Supplementary material for "Optically stimulated luminescence dating of heat retainer hearths from the Sahara: Insights into signal accumulation and measurement." Armitage et al.

A. Sample preparation procedure

Having removed the light exposed layers of the heat retainer, dating samples were prepared using standard laboratory techniques. Briefly, carbonate was removed by immersion in 1.16 M HCl, followed by immersion in H_2O_2 to oxidise organic material. Samples FZ84, 85, 95 and 96 were sieved at 40 µm, and the material which passed through the mesh was subsequently Stokes settled to 4-11, 11-20 and 20-40 µm. Material retained in the mesh was sieved to yield 40-60, 60-90, 90-125, 125-150, 150-180 and 180-210 size fractions. Quartz was extracted from the <90 µm fractions via prolonged immersion in H_2SiF_6. Quartz was extracted from the >90 µm fractions via density separation at 2.62 and 2.75 g/cm^3, and 1 hour immersion in 23 M HF. Following H_2SiF_6 or HF treatment, samples were immersed 11 M HCl for a minimum of 12 hours to remove fluoride precipitates. The <60 µm fractions were settled from suspension onto 10 mm diameter aluminium discs, resulting in a monolayer of quartz across the entire upper face of the disc. The >60 µm fractions were mounted on aluminium discs using Silkospray silicone oil applied via a 5 mm mask, resulting in a monolayer of grains across the middle 5 mm of the disc. Single-grain analysis was performed on 180-210 µm grains mounted in Risø single-grain sample holders, containing a 10x10 array of 300 µm diameter holes. Sample preparation and mounting methods are summarised in Table S1.

Table S1. Summary sample preparation and mounting methods for the various grain size fractions reported in this study.

Grain size (µm)	Size separation method	Quartz purification method	Sample mounting method
4-11	Stokes settling	H_2SiF_6 + HCl	Settled from suspension
11-20	Stokes settling	H_2SiF_6 + HCl	Settled from suspension
20-40	Stokes settling	H_2SiF_6 + HCl	Settled from suspension
40-60	Sieve	H_2SiF_6 + HCl	Silkospray silicone oil via a 5 mm mask
60-90	Sieve	H_2SiF_6 + HCl	Silkospray silicone oil via a 5 mm mask
90-125	Sieve	Density separation + HF	Silkospray silicone oil via a 5 mm mask
125-150	Sieve	Density separation + HF	Silkospray silicone oil via a 5 mm mask
150-180	Sieve	Density separation + HF	Silkospray silicone oil via a 5 mm mask
180-210	Sieve	Density separation + HF	Silkospray silicone oil via a 5 mm mask

B. Single-grain rejection criteria.

Grains were rejected where one or more of the following conditions are met: (1) the T_n signal from the grain is too low to distinguish it from the variability in the background signal (the difference between T_n and the average count from the background interval is less than three times the standard deviation of the counts in the background interval, determined using the "sig. >3 sigma above BG" rejection criterion in Luminescence Analyst, (Duller, 2007)); (2) the recycling ratio (Murray and Wintle, 2000) differs from unity by greater than 10%; (3) the OSL IR-depletion ratio (Duller, 2003) is greater than two standard errors below unity; (4) recuperation is high (i.e. L_x/T_x for the 0 Gy regeneration point is greater than 5% of L_n/T_n,(Murray and Wintle, 2000)); (5) L_n/T_n does not intercept the growth curve (Armitage et al., 2000; Bailey et al., 2005) – no grains failed this criterion due to the low D_e of the samples, or (6) the dose response curve shape precludes the generation of a meaningful equivalent dose. Unlike the other five criteria, criterion six requires user-judgement and hence cannot be applied mechanistically. It was used where the signal does not increase monotonically in response to increasing dose, or where L_n/T_n intercepted the dose-response curve at the point where growth ceased, with the former being more common.

