

1 **Optically stimulated luminescence dating of heat retainer hearths from the Sahara: Insights**  
2 **into signal accumulation and measurement.**

3  
4 S.J. Armitage<sup>1,2\*</sup>, A. Krishna<sup>1</sup>, L.E. Parker<sup>1,3</sup>, G.E. King<sup>4</sup>

5  
6 <sup>1</sup>Centre for Quaternary Research, Department of Geography, Royal Holloway, University of  
7 London, Egham, Surrey, TW20 0EX, United Kingdom.

8 <sup>2</sup>SFF Centre for Early Sapiens Behaviour (SapienCE), University of Bergen, Post Box 7805, 5020,  
9 Bergen, Norway.

10 <sup>3</sup>Archaeological Research Services Ltd, Angel House, Portland Square, Bakewell, Derbyshire,  
11 DE45 1HB, United Kingdom.

12 <sup>4</sup>Universität Bern, Institut für Geologie, Baltzerstrasse 1+3, 3012 Bern, Switzerland.  
13

14 **Abstract**

15 Heat retainer hearths are a prominent component of the Holocene archaeological record of a number  
16 of drylands. Rocks within these hearths were fired in antiquity, emptying the optically stimulated  
17 luminescence (OSL) source traps of mineral grains within the rock. Since partial bleaching and  
18 mixing of grains within a lithified heat retainer is impossible, these rocks offer the opportunity to  
19 test our understanding of OSL signal accumulation and measurement processes. First, we show that  
20 OSL ages calculated using grains size fractions from 4-11  $\mu\text{m}$  up to 180-210  $\mu\text{m}$  are  
21 indistinguishable for a single heat retainer, indicating that the environmental and instrumental dose  
22 rate correction factors routinely used in luminescence dating are accurate. Second, we used single-  
23 grain dose recovery and equivalent dose measurements to determine the overdispersion due to beta  
24 microdosimetry. For the heat retainers measured in this study, overdispersion due to beta  
25 microdosimetry ranges from  $8.9 \pm 1.8$  to  $20.3 \pm 1.6$  %. Third, we investigate the impact of  
26 mechanical crushing on the measured equivalent dose from quartz, to test the potential of using this  
27 technique to liberate dateable material from heat retainers which are not acid soluble. A small (<1  
28 Gy) but significant increase in equivalent dose is observed in crushed zero-age samples, but the  
29 equivalent doses of crushed and uncrushed Holocene quartzes are indistinguishable. We conclude  
30 that crushing is a viable method for extracting dateable material from a heat retainer, but that some  
31 knowledge of the dosimeter's mean grain size is required for calculation of an accurate  
32 environmental dose rate.  
33

---

\* e-mail: simon.armitage@rhul.ac.uk

34 *Keywords:* optically stimulated luminescence; hearths; dose rate; microdosimetry; crushing.

35

## 36 **1. Introduction**

37 Heat retainer hearths are a prominent component of the Holocene archaeological record, and are  
38 particularly prevalent in the Sahara and Arabian Peninsula, where they are frequently associated with  
39 surface scatters which attest to the presence of ancient nomadic pastoralists. Rocks, which acted as  
40 heat retainers while the hearth was in use, are heated to sufficiently high temperatures to completely  
41 empty the OSL source traps. Consequently, mineral grains from within these heat retainer rocks are  
42 dateable using optically stimulated luminescence (OSL) techniques, providing an estimate of the time  
43 elapsed since last firing (Armitage and King, 2013; Rhodes et al., 2010; Rhodes et al., 2009). These  
44 fired heat retainers are often the only dateable and unequivocally anthropogenic component of the  
45 surface archaeological record. OSL dating of hearths is therefore potentially an important tool for  
46 understanding the nature of the surface archaeological record in drylands. Furthermore, fired hearth  
47 stones offer a number of advantages when studying the accumulation of the OSL signal. First, since  
48 the heat retainers are fired in a lithified state, and since post-firing mixing of mineral grains within a  
49 heat retainer is impossible, OSL dating of multiple grain-size fractions from a single rock allows the  
50 accuracy of grain-size specific corrections to environmental and instrumental dose rates to be tested.  
51 Second, since post-firing mixing of mineral grains is impossible, and since Armitage and King (2013,  
52 their Figure 5) have demonstrated that sunlight does not deplete the OSL signal in the inner portion  
53 of hearth rocks, sand sized quartz extracted from hearth rocks may be used to investigate single-grain  
54 equivalent dose ( $D_e$ ) overdispersion due to beta microdosimetry. In the present study we: 1) Conduct  
55 paired single-grain dose recovery and  $D_e$  measurement experiments to investigate the degree of  
56 overdispersion caused by beta microdosimetry; 2) Calculate ages for a range of grain-size fractions  
57 extracted from a single heat retainer to test the internal consistency of the OSL method, and 3)  
58 Investigate the possibility of extending the OSL dating technique to non-carbonate lithologies where  
59 mineral grains cannot be extracted from a heat retainer by acid dissolution.

