quartz grains before (4) reburial by continuing deposition. 422 Because feldspar signals bleach more slowly, they were re-buried before losing much of their 423 signal. But, animals and soil processes could potentially also have moved some modern 424 material to the top of L1, and our samples could have been taken inadvertently from the many 425 krotovinas identified in layer 2.

426 Dust accumulation rates (DAR) have been calculated and plotted as a function of age in

Figure 2d. The end of MIS 3 is characterised by a change in the DAR at ~ 36.5 ka, with an

428 increase from ~23 to 33 cm ka⁻¹. The average DAR during MIS 2 is ~ 41 cm ka⁻¹, but a peak

429 in the accumulation reaches 71 cm ka⁻¹ at ~ 21 ka. It is interesting to note that this high

430 accumulation rate leads to indistinguishable luminescence ages between 20 and 23 ka. The

431 DAR decreases after the LGM, to ~ 16 cm ka⁻¹ during the Holocene.

432 Similar observations have been reported for other Tajik loess deposits. At the Hoalin section

433 in Southern Tajikistan, Wang et al. (2018) obtained DARs ranging from 46 cm ka⁻¹ to 54 cm

434 ka⁻¹ for the MIS 2 deposit. The DARs then decrease to \sim 2-5 cm ka⁻¹ between 21 and 14 ka.

435 Yang et al. (2020) also reported a sharp decrease in dust accumulation between the LGM and 436 the Holocene at a section located 10 km east of Dushanbe, with DARs ranging from ~56.8 to 6.0 cm ka⁻¹. Luminescence dating of Holocene soils in various parts of Tajikistan also show 437 438 several erosion events both during LGM and in the Holocene (Murray et al., to be submitted). 439 At the Darai Kalon section, the Holocene deposit is missing (Frechen and Dodonov, 1998) 440 and the new luminescence chronology at the Kuldara section (Buylaert et al., this issue) 441 shows that the upper stratigraphic unit is a reworked MIS 3 loess, it did not accumulate during the Holocene. Discontinuities in the sedimentary record have also been identified in other 442 443 loess deposits across Arid Central Asia (Frechen et al., 2009; Lauer et al. 2017, Song et al., 444 2021, Zhang et al., 2020).

Our study shows no evidence of pedogenesis at the end of MIS 3 and during MIS 2 in the Khonako II section, which, together with the DARs and the firm magnetic susceptibility properties of loess, argues for stable dry and cold conditions in the piedmonts of the Pamir Mountains (Meshcheryakova et al., 2023). Even though our data provide additional evidence for a deflation event or soil reworking processes between the LGM and the Holocene, the Khovaling plateau was still dominated by active loess sedimentation over the last ~39 ka.

451 6. Conclusion

We have applied high-resolution quartz OSL and pIRIR_{200,290} dating to the upper part of the Khonako II section. The elevated temperature polymineral pIRIR signal has satisfactory luminescence characteristics, and the quartz OSL ages are generally consistent with the pIRIR ages. This indicates that our quartz doses can be reliably determined up to ~120 Gy, despite the addition of a linear component to the dose response curve fit. The calculated residual of the pIRIR signal is equivalent to 9.2 \pm 0.4 Gy, which corresponds to a few ka, and thus is not significant when dating older samples.

459 The luminescence chronology confirms the deposition of the upper part of the Late 460 Pleistocene loess unit during MIS 3 and 2, between ~20 and ~39 ka. Our results show an active and continuous sedimentation during the last part of the last glacial stage. The DAR 461 increased at the end of MIS 3 and reached a peak during the LGM. The DAR is much lower 462 463 during the Holocene. We also identified a hiatus at the transition between the LGM and the 464 Holocene, which was not detected in the field. This highlights the importance of high-465 resolution dating in loess for reliable interpretation of climate proxies and accurate calculation 466 of DAR.

467

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- thanked for help with preparation of gamma spectrometry samples.

472 **Figure captions**

473 Figure 1. Location of the Khonako II section (A-B) and general view of the upper part of the
474 Khonako II section (C). The red band (C) indicates the studied sections. ©Ramona Schneider
475 (A) and ©Gábor Újvári (B).