C. Equivalent dose, dose rate and calculated age for different size fractions from Al Wafa heat retainers.

Equivalent dose (D_e), dose rate and calculated age data for different size fractions from Al Wafa heat retainers are presented in Tables S2-S5. In these tables, "Mean 4-210" denotes the mean age determined for grain size fraction in the range 4-210 µm. The "effective grain size" is calculated as the grain size with a dose rate which yields the mean 4-210 µm age for the 4-11 µm quartz produced by crushing the bulk sample (Section 5). Dose rates and ages are calculated using a mean water content of 5±2 %, a burial depth of 30 cm (yielding a cosmic dose rate of 0.235±0.020 Gy/ka) and partitioning the gamma dose between the heat retainer and underlying sediments (Armitage and King, 2013). For alpha dose rates, an a-value of 0.04 ± 0.02 was used throughout. Instrumental beta dose rates were corrected for grain size using the values measured by Armitage and Bailey (2005, Table 1).

Table S2: Heat retainer FZ84

		Dose rates (Gy/ka)				
Grain size (µm)	De (Gy)	Alpha	Beta	Gamma	Total	Age (ka)
4-11	7.5 ± 0.2	0.10 ± 0.05	0.41 ± 0.04	0.23 ± 0.02	0.98 ± 0.07	7.7 ± 0.6
11-20	7.9 ± 0.2	0.08 ± 0.04	0.41 ± 0.04	0.23 ± 0.02	0.96 ± 0.07	8.3 ± 0.6
20-40	7.1 ± 0.2	0.06 ± 0.03	0.40 ± 0.04	0.23 ± 0.02	0.93 ± 0.06	7.7 ± 0.5
40-60	7.4 ± 0.2	0.04 ± 0.02	0.40 ± 0.04	0.23 ± 0.02	0.90 ± 0.05	8.2 ± 0.6
60-90	6.9 ± 0.2	0.03 ± 0.01	0.39 ± 0.04	0.23 ± 0.02	0.88 ± 0.05	7.8 ± 0.5
90-125	6.5 ± 0.2	-	0.39 ± 0.04	0.23 ± 0.02	0.85 ± 0.05	7.7 ± 0.5
125-150	6.4 ± 0.2	-	0.38 ± 0.04	0.23 ± 0.02	0.84 ± 0.05	7.6 ± 0.5
150-180	6.1 ± 0.2	-	0.37 ± 0.04	0.23 ± 0.02	0.84 ± 0.05	7.3 ± 0.5
180-210	6.4 ± 0.2	-	0.37 ± 0.04	0.23 ± 0.02	0.83 ± 0.05	7.7 ± 0.5
Mean 4-210	-	-	-	-	-	**7.8 ± 0.5**
			Effective grain size 40-60 µm			
4-11 (crushed)	**7.0 ± 0.2**				**0.90 ± 0.05**	**7.8 ± 0.5**

Table S3: Heat retainer FZ85

		Dose rates (Gy/ka)				
Grain size (µm)	De (Gy)	Alpha	Beta	Gamma	Total	Age (ka)
4-11	10.7 ± 0.3	0.10 ± 0.05	0.37 ± 0.04	0.22 ± 0.02	0.93 ± 0.07	11.5 ± 0.9
11-20	10.8 ± 0.3	0.09 ± 0.04	0.36 ± 0.04	0.22 ± 0.02	0.91 ± 0.07	11.9 ± 0.9
20-40	10.1 ± 0.3	0.06 ± 0.03	0.36 ± 0.04	0.22 ± 0.02	0.88 ± 0.06	11.5 ± 0.8
40-60	9.6 ± 0.3	0.04 ± 0.02	0.35 ± 0.04	0.22 ± 0.02	0.85 ± 0.05	11.3 ± 0.8
60-90	9.4 ± 0.3	0.03 ± 0.01	0.35 ± 0.04	0.22 ± 0.02	0.84 ± 0.05	11.3 ± 0.7
90-125	8.6 ± 0.3	-	0.34 ± 0.04	0.22 ± 0.02	0.80 ± 0.05	10.7 ± 0.7
125-150	8.6 ± 0.3	-	0.34 ± 0.04	0.22 ± 0.02	0.80 ± 0.05	10.8 ± 0.7
150-180	8.3 ± 0.3	-	0.33 ± 0.04	0.22 ± 0.02	0.79 ± 0.05	10.5 ± 0.7
180-210	8.9 ± 0.3	-	0.33 ± 0.04	0.22 ± 0.02	0.79 ± 0.04	11.3 ± 0.7
Mean 4-210	-	-	-	-	-	**11.2 ± 0.8**
			Effective grain size 40-60 µm			
4-11 (crushed)	**9.5 ± 0.3**				**0.85 ± 0.05**	**11.2 ± 0.8**