60

## 61 **2 Materials and methods**

62 A total of thirteen heat retainers were collected from five hearths at the Al Wafa archaeological site,  
63 in the Libyan Sahara. A full site description is found in Armitage and King (2013). Briefly, Al Wafa  
64 is a gently sloping basin characterised by shallow, ephemeral drainage channels cut into an  
65 extensively deflated surface composed of unconsolidated sand protected by gravel-pebble sized  
66 carbonate clasts. The landscape is dominated by ~100 discrete hearths, which are usually seen as  
67 mounds rising a few tens of centimetres above the surrounding land surface (Figure 1), and date from  
68 ~7-9 ka (Armitage and King, 2013). These mounds are believed to result from topographic inversion,

69 with the hearths originally consisting of cobble lined pits dug into soil, which was subsequently  
70 deflated when the Sahara dried ~5 ka (Armitage et al., 2015). The cobbles, which served as heat  
71 retainers when the hearth was in use, are clasts of sandy limestone, which outcrops at the margin of  
72 the Al Wafa basin. Approximately cuboid heat retainers with no visible fracturing were sampled from  
73 discrete hearths.

74  
75 The outer light exposed portions of the heat retainers was removed by repeated immersion in 1.16 M  
76 HCl, until the length of each axis of the clast had been reduced by a minimum of 25 mm. The dating  
77 sample was obtained by dissolution of the remaining material. Quartz separates were produced using  
78 the methods summarised in Supplemental Material Section A. For samples FZ39-48, only the 210-  
79 180  $\mu\text{m}$  fraction was prepared for measurement. For samples FZ84, 85, 95 and 96, dating samples  
80 were prepared from the 4-11, 11-20, 20-40, 40-60, 60-90, 90-125, 125-150, 150-180 and 180-210  $\mu\text{m}$   
81 fractions. All size fractions were mounted on 10 mm aluminium discs. Additional single-grain  
82 analysis (212-180  $\mu\text{m}$  grains) was performed using standard Risø single-grain sample holders.

83  
84 All OSL measurements presented in this study were carried out using a Risø TL/OSL-DA-15 dating  
85 system (Bøtter-Jensen et al., 2003), fitted with a single-grain OSL attachment (Duller et al., 1999).  
86 Optical stimulation of single aliquots was carried out using a blue ( $470 \pm 30$  nm) light emitting diode  
87 (LED) array with a power density of  $33 \text{ mW/cm}^2$ , while single-grains were stimulated using a 10 mW  
88 Nd : YVO<sub>4</sub> solid-state diode-pumped green laser (532 nm) focussed to yield a nominal power density  
89 of  $50 \text{ W/cm}^2$ . Infra-red (IR) stimulation was carried out using an IR (870 nm) laser diode array. OSL  
90 was measured using an Electron Tubes Ltd 9235QB15 photomultiplier tube with 7.5 mm of Hoya U-  
91 340 filter interposed between the sample and photomultiplier. Irradiation was carried out using a 40  
92 mCi <sup>90</sup>Sr/<sup>90</sup>Y beta source, calibrated relative to the National Physical Laboratory, Teddington <sup>60</sup>Co  $\gamma$ -  
93 source (Hotspot 800) (Armitage and Bailey, 2005). Due to the spatial heterogeneity of beta emitters  
94 across the active face of the <sup>90</sup>Sr/<sup>90</sup>Y beta source, it was necessary to apply a grain position correction  
95 to single-grain D<sub>e</sub> values, using the method of (Armitage et al., 2011).