Figure 2. A. Stratigraphic chart of the upper part of the Khonako II section. The ages quoted

- 477 on the stratigraphic chart are the measured OSL ages (in blue) and the $pIRIR_{200,290}$ ages (in
- red) after subtraction of a residual of 9.2±0.4 Gy. B. Frequency-dependent magnetic
- 479 susceptibility. C. Luminescence ages and Bayesian chronological model. The quartz ages
- 480 (blue circles) have been obtained by interpolation onto dose response curves fitted with a
- 481 single exponential with a linear component function. The $pIRIR_{200,290}$ ages (red open circles)
- are given after subtraction of the residual. Bayesian modelling results (solid line) obtained
- using the Bacon Model (Blaauw and Christen, 2011) of the quartz OSL ages (without
- 484 systematic uncertainties) are shown at 95% confidence (dashed lines). D. Dust accumulation
- rate (DAR) as a function of the Bayesian mean age estimates calculated every 5 cm.
- 486 Figure 3. Quartz luminescence characteristics. A) Typical dose response curve fitted with a
- 487 single saturating exponential function (dashed line) and a single exponential with a linear
- 488 component function (solid line). The inset shows the initial 5 s of a typical normalised OSL
- stimulation curve (sample 228881, note the initial 10 dead channels) as well as the
- 490 corresponding curve for Risø calibration quartz (Hansen et al., 2015). B) Estimated average
- dose for sample 228844 as a function of preheat temperature. Each point is an average of 4
- aliquots. The solid horizontal line indicates the average dose irrespective of preheat
- temperature and the dashed lines indicate the 68% confidence level. The solid vertical line
- 494 indicates the chosen preheat temperature for measurement of all quartz samples.
- 495 Figure 4. Polymineral pIRIR characteristics. A) Typical polymineral pIRIR_{200,290} dose
- 496 response curve. B) Estimated average pIRIR(x,290) dose for sample 228845 as a function of
- 497 the first IR stimulation temperature (x). Each point is an average of 6 aliquots, except for the
- 498 200 °C point (n=12). C) Dose recovery ratios for samples 228842 and 228871 bleached for 8
- days in a solar simulator. D) Additive dose recovery experiment (SARA) on sample 228842
- using test doses of 30% and 80-100% of the expected dose.

- 501 Figure 5. Comparison of the quartz OSL ages and the polymineral pIRIR_{200,290} ages (solid
- 502 circles). The black dashed line corresponds to the y=x line; the solid black line is a linear fit
- with a forced unit slope of the data and an intercept of 2.18±0.09 ka. The blue squares
- represent the modern dust samples. The samples (228846,-47,-48) in the ellipse are not
- 505 considered in the fitting (see text for details).

506 Supplementary information

- Figure S1. Quartz dose recovery ratios for samples 228842, 228844, 228855, and 228870 for
 an EXPLIN fit of the quartz dose response curve.
- 509 Table S1. Quartz and polymineral luminescence results. w.c. water content, (n_a) number of
- 510 accepted aliquots. Uncertainties are given at 1 σ and contain both random and systematic
- 511 components. Samples with poor OSL IR depletion ratios are denoted with N.A. (no De
- 512 given).
- 513 Table S2. Radionuclide concentrations, dry alpha, beta and gamma dose rates.
- Table S3. Water content values from the pits dug during autumn 2022 and spring 2023
- seasons at the top of the plateau, ~35 m from the cliff where the Khonako II section is located.
- 516 Table S4. Long-term burial water content model. The observed water content values and the
- 517 Holocene duration are used as input in the model. Note: for samples 228842 to 228847, the
- average of the observed water content values from the pits $(13\pm5\%)$ was used for the dose
- 519 rate calculation.
- 520 Table S5. Modern dust sample results: quartz OSL, IR₅₀, pIRIR_{50,180}, pIRIR_{200,290} D_e values.
- 521 IR50 results come from the pIRIR_{50,180} protocol. IQR (interquartile range) criterion was used
- 522 to reject outliers $(\pm 1.5 \text{xIQR})$, number of outliers denoted with (nr).