Table S4: Heat retainer FZ95

Grain size (µm)	De (Gy)	Dose rates (Gy/ka)				Age (ka)
		Alpha	Beta	Gamma	Total	
4-11	5.8 ± 0.2	0.07 ± 0.04	0.32 ± 0.03	0.22 ± 0.02	0.85 ± 0.06	6.9 ± 0.5
11-20	9.4 ± 0.3	0.06 ± 0.03	0.32 ± 0.03	0.22 ± 0.02	0.83 ± 0.05	11.3 ± 0.8*
20-40	4.1 ± 0.1	0.04 ± 0.02	0.31 ± 0.03	0.22 ± 0.02	0.81 ± 0.05	5.1 ± 0.3
40-60	4.8 ± 0.1	0.03 ± 0.01	0.31 ± 0.03	0.22 ± 0.02	0.79 ± 0.04	6.1 ± 0.4
60-90	4.8 ± 0.1	0.02 ± 0.01	0.31 ± 0.03	0.22 ± 0.02	0.77 ± 0.04	6.2 ± 0.4
90-125	4.3 ± 0.1	-	0.30 ± 0.03	0.22 ± 0.02	0.75 ± 0.04	5.7 ± 0.4
125-150	4.6 ± 0.1	-	0.29 ± 0.03	0.22 ± 0.02	0.75 ± 0.04	6.2 ± 0.4
150-180	6.2 ± 0.2	-	0.29 ± 0.03	0.22 ± 0.02	0.74 ± 0.04	8.4 ± 0.5
180-210	4.4 ± 0.1	-	0.29 ± 0.03	0.22 ± 0.02	0.74 ± 0.04	6.0 ± 0.4
Mean 4-210	-	-	-	-	-	**6.3 ± 1.0**
Effective grain size 90-150 µm						
4-11 (crushed)	**4.7 ± 0.1**				**0.75 ± 0.04**	**6.3 ± 0.4**

*The age for 11-20 µm grain size has been omitted from the calculation of the mean age.

Table S5: Heat retainer FZ96

Grain size (µm)	De (Gy)	Dose rates (Gy/ka)				Age (ka)
		Alpha	Beta	Gamma	Total	
4-11	7.9 ± 0.2	0.07 ± 0.04	0.53 ± 0.06	0.24 ± 0.02	1.09 ± 0.07	7.3 ± 0.5
11-20	8.4 ± 0.2	0.06 ± 0.03	0.53 ± 0.06	0.24 ± 0.02	1.07 ± 0.07	7.9 ± 0.6
20-40	7.4 ± 0.2	0.04 ± 0.02	0.52 ± 0.06	0.24 ± 0.02	1.04 ± 0.07	7.1 ± 0.5
40-60	6.9 ± 0.2	0.03 ± 0.02	0.51 ± 0.06	0.24 ± 0.02	1.02 ± 0.06	6.8 ± 0.5
60-90	6.3 ± 0.2	0.02 ± 0.01	0.51 ± 0.05	0.24 ± 0.02	1.00 ± 0.06	6.3 ± 0.4
90-125	6.7 ± 0.2	-	0.50 ± 0.05	0.24 ± 0.02	0.98 ± 0.06	6.9 ± 0.5
125-150	6.2 ± 0.2	-	0.49 ± 0.05	0.24 ± 0.02	0.97 ± 0.06	6.4 ± 0.4
150-180	5.9 ± 0.2	-	0.48 ± 0.05	0.24 ± 0.02	0.96 ± 0.06	6.1 ± 0.4
180-210	5.9 ± 0.2	-	0.47 ± 0.05	0.24 ± 0.02	0.95 ± 0.06	6.2 ± 0.4
Mean 4-210	-	-	-	-	-	**6.8 ± 0.6**
Effective grain size >210 µm						
4-11 (crushed)	**5.9 ± 0.2**				**0.87 ± 0.06**	**6.8 ± 0.5**