96  
97 Equivalent doses were determined using the single-aliquot regenerative-dose (SAR) method (Murray  
98 and Wintle, 2000). Following Armitage and King (2013), who worked on heat retainers from the Al  
99 Wafa site, we adopted a preheating regime of 260 °C, 10 s for PH1 (the pre-heat prior to measurement  
100 of natural or regenerated luminescence) and 220 °C, 10 s for PH2 (the pre-heat prior to measurement  
101 of the test dose luminescence response) for all measurements. OSL measurements were made at 125  
102 °C, using blue diodes (40 s) for aliquot measurements or a green laser (2 s per grain) for single-grain  
103 measurements. For blue diode stimulation, the OSL signal was that recorded during the first 0.36 s of

104 stimulation, and a background signal from the last 4 s of stimulation was subtracted. For green laser  
105 stimulation, the OSL signal was that recorded during the first 0.3 s of stimulation, and a background  
106 signal from the last 0.3 s of stimulation was subtracted (Thomsen et al., 2005). Curve fitting, and  $D_e$   
107 determination, were performed using version 3.24 of the Luminescence Analyst software (Duller,  
108 2007). We adopted the single-grain rejection criteria of Armitage et al. (2011), Supplementary  
109 Material Section B.

110

### 111 **3 Age estimates generated using different grain-size fractions**

112 In principle, any grain-size fraction from fine silt to fine sand can be used to measure the equivalent  
113 dose of a sample. For any given sample, the grain size used may be dictated by availability or the  
114 post-depositional processes which a sample has experienced e.g. silt may be translocated by  
115 percolating water in a sandy sediment, but be the only clast size available in a loess deposit.  
116 Consequently, sedimentary characteristics and geomorphic setting often necessitates the  
117 measurement of different grain size fractions for samples within the same dating study. However,  
118 numerous factors determining the environmental or laboratory dose rate experienced by the  
119 luminescence dosimeter vary with grain size. These variations may be progressive, e.g. the ~13%  
120 increase in beta dose rate from a Risø irradiation unit as grain size increases from 4-11  $\mu\text{m}$  to  $>63 \mu\text{m}$   
121 (Armitage and Bailey, 2005) or abrupt e.g. at the transition between sand-sized material, which are  
122 routinely etched to remove the alpha dosed rind, and silt-sized material. If a single dating project uses  
123 more than one grain size fraction, then the minimum requirement for a successful study is that  
124 different grain sizes yield the same luminescence age for sediments which were deposited  
125 simultaneously. In practice, the types of sedimentary deposit which are normally targeted for OSL  
126 dating tend to be well sorted. Conversely, the heat retainers from Al Wafa contain quartz grains from  
127  $<4$  to  $>210 \mu\text{m}$  in diameter, all of which must have a common OSL age, making these samples ideal  
128 for testing the internal consistency of OSL ages produced using different grain size fractions.

129

130 Twelve aliquots of quartz from each size fraction from each of four samples were measured using a  
131 single luminescence reader. Across the range of grain sizes measured, progressive variations in  
132 environmental alpha (Brennan et al., 1991) and beta (Guérin and Mercier, 2012) attenuation factors,  
133 and laboratory beta dose rate (Armitage and Bailey, 2005) occurred. An abrupt alteration in dose rate  
134 occurred between the 60-90 and 90-125  $\mu\text{m}$  fractions due to HF etching of  $>90 \mu\text{m}$  grains, resulting  
135 in the loss of 100% of the environmental alpha dose (making the common assumption of isotropic  
136 etching), and 1% of the environmental beta dose (Brennan, 2003). An abrupt change in laboratory  
137 dose rate is also present between 20-40 and 40-60  $\mu\text{m}$ , since the  $<40 \mu\text{m}$  grains were deposited from  
138 suspension, coating the entire face of the disc, whereas the  $>40 \mu\text{m}$  grains were attached using

139 Silkospray applied via a 5 mm diameter mask. However, since the instrument was calibrated using  
140 samples mounted by the same methods, no correction for the known variation in instrumental dose  
141 rate between the core and periphery of the disc was made (Ballarini et al., 2006).

142  
143 Equivalent dose, dose rate and calculated age are presented for nine different grain size fractions in  
144 the range 4-210  $\mu\text{m}$  (Tables S2-S4). For sample FZ84 (Figure 2), dose rate falls steadily as grain size  
145 increases, from a value of  $0.98 \pm 0.07$  Gy/ka (4-11  $\mu\text{m}$ ) to  $0.83 \pm 0.05$  Gy/ka (180-212  $\mu\text{m}$ ), a decrease  
146 of ~18 %. For all four of the samples for which data are available, equivalent dose also falls with  
147 increasing grain size, though for each sample there are outliers from this general trend (e.g. 11-20  $\mu\text{m}$   
148 in Figure 2). Nonetheless, ages calculated for each grain size fraction from a single sample are  
149 consistent with each other. This result indicates that for these samples at least, the dose rate correction  
150 factors used in this study produce consistent ages across the full range of grain sizes commonly  
151 adopted for luminescence dating work.