523 **Bibliography**

- Autzen, M., Andersen, C.E., Bailey, M., Murray, A.S., 2022. Calibration quartz: An update
 on dose calculations for luminescence dating. Radiation Measurements 157, 106828.
- 526 <u>https://doi.org/10.1016/j.radmeas.2022.106828</u>
- 527 Ballarini, M., Wallinga, J., Wintle, A.G., Bos, A.J.J., 2007. A modified SAR protocol for
- optical dating of individual grains from young quartz samples. Radiation Measurements 42,
 360–369. https://doi.org/10.1016/j.radmeas.2006.12.016
- 530 Blaauw, M., Christen, J.A., 2011. Flexible palaeoclimate age-depth models using an
- autoregressive gamma process. Bayesian Analysis 6, 457–474. <u>https://doi.org/10.1214/11-</u>
 BA618
- 533 Bøtter-Jensen, L., Thomsen, K.J., Jain, M., 2010. Review of optically stimulated
- luminescence (OSL) instrumental developments for retrospective dosimetry. Radiation
 Measurements 45, 253–257. https://doi.org/10.1016/j.radmeas.2009.11.030
- Buylaert J.-P., Challier A., Taratunina1 N.A., Kulakova E.P., Thomsen K.J., Utkina A.O.,
 P.M., Tokareva O.A., Anoikin A.A., Khujageldiev T.U., Karayev A.C., Ubaydulloev N.K.,
- P.M., Tokareva O.A., Anoikin A.A., Khujageldiev T.U., Karayev A.C., Ubaydulloev N.K.,
 Murray A.S., Kurbanov R.N. A luminescence dating study of the upper part of the loess-
- 538 Multay A.S., Kuldanov K.N. A luminescence dating study of the upper part of the loss-
- palaeosol sequence at Kuldara, Khovaling Loess Plateau, Tajikistan. Submitted to QuaternaryGeochronology.
- 541 Buylaert, J.P., Murray, A.S., Vandenberghe, D., Vriend, M., De Corte, F., Van den haute, P.,
- 542 2008. Optical dating of Chinese loess using sand-sized quartz: Establishing a time frame for
- 543 Late Pleistocene climate changes in the western part of the Chinese Loess Plateau. Quaternary
- 544 Geochronology 3, 99–113. https://doi.org/10.1016/j.quageo.2007.05.003
- 545 Buylaert, J.P., Vandenberghe, D., Murray, A.S., Huot, S., De Corte, F., Van den Haute, P.,
- 546 2007. Luminescence dating of old (>70ka) Chinese loess: A comparison of single-aliquot
- 547 OSL and IRSL techniques. Quaternary Geochronology 2, 9–14.
- 548 https://doi.org/10.1016/j.quageo.2006.05.028
- 549 Buylaert, J.-P., Thiel, C., Murray, A., Vandenberghe, D., Yi, S., Lu, H., 2011. IRSL and post-
- 550 IR IRSL residual doses recorded in modern dust samples from the Chinese Loess Plateau.
- 551 Geochronometria 38, 432–440. <u>https://doi.org/10.2478/s13386-011-0047-0</u>
- Buylaert, J.-P., Thiel, C., Murray, A.S., Jain, M., Thomsen, K.J., Sohbati, R., 2012. Extending
 the luminescence dating age range using feldspar, Quaternary Geochronology 17, 55-67.
- 554 Buylaert, J.-P., Yeo, E.-Y., Thiel, C., Yi, S., Stevens, T., Thompson, W., Frechen, M.,
- 555 Murray, A., Lu, H., 2015. A detailed post-IR IRSL chronology for the last interglacial soil at
- the Jingbian loess site (northern China). Quaternary Geochronology 30, 194–199.
- 557 <u>https://doi.org/10.1016/j.quageo.2015.02.022</u>
- 558 Chapot, M.S., Roberts, H.M., Duller, G.A.T., Lai, Z.P., 2012. A comparison of natural- and
- laboratory-generated dose response curves for quartz optically stimulated luminescence
- signals from Chinese Loess. Radiation Measurements 47, 1045–1052.
- 561 <u>https://doi.org/10.1016/j.radmeas.2012.09.001</u>
- 562 Constantin, D., Veres, D., Panaiotu, C., Anechitei-Deacu, V., Groza, S.M., Begy, R.,
- 563 Kelemen, S., Buylaert, J.P., Hambach, U., Marković, S.B. and Gerasimenko, N., 2019.