152

#### 153 **4 Single-grain overdispersion due to beta microdosimetry**

154 Overdispersion (OD), the relative spread of equivalent doses after measurement uncertainties are  
155 excluded, is a key input for several of the statistical models used in single-grain dating e.g. the  
156 Minimum Age Model (MAM) (Galbraith et al., 1999) and the Finite Mixture Model (FMM) .  
157 However, successful application of these models requires an estimate of the OD of a well-bleached  
158 sample, which is otherwise identical to the sample being measured. Incorrect estimation of the well-  
159 bleached OD may lead to the adoption of the incorrect statistical model, and the production of an  
160 incorrect age. For example, an erroneously young age would be calculated if an underestimate of the  
161 well-bleached OD caused the MAM to be applied to a sample that was not partially bleached. The  
162 heterogeneous distribution of beta activity within a sediment, e.g. where occasional potassium  
163 feldspars are present within a quartz rich sand (Mayya et al., 2006), has the potential to cause such an  
164 underestimation of the well-bleached OD. Although some success has been achieved in modelling  
165 OD due to beta microdosimetry (Cunningham et al., 2012; Guérin et al., 2015; Martin et al., 2015),  
166 few studies have measured OD in ancient sediments where other causes may be excluded or isolated.  
167 Heat retainers which were fired in antiquity offer the opportunity to isolate OD due to beta  
168 microdosimetry, since lithified samples cannot experience post-depositional mixing, and firing  
169 completely empties the OSL source traps (Armitage and King, 2013, section 8), eliminating the  
170 possibility of heterogeneous bleaching. Consequently, OD observed in a  $D_e$  dataset ( $OD_{D_e}$ ) from a  
171 heat retainer consists of contributions from the measurement process (e.g. instrumental variability  
172 and intra-sample variation in luminescence characteristics) and from beta microdosimetry. Since the  
173 OD observed in a dose recovery dataset ( $OD_{Rec}$ ) is caused by the measurement process alone, it is

174 possible to isolate OD due to beta microdosimetry ( $OD_{\beta}$ ) by subtracting quadratically  $OD_{Rec}$  from  
175  $OD_{De}$ .

176

177 Dose recovery measurements were made on six samples from Al Wafa. Following Armitage and  
178 King (2013), single-grain dose recovery experiments were performed by heating samples to 350 °C  
179 for 120 s (to empty the OSL source traps) after which a beta dose similar to  $D_e$  (the “known dose”)  
180 was applied. The “recovered dose” was then measured, using an identical measurement sequence to  
181 that used for measuring the equivalent dose. Single-grain equivalent dose, dose recovery and OD  
182 values are presented in Table S6. The “dose recovery ratios” (recovered dose/known dose) ranged  
183 from  $0.96 \pm 0.01$  to  $1.00 \pm 0.01$ , with a mean value of  $0.99 \pm 0.01$ , indicating that the measurement  
184 parameters used in this study are appropriate for the Al Wafa samples.  $OD_{rec}$  values ranged from  $6.3$   
185  $\pm 0.6 \%$  to  $9.7 \pm 1.1 \%$ , with a mean value of  $7.6 \pm 1.2 \%$ .  $OD_{\beta}$  values ranged from  $15.8 \pm 1.8$  to  
186  $27.1 \pm 1.6 \%$ , with a mean value of  $22.6 \pm 4.1$ , while  $OD_{De}$  values ranged from  $18.5 \pm 1.5 \%$  to  $28.2$   
187  $\pm 1.5 \%$ , with a mean value of  $23.9 \pm 3.6 \%$ .