- 564 Luminescence age constraints on the Pleistocene-Holocene transition recorded in loess
- sequences across SE Europe. Quaternary Geochronology, 49, 71-77.
- 566 Cresswell, A.J., Carter, J., Sanderson, D.C.W., 2018. Dose rate conversion parameters:
- 567 Assessment of nuclear data. Radiation Measurements 120, 195–201.
- 568 <u>https://doi.org/10.1016/j.radmeas.2018.02.007</u>
- 569 Cunningham, A.C., Wallinga, J., 2010. Selection of integration time intervals for quartz OSL
- 570 decay curves. Quaternary Geochronology 5, 657–666.
- 571 <u>https://doi.org/10.1016/j.quageo.2010.08.004</u>
- 572 Dave, A.K., Timar-Gabor, A., Scardia, G., Safaraliev, N., Fitzsimmons, K.E., 2022. Variation
- in Luminescence Characteristics and Paramagnetic Defect Centres in Fine-Grained Quartz
- 574 From a Loess-Palaeosol Sequence in Tajikistan: Implications for Provenance Studies in
- Aeolian Environments. Frontiers in Earth Science 10, 835281.
- 576 <u>https://doi.org/10.3389/feart.2022.835281</u>
- 577 De Corte, F., Vandenberghe, D., De Wispelaere, A., Buylaert, J.-P., Van den Haute, P., 2006.
- 578Radon loss from encapsulated sediments in Ge gamma-ray spectrometry for the annual
- radiation dose determination in luminescence dating. Czech J Phys 56, D183–D194.
 https://doi.org/10.1007/s10582-006-1016-3
- 581 Ding, Z.L., Ranov, V., Yang, S.L., Finaev, A., Han, J.M., Wang, G.A., 2002. The loess record
- 581 Ding, Z.L., Ranov, V., Tang, S.L., Finacy, A., Han, J.M., Wang, G.A., 2002. The locus record in southern Tajikistan and correlation with Chinese loess. Earth and Planetary Science Letters 200, 387–400. <u>https://doi.org/10.1016/S0012-821X(02)00637-4</u>
- 584 Dodonov, A.E., 1991. Loess of Central Asia. GeoJournal 24, 185–194.
 585 https://doi.org/10.1007/BF00186015
- 586 Dodonov, A.E., 2002. Quaternary Period of Central Asia: Stratigraphy, Correlation,
- 587 Palaeogeography. GEOS, Moscow. (In Russian)
- 588 Dodonov, A.E., Baiguzina, L.L., 1995. Loess stratigraphy of Central Asia: Palaeoclimatic and
 589 palaeoenvironmental aspects. Quaternary Science Reviews 14, 707–720.
 590 https://doi.org/10.1016/0277-3791(95)00054-2
- 591 Dodonov, A.E., Sadchikova, T.A., Sedov, S.N., Simakova, A.N., Zhou, L.P., 2006.
- 592 Multidisciplinary approach for palaeoenvironmental reconstruction in loess-palaeosol studies
- of the Darai Kalon section, Southern Tajikistan. Quaternary International 152–153, 48–58.
 https://doi.org/10.1016/j.quaint.2005.12.001
- 595 Dodonov, A.E., Zhou, L., 2008. Loess deposition in Asia: its initiation and development
- before and during the Quaternary. Episodes Journal of International Geoscience 31, 222–225.
 https://doi.org/10.18814/epiiugs/2008/v31i2/006
- 598 Duller, G.A.T., 2003. Distinguishing quartz and feldspar in single grain luminescence
 599 measurements. Radiation Measurements 37, 161–165. <u>https://doi.org/10.1016/S1350600 4487(02)00170-1
 </u>
- Frechen, M., Dodonov, A.E., 1998. Loess chronology of the Middle and Upper Pleistocene in
 Tadjikistan. Geol Rundsch 87, 2–20. https://doi.org/10.1007/s005310050185