188

189 The wide range of  $OD_{\beta}$  values from heat retainers from a single location, and derived from a single  
190 geological unit, suggests that the effects of beta microdosimetry is highly sample dependant. This  
191 implies that, even for multiple samples from a single location, the well-bleached OD required for the  
192 application of several statistical models may need to be determined for each individual sample. In  
193 addition, only one of the six samples measured yielded an  $OD_{De}$  value below 20%. Several studies  
194 (e.g. Armitage et al., 2011) have used an  $OD_{De}$  value of 20% to distinguish between well-bleached,  
195 undisturbed samples ( $OD_{De} < 20\%$ ) and samples which have been subject to partial bleaching or post  
196 depositional mixing ( $OD_{De} > 20\%$ ). Our results contribute to a growing body of literature (Guérin et  
197 al., 2015; Thomsen et al., 2016) which does not support the use of a 20%  $OD_{De}$  threshold. Instead a  
198 site-specific, or possibly even a sample specific,  $OD_{De}$  estimate for a well-bleached sample is  
199 required. In the absence of such an estimate, partial bleaching or post depositional mixing should be  
200 diagnosed based upon a knowledge of the sample’s depositional context and burial history. This  
201 approach risks confirmation bias (the tendency to observe effects which are expected to be present),  
202 and highlights the need for a robust method for estimating the OD expected from a well-bleached  
203 sample.

204

## 205 **5 Extending the OSL dating technique to non-carbonate lithologies**

206 Where heat retainers are composed of carbonate rock, extraction of dateable material is easily  
207 achieved using progressive HCl dissolution to remove the outer light-exposed portions of the rock  
208 (e.g. Armitage and King, 2013). Conversely non-carbonate heat retainers have been subject to more

209 complicated preparation procedures. For example, Rhodes et al. (2010) removed the light exposed  
210 portion of the rock using a geological hammer, followed by two cycles of mechanical crushing (one  
211 producing pebble sized fragments, and the other yielding sand sized fragments) and a short HF etch  
212 to remove surface crushing effects and feldspar. This latter approach has the disadvantage of being  
213 time-consuming and, depending upon the mineralogical composition of the parent rock, potentially  
214 yielding a polymineral sample with unpredictable luminescence characteristics. An alternative  
215 approach is to remove the outer portions of the rock with a geological hammer, and crush the  
216 remaining dating fraction in a mill, preparing the resulting powder using the standard method for 4-  
217 11  $\mu\text{m}$  silt. The advantages of this approach is that it is less labour intensive, is likely to yield a more  
218 predictably pure quartz separate, and presents the sample to the luminescence reader in a predictable  
219 geometry. As with the method of Rhodes et al, (2010), this method requires a realistic approximation  
220 of the mean grain size of the dosimeter before crushing to calculate an accurate dose rate. More  
221 significantly, a crushing induced increase in signal has been observed for burnt flint samples (Aitken,  
222 1985), section 7.1), while recent evidence suggests that crushing or shearing may either reduce or  
223 reset the natural luminescence signal (Bateman et al., 2012; Swift et al., 2011).

224

225 To test the possibility that milling induces a luminescence signal, a selection of seven zero age quartz  
226 separates were measured in their crushed and uncrushed states. Similarly, to test the possibility that  
227 milling reduces the luminescence signal, a selection of five quartz separates from Holocene dune  
228 samples ( $D_e$  ranging from 2-20 Gy) were measured in their crushed and uncrushed states. Finally, to  
229 test the utility of crushing as a method for extracting dateable quartz from non-carbonate heat  
230 retainers, small chips from the core of the four heat retainers reported in Section 3 were crushed, and  
231 the  $D_e$  compared to that for material extracted via sequential dissolution in HCl. Crushed samples  
232 were prepared by loading  $\sim 2$  g of material into a light-tight stainless steel grinding jar, and grinding  
233 at 20 Hz for 120 s using a Retsch Mixer Mill MM400. The 4-11  $\mu\text{m}$  fraction was subsequently  
234 extracted from the milled material using the method described in Supplemental Material Section A.  
235 Results are presented in Supplementary Material Tables 3-5.

236

237 Of the seven zero age samples measured (Table S7), four showed a significant increase in equivalent  
238 dose upon crushing, while three yielded indistinguishable values. The mean increase in equivalent  
239 dose due to crushing was  $0.25 \pm 0.14$  Gy, ranging from  $0.08 \pm 0.05$  Gy for a Libyan desert dune sand  
240 (Armitage et al., 2007) to  $0.93 \pm 0.33$  Gy for a South African coastal dune sand (this study). From  
241 these data it is apparent that crushing can induce a significant increase in  $D_e$ , but that the effect is  
242 neither universal nor uniform. None of the five Holocene samples yielded crushed/uncrushed ratios  
243 distinguishable from unity (Table S8), suggesting that any signal increase due to crushing is either