- Frechen, M., Kehl, M., Rolf, C., Sarvati, R., Skowronek, A., 2009. Loess chronology of the
- 604 Caspian Lowland in Northern Iran. Quaternary International 198, 220–233.
- 605 <u>https://doi.org/10.1016/j.quaint.2008.12.012</u>
- 606 Guérin, G., Christophe, C., Philippe, A., Murray, A.S., Thomsen, K.J., Tribolo, C., Urbanova,
- 607 P., Jain, M., Guibert, P., Mercier, N., Kreutzer, S., Lahaye, C., 2017. Absorbed dose,
- equivalent dose, measured dose rates, and implications for OSL age estimates: Introducing
- the Average Dose Model. Quaternary Geochronology 41, 163–173.
- 610 <u>https://doi.org/10.1016/j.quageo.2017.04.002</u>
- Hansen, V., Murray, A., Buylaert, J.-P., Yeo, E.-Y., Thomsen, K., 2015. A new irradiated
- quartz for beta source calibration. Radiation Measurements 81, 123–127.
- 613 <u>https://doi.org/10.1016/j.radmeas.2015.02.017</u>
- Hansen, V., Murray, A., Thomsen, K., Jain, M., Autzen, M., Buylaert, J.-P., 2018. Towards
- the origins of over-dispersion in beta source calibration. Radiation Measurements120, 157–
 <u>https://doi.org/10.1016/j.radmeas.2018.05.014</u>
- Huntley, D.J., Baril, M.R., 1997. The K content of the K-feldspars being measured in optical
 dating or in thermoluminescence dating. Ancient Tl 15, 11–13.
- 619 Kurbanov, R.N., Buylaert, J.-P., Stevens, T., Taratunina, N.A., Belyaev, V.R., Makeev, A.O.,
- 620 Lebedeva, M.P., Rusakov, A.V., Solodovnikov, D., Költringer, C., Rogov, V.V., Streletskaya,
- 621 I.D., Murray, A.S., Yanina, T.A., 2022. A detailed luminescence chronology of the Lower
- 622 Volga loess-palaeosol sequence at Leninsk. Quaternary Geochronology 73, 101376.
- 623 <u>https://doi.org/10.1016/j.quageo.2022.101376</u>
- Lai, Z.P., Zöller, L., Fuchs, M., Brückner, H., 2008. Alpha efficiency determination for OSL
- of quartz extracted from Chinese loess. Radiation Measurements 43, 767–770.
- 626 <u>https://doi.org/10.1016/j.radmeas.2008.01.022</u>
- 627 Lai, Z., 2010. Chronology and the upper dating limit for loess samples from Luochuan section
- 628 in the Chinese Loess Plateau using quartz OSL SAR protocol. Journal of Asian Earth
- 629 Sciences 37, 176–185. https://doi.org/10.1016/j.jseaes.2009.08.003
- 630 Lauer, T., Vlaminck, S., Frechen, M., Rolf, C., Kehl, M., Sharifi, J., Lehndorff, E., Khormali,
- 631 F., 2017. The Agh Band loess-palaeosol sequence A terrestrial archive for climatic shifts
- during the last and penultimate glacial–interglacial cycles in a semiarid region in northern
- 633 Iran. Quaternary International 429, 13–30. <u>https://doi.org/10.1016/j.quaint.2016.01.062</u>
- Li, B., Li, S.-H., 2012a. Luminescence dating of Chinese loess beyond 130 ka using the non-fading signal from K-feldspar. Quaternary Geochronology 10, 24–31.
- 636 <u>https://doi.org/10.1016/j.quageo.2011.12.005</u>
- 637 Li, B., Li, S., 2012b. A reply to the comments by Thomsen et al. on "Luminescence dating of
- K-feldspar from sediments: a protocol without anomalous fading correction." Faculty of
 Science Papers (Archive) 49–51. <u>https://doi.org/10.1016/j.quageo.2011.10.001</u>
- 640 Li, B., Li, S.-H., 2011. Luminescence dating of K-feldspar from sediments: A protocol
- 641 without anomalous fading correction. Quaternary Geochronology 6, 468–479.
- 642 <u>https://doi.org/10.1016/j.quageo.2011.05.001</u>

- Li, Y., Zhou, L., 2021. Variations of Thermally and Optically Stimulated Luminescence 643
- 644 Sensitivity of Loess and Pedocomplex Samples from Southern Tajikistan, Central Asia.
- Geochronometria 48, 242-252. https://doi.org/10.1515/geochr-2015-0118 645

646 Li, G., Wen, L., Xia, D., Duan, Y., Rao, Z., Madsen, D.B., Wei, H., Li, F., Jia, J., Chen, F.,

- 2015. Quartz OSL and K-feldspar pIRIR dating of a loess/palaeosol sequence from arid 647 central Asia, Tianshan Mountains, NW China. Quaternary Geochronology 28, 40-53. 648
- 649 https://doi.org/10.1016/j.quageo.2015.03.011
- 650 Li, G., Yang, H., Stevens, T., Zhang, X., Zhang, H., Wei, H., Zheng, W., Li, L., Liu, X.,
- Chen, J., Xia, D., Oldknow, C., Ye, W., Chen, F., 2020. Differential ice volume and orbital 651
- 652 modulation of Quaternary moisture patterns between Central and East Asia. Earth and
- Planetary Science Letters 530, 115901. https://doi.org/10.1016/j.epsl.2019.115901 653
- 654 Martin, L., Mercier, N., Incerti, S., 2014. Geant4 simulations for sedimentary grains in infinite matrix conditions: The case of alpha dosimetry. Radiation Measurements 70, 39-47. 655 656 https://doi.org/10.1016/j.radmeas.2014.09.003
- 657 Mejdahl, V., 1979. Thermoluminescence dating: beta-dose attenuation in quartz grains. 658 Archaeometry 21, 61-72.
- Mejdahl, V., Bøtter-Jensen, L., 1994. Luminescence dating of archaeological materials using 659 660 a new technique based on single aliquot measurements. Quaternary Science Reviews 13, 551-554. https://doi.org/10.1016/0277-3791(94)90076-0 661
- Meshcheryakova, O.A., Kurbanov, R.N., Pavlov, V.E., 2023. Reconstructions of paleowind 662 663 directions in the Pleistocene: Evidence from the anisotropy of magnetic susceptibility of the 664 loess-paleosol series of Tajikistan and the Azov region. Izvestiya. Physics of the Solid Earth 59, 704–716. 665
- Murray A., Challier A., Buylaert J.-P., Schneider R., Khormali F., Navi S.V., Sosin P., 666
- 667 Kurbanov R.N. Did the surface soils to be used for calibration of Central Asian climate
- 668 proxies form under the current climate? Quaternary Geochronology. To be submitted
- 669 Murray, A., Arnold, L.J., Buylaert, J.-P., Guérin, G., Qin, J., Singhvi, A.K., Smedley, R.,
- 670 Thomsen, K.J., 2021. Optically stimulated luminescence dating using quartz. Nat Rev
- 671 Methods Primers 1, 72. https://doi.org/10.1038/s43586-021-00068-5
- Murray, A.S., 1996. Developments in optically transferred luminescence and photo-672
- 673 transferred thermoluminescence dating: application to a 2000-year sequence of flood deposits.
- 674 Geochimica et Cosmochimica Acta 60, 565-576.
- Murray, A.S., Helsted, L.M., Autzen, M., Jain, M., Buylaert, J.P., 2018. Measurement of 675
- 676 natural radioactivity: Calibration and performance of a high-resolution gamma spectrometry
- facility. Radiation Measurements 120, 215-220. 677
- 678 https://doi.org/10.1016/j.radmeas.2018.04.006
- Murray, A.S., Marten, R., Johnston, A., Martin, P., 1987. Analysis for naturally occuring 679
- 680 radionuclides at environmental concentrations by gamma spectrometry. Journal of
- Radioanalytical and Nuclear Chemistry Articles 115, 263–288. 681
- https://doi.org/10.1007/BF02037443 682