244 negligible or masked by other sources of  $D_e$  scatter. Crushed material from the cores of the four heat  
245 retainers yielded equivalent doses consistent with the range of values obtained from uncrushed  
246 material (Tables S2-S5). However, to determine an age, the mean grain size of the uncrushed quartz  
247 must be known to determine the applicable dose rate. Without this information i.e. assuming mean  
248 grain size of the dosimeter to be somewhere between 4 and 210  $\mu\text{m}$ , a large uncertainty term needs  
249 to be used to account for the  $\sim 15\%$  variation in environmental dose rate between these grain sizes.  
250 In the case of the four heat retainers measured here, the effective grain size of the quartz prior to  
251 crushing may be determined by calculating the dose rate required for the equivalent dose of the  
252 crushed material to yield the mean age determined using the various grain size fractions presented in  
253 Section 3. This analysis indicates that heat retainers contained grains with a mean effective size of  
254 40-60  $\mu\text{m}$  (n=2), 90-150  $\mu\text{m}$  (n=1) and  $>210\ \mu\text{m}$  (n=1). For the samples in question, environmental  
255 dose rates decrease by  $7.7 \pm 0.8\%$  from 40-60 to 180-210  $\mu\text{m}$ . Consequently, estimation of the mean  
256 grain size either by visual inspection or petrography may yield acceptably precise estimates of the  
257 environmental dose rate. These data suggest that crushing is a viable technique for extracting dateable  
258 mineral separates from heat retainers with non-carbonate lithologies.

259

## 260 **6 Conclusions**

261 Heat retainers from ancient hearths offer the opportunity to test our understanding of OSL signal  
262 accumulation and measurement processes. We measure equivalent doses from a range of grain sizes  
263 commonly adopted in luminescence dating studies. For each hearth stone, different size fractions  
264 yield similar ages, indicating that for our samples at least, the instrumental and environmental dose  
265 rate correction factors used in luminescence dating yield internally consistent (accurate?) results. In  
266 addition, since mineral grains within heat retainers cannot be subject to partial bleaching or post firing  
267 mixing, overdispersion in single-grain data from these samples is due to measurement processes and  
268 beta microdosimetry. The latter may be isolated by subtracting quadratically the single-grain  
269 overdispersion obtained from a dose recovery experiment on the same sample. This approach yielded  
270 values for overdispersion resulting from beta microdosimetry ranging from  $15.8 \pm 1.8$  to  $27.1 \pm 1.6$   
271  $\%$ . Our results suggest that the application of a standard overdispersion threshold (e.g. 20%), above  
272 which processes such as mixing or partial bleaching may be diagnosed, may not yield accurate results.  
273 Lastly, the possibility of extracting dateable material from heat retainers via mechanical crushing was  
274 explored. A small but significant increase in equivalent dose was observed for a number of quartz  
275 separates from zero-age samples after crushing. However, it was not possible to distinguish the  
276 equivalent dose from crushed and uncrushed fractions of five Holocene samples. These results  
277 suggest that crushing is a viable method for extracting dateable material from a rock, but we note that



278 some knowledge of the dosimeter's mean grain size is required for calculation of an accurate  
279 environmental dose rate.

280

## 281 **7 Acknowledgements**

282 SJA's contribution to this work was partly supported by the Research Council of Norway, through its  
283 Centres of Excellence funding scheme, SFF Centre for Early Sapiens Behaviour (SapienCE), project  
284 number 262618. GEK acknowledges support from Swiss National Science Foundation grant number  
285 PZ00P2-167960.