- 683 Murray, A.S., Thomsen, K.J., Masuda, N., Buylaert, J.P., Jain, M., 2012. Identifying well-
- bleached quartz using the different bleaching rates of quartz and feldspar luminescence
- signals. Radiation Measurements 47, 688–695. <u>https://doi.org/10.1016/j.radmeas.2012.05.006</u>
- 686 Murray, A.S., Wintle, A.G., 2003. The single aliquot regenerative dose protocol: potential for
- 687 improvements in reliability. Radiation Measurements 37, 377–381.
- 688 <u>https://doi.org/10.1016/S1350-4487(03)00053-2</u>
- 689 Murray, A.S., Wintle, A.G., 2000. Luminescence dating of quartz using an improved single-690 aliquot regenerative-dose protocol. Radiation Measurements 32, 57–73.
- 691 https://doi.org/10.1016/S1350-4487(99)00253-X
- 692 Parviz, N., Shen, Z., Yunus, M., Zulqarnain, S., 2020. Loess deposits in southern Tajikistan
- 693 (Central Asia): Magnetic properties and palaeoclimate. Quaternary Geochronology 60,
 694 101114. <u>https://doi.org/10.1016/j.quageo.2020.101114</u>
- Prescott, J.R., Hutton, J.T., 1994. Cosmic ray contributions to dose rates for luminescence and
 ESR dating: Large depths and long-term time variations. Radiation Measurements 23, 497–
- 697 500. <u>https://doi.org/10.1016/1350-4487(94)90086-8</u>
- 698 Schäfer, J., Ranov, V.A., Sosin, P.M., 1998. The "Cultural Evolution" of man and the
- 699 chronostratigraphical background of changing environments in the loess palaeosol sequences
- of Obi-Mazar and Khonako (Tadjikistan). Anthropologie (1962-) 36, 121–135.
- Schmidt, C., Bösken, J., Kolb, T., 2018. Is there a common alpha-efficiency in polymineral
 samples measured by various infrared stimulated luminescence protocols? Geochronometria
 45, 160–172. https://doi.org/10.1515/geochr-2015-0095
- Sohbati, R., Kook, M., Pirtzel, L.P., Thomsen, K.J., 2021. Safelight for OSL dating
 laboratories: a follow-up study, Ancient TL 39.
- Song, Y., Li, Y., Cheng, L., Zong, X., Kang, S., Ghafarpour, A., Li, X., Sun, H., Fu, X.,
- 707 Dong, J., Mamadjanov, Y., Orozbaev, R., Shukurov, N., Gholami, H., Shukurov, S., Xie, M.,
- 2021. Spatio-temporal distribution of Quaternary loess across Central Asia. Palaeogeography,
- Palaeoclimatology, Palaeoecology 567, 110279. <u>https://doi.org/10.1016/j.palaeo.2021.110279</u>
- 710 Spooner, N.A., 1994. The anomalous fading of infrared-stimulated luminescence from
- feldspars. Radiation Measurements 23, 625–632. <u>https://doi.org/10.1016/1350-</u>
- 712 <u>4487(94)90111-2</u>
- 713 Stevens, T., Buylaert, J.-P., Thiel, C., Újvári, G., Yi, S., Murray, A.S., Frechen, M., Lu, H.,
- 2018. Ice-volume-forced erosion of the Chinese Loess Plateau global Quaternary stratotype
- 715 site. Nat Commun 9, 983. <u>https://doi.org/10.1038/s41467-018-03329-2</u>
- 716 Taratunina, N.A., Buylaert, J.-P., Kurbanov, R.N., Yanina, T.A., Makeev, A.O., Lebedeva,
- 717 M.P., Utkina, A.O., Murray, A.S., 2022. Late Quaternary evolution of lower reaches of the
- 718 Volga River (Raygorod section) based on luminescence dating. Quaternary Geochronology
- 719 72, 101369. https://doi.org/10.1016/j.quageo.2022.101369
- 720 Thiel, C., Buylaert, J.-P., Murray, A., Terhorst, B., Hofer, I., Tsukamoto, S., Frechen, M.,
- 721 2011. Luminescence dating of the Stratzing loess profile (Austria) Testing the potential of

an elevated temperature post-IR IRSL protocol. Quaternary International 234, 23–31.
 <u>https://doi.org/10.1016/j.quaint.2010.05.018</u>