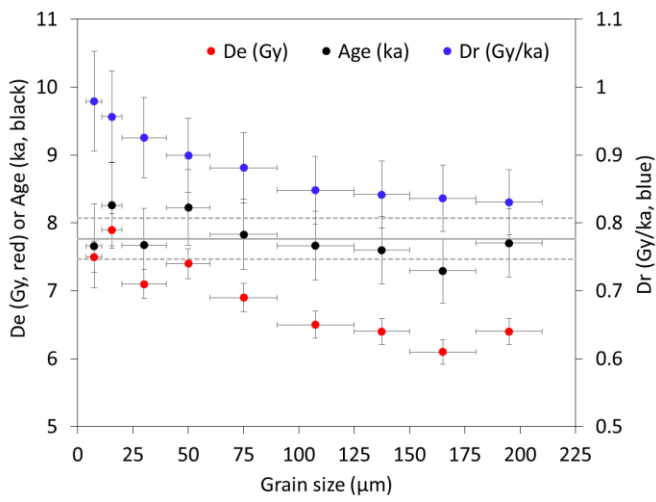
286 **Figures**



287

288 Figure 1: A small hearth at Al Wafa. The hearth mound is ~1 m in diameter.

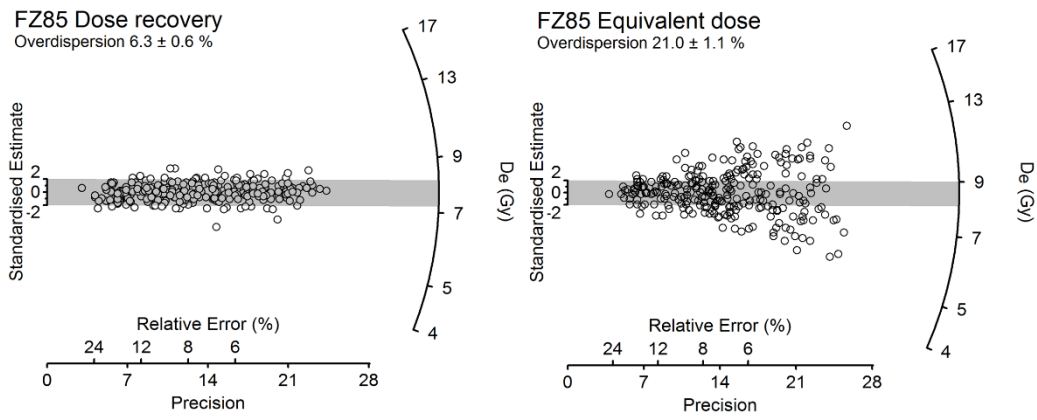
289



290

291 Figure 2: Equivalent dose ( $D_e$ ), environmental dose rate ( $D_r$ ) and calculated age for different grain  
 292 size fractions of quartz from heat retainer sample FZ84. The thick grey line represents the mean  
 293 age, calculated using data from all grain sizes, while the dashed lines represent  $\pm 1$  standard  
 294 deviation.

295



296

297 Figure 3: Radial plots of equivalent doses for individual quartz grains from heat retainer sample  
 298 FZ85. The grey bars are centred at the equivalent dose calculated using the central age model  
 299 (Galbraith et al., 1999). The left radial plot represents data from a dose recovery experiment,  
 300 whereas the right panel shows the natural equivalent dose distribution.

301

302

303

304  
305  
306  
307  
308  
309  
310  
311  
312  
313  
314  
315  
316  
317  
318  
319  
320  
321  
322  
323  
324  
325  
326  
327  
328  
329  
330  
331  
332  
333  
334  
335  
336  
337  
338  
339  
340  
341  
342  
343  
344  
345  
346  
347  
348  
349  
350  
351  
352  
353

## References

Aitken, M.J., 1985. Thermoluminescence dating. Academic press.

Armitage, S.J., Bailey, R.M., 2005. The measured dependence of laboratory beta dose rates on sample grain size. *Radiat Meas* 39, 123-127.

Armitage, S.J., Bristow, C.S., Drake, N.A., 2015. West African monsoon dynamics inferred from abrupt fluctuations of Lake Mega-Chad. *P Natl Acad Sci USA* 112, 8543-8548.

Armitage, S.J., Drake, N.A., Stokes, S., El-Hawat, A., Salem, M., White, K., Turner, P., McLaren, S.J., 2007. Multiple phases of north African humidity recorded in lacustrine sediments from the fazzan basin, Libyan sahara. *Quat Geochronol* 2, 181-186.

Armitage, S.J., Jasim, S.A., Marks, A.E., Parker, A.G., Usik, V.I., Uerpmann, H.-P., 2011. The Southern Route "Out of Africa": Evidence for an Early Expansion of Modern Humans into Arabia. *Science* 331, 453-456.

Armitage, S.J., King, G.E., 2013. Optically stimulated luminescence dating of hearths from the Fazzan Basin, Libya: A tool for determining the timing and pattern of Holocene occupation of the Sahara. *Quat Geochronol* 15, 88-97.

Ballarini, M., Wintle, A.G., Wallinga, J., 2006. Spatial variation of dose rate from beta sources as measured using single grains. *Ancient TL* 24, 1-7.

Bateman, M.D., Swift, D.A., Piotrowski, J.A., Sanderson, D.C.W., 2012. Investigating the effects of glacial shearing of sediment on luminescence. *Quat Geochronol* 10, 230-236.

Bøtter-Jensen, L., Andersen, C.E., Duller, G.A.T., Murray, A.S., 2003. Developments in radiation, stimulation and observation facilities in luminescence measurements. *Radiat Meas* 37, 535-541.

Brennan, B.J., 2003. Beta doses to spherical grains. *Radiat Meas* 37, 299-303.

Brennan, B.J., Lyons, R.G., Phillips, S.W., 1991. Attenuation of alpha particle track dose for spherical grains. *International Journal of Radiation Applications and Instrumentation. Part 18*, 249-253.

Cunningham, A.C., Devries, D.J., Schaart, D.R., 2012. Experimental and computational simulation of beta-dose heterogeneity in sediment. *Radiat Meas* 47, 1060-1067.

Duller, G., 2007. Assessing the error on equivalent dose estimates derived from single aliquot regenerative dose measurements. *Ancient TL* 25, 15-24.

Duller, G.A.T., Bøtter-Jensen, L., Murray, A.S., Truscott, A.J., 1999. Single grain laser luminescence (SGLL) measurements using a novel automated reader. *Nuclear Instruments and Methods in Physics Research, Section B: Beam Interactions with Materials and Atoms* 155, 506-514.

Galbraith, R.F., Roberts, R.G., Laslett, G.M., Yoshida, H., Olley, J.M., 1999. Optical dating of single and multiple grains of quartz from Jinmium rock shelter, northern Australia: Part I, experimental design and statistical models. *Archaeometry* 41, 339-364.

Guérin, G., Jain, M., J. Thomsen, K., S. Murray, A., Mercier, N., 2015. Modelling dose rate to single grains of quartz in well-sorted sand samples: The dispersion arising from the presence of potassium feldspars and implications for single grain OSL dating. *Quat Geochronol* 27, 52-65.

Guérin, G., Mercier, N., 2012. Preliminary insight into dose deposition processes in sedimentary media on a scale of single grains: Monte Carlo modelling of the effect of water on the gamma dose rate. *Radiat Meas* 47, 541-547.

Martin, L., Mercier, N., Incerti, S., Lefrais, Y., Pecheyran, C., Guérin, G., Jarry, M., Bruxelles, L., Bon, F., Pallier, C., 2015. Dosimetric study of sediments at the beta dose rate scale: Characterization and modelization with the DosiVox software. *Radiat Meas* 81, 134-141.

Mayya, Y.S., Morthekai, P., Murari, M.K., Singhvi, A.K., 2006. Towards quantifying beta microdosimetric effects in single-grain quartz dose distribution. *Radiat Meas* 41, 1032-1039.

Murray, A.S., Wintle, A.G., 2000. Luminescence dating of quartz using an improved single-aliquot regenerative-dose protocol. *Radiat Meas* 32, 57-73.

354 Rhodes, E.J., Fanning, P.C., Holdaway, S.J., 2010. Developments in optically stimulated  
355 luminescence age control for geoarchaeological sediments and hearths in western New South  
356 Wales, Australia. *Quat Geochronol* 5, 348-352.

357 Rhodes, E.J., Fanning, P.C., Holdaway, S.J., Bolton, C.C., 2009. Archaeological surfaces in western  
358 NSW: stratigraphic contexts and preliminary OSL dating of hearths. *Terra Australis* 28, 12.

359 Swift, D.A., Sanderson, D.C.W., Nienow, P.W., Bingham, R.G., Cochrane, I.C., 2011. Anomalous  
360 luminescence of subglacial sediment at Haut Glacier d'Arolla, Switzerland - a consequence of  
361 resetting at the glacier bed? *Boreas* 40, 446-458.

362 Thomsen, K.J., Murray, A.S., Bøtter-Jensen, L., 2005. Sources of variability in OSL dose  
363 measurements using single grains of quartz. *Radiat Meas* 39, 47-61.

364 Thomsen, K.J., Murray, A.S., Buylaert, J.P., Jain, M., Hansen, J.H., Aubry, T., 2016. Testing  
365 single-grain quartz OSL methods using sediment samples with independent age control from the  
366 Bordes-Fitte rockshelter (Roches d'Abilly site, Central France). *Quat Geochronol* 31, 77-96.

367