Thomsen, K.J., Murray, A.S., Jain, M., Bøtter-Jensen, L., 2008. Laboratory fading rates of
various luminescence signals from feldspar-rich sediment extracts. Radiation Measurements
43, 1474–1486. https://doi.org/10.1016/j.radmeas.2008.06.002

Tian, S., Sun, J., Zhang, Z., Abdulov, S., Cao, M., Gadoev, M., Oimahmadov, I., 2021. Loess
deposits in the Tajik Basin, Central Asia: chronology, provenance and palaeoclimatic

implications since the Last Glacial. Boreas 50, 147–166. <u>https://doi.org/10.1111/bor.12467</u>

Timar-Gabor, A., Wintle, A.G., 2013. On natural and laboratory generated dose response
curves for quartz of different grain sizes from Romanian loess. Quaternary Geochronology
18, 34–40. <u>https://doi.org/10.1016/j.quageo.2013.08.001</u>

733 Volvakh, N.E., Kurbanov, R.N., Zykina, V.S., Murray, A.S., Stevens, T., Költringer, C.A.,

Volvakh, A.O., Malikov, D.G., Taratunina, N.A., Buylaert, J.-P., 2022. First high-resolution
luminescence dating of loess in Western Siberia. Quaternary Geochronology 73, 101377.
<u>https://doi.org/10.1016/j.quageo.2022.101377</u>

737 Wang, L., Jia, J., Li, G., Li, Z., Wang, X., Chen, F., 2018. Fine-grained quartz OSL dating

chronology of loess sequence from southern Tajikistan: Implications for climate change in
arid central Asia during MIS 2. Journal of Asian Earth Sciences 155, 116–123.

740 <u>https://doi.org/10.1016/j.jseaes.2017.11.001</u>

741 Yang, S., Li, D., Liu, N., Zan, J., Liu, W., Kang, J., Murodov, A., Fang, X., 2020. Quartz

optically stimulated luminescence dating of loess in Tajikistan and its palaeoclimatic

implications for arid Central Asia since the Lateglacial. Palaeogeography, Palaeoclimatology,
Palaeoecology 556, 109881. <u>https://doi.org/10.1016/j.palaeo.2020.109881</u>

Yi, S., Buylaert, J.-P., Murray, A.S., Thiel, C., Zeng, L., Lu, H., 2015. High resolution OSL

and post-IR IRSL dating of the last interglacial–glacial cycle at the Sanbahuo loess site

747 (northeastern China). Quaternary Geochronology 30, 200–206.

748 https://doi.org/10.1016/j.quageo.2015.02.013

Yi, S., Buylaert, J.-P., Murray, A.S., Lu, H., Thiel, C., Zeng, L., 2016. A detailed post-IR

750 IRSL dating study of the Niuyangzigou loess site in northeastern China. Boreas 45, 644–657.
 751 <u>https://doi.org/10.1111/bor.12185</u>

Yi, S., Li, X., Han, Z., Lu, H., Liu, J., Wu, J., 2018. High resolution luminescence chronology

for Xiashu Loess deposits of Southeastern China. Journal of Asian Earth Sciences 155, 188–

754 197. <u>https://doi.org/10.1016/j.jseaes.2017.11.027</u>

Zan, J., Ning, W., Heller, F., Fang, X., Zhang, W., Kang, J., 2022. Intensified Northern

Hemisphere Glaciation Facilitates Continuous Accumulation of Late Pliocene Loess on the
 Western Margin of the Pamir. Geophysical Research Letters 49, e2022GL099629.

758 https://doi.org/10.1029/2022GL099629

759 Zhang, J., Tsukamoto, S., Long, H., 2023. Testing the potential of pulsed post-IR IRSL dating

on Chinese loess deposits. Quaternary Geochronology 78, 101469.

761 <u>https://doi.org/10.1016/j.quageo.2023.101469</u>

- 762 Zhang, J., Zhou, X., Long, H., 2020. Late Quaternary loess accumulation at the Rudak section
- in Uzbekistan, central Asia: Chronology and palaeoclimate implications. Palaeogeography,
- 764 Palaeoclimatology, Palaeoecology 547, 109695. <u>https://doi.org/10.1016/j.palaeo.2020.109</u>

Journal Prevention











Declaration of interests

 The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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