D. Single grain overdispersion values

Table S6: Single-grain equivalent dose, dose recovery and overdispersion data.

Sample	Equivalent dose data			Dose recovery data			OD_{β} (%)
	D_e (Gy)	Grains (accepted/ measured)	OD_{De} (%)	Ratio (recovered/known)	Grains (accepted/ measured)	OD_{Rec} (%)	
FZ40	9.2 ± 0.3	131/1000	18.5 ± 1.5	0.98 ± 0.02	135/1200	9.7 ± 1.1	15.8 ± 1.8
FZ47	7.3 ± 0.3	89/1200	24.9 ± 2.3	0.99 ± 0.01	164/1200	7.7 ± 0.7	23.7 ± 2.4
FZ84	6.0 ± 0.1	171/1200	26.5 ± 1.6	1.00 ± 0.01	242/1200	7.5 ± 0.7	25.4 ± 1.8
FZ85	8.6 ± 0.1	262/1200	21.0 ± 1.1	0.96 ± 0.01	276/1200	6.3 ± 0.6	20.0 ± 1.2
FZ95	4.5 ± 0.1	242/1100	28.2 ± 1.5	0.98 ± 0.01	281/1200	7.9 ± 0.7	27.1 ± 1.6
FZ96	6.0 ± 0.1	176/1200	24.5 ± 1.5	0.99 ± 0.01	195/1200	6.3 ± 0.8	23.7 ± 1.7

E. Equivalent doses for uncrushed and crushed quartz separates

Table S7: Sample description and equivalent doses for uncrushed and crushed fractions of zero-age quartzes.

Sample	Provenance	Equivalent dose (Gy)		Increase (Gy)
		Uncrushed	Crushed	(crushed-uncrushed)
BA5	Coastal dune (Armitage et al., 2006)	0.04 ± 0.01	0.23 ± 0.02	0.19 ± 0.02
CH62	Shoreline (Armitage et al., 2015)	0.01 ± 0.01	0.11 ± 0.02	0.10 ± 0.02
FZ16	Desert dune (Armitage et al., 2007)	0.04 ± 0.03	0.12 ± 0.04	0.08 ± 0.05
FZ32	Desert dune (Armitage and Bailey, 2005)	0.08 ± 0.08	0.16 ± 0.08	0.08 ± 0.12
IN22	Coastal dune (Armitage et al., 2006)	0.01 ± 0.02	0.02 ± 0.01	0.01 ± 0.02
Risø quartz	Calibration sample (Hansen et al., 2015)	0.00 ± 0.00	0.09 ± 0.01	0.09 ± 0.01
SA10	Coastal dune (this study)	0.00 ± 0.00	0.93 ± 0.33	0.93 ± 0.33
			Mean	**0.21 ± 0.12**

Table S8: Sample description and equivalent doses for uncrushed and crushed fractions of Holocene quartzes.

Sample	Provenance	Equivalent dose (Gy)		Ratio
		Uncrushed	Crushed	(uncrushed/crushed)
CH22	Shoreline (Armitage et al., 2015)	3.72 ± 0.13	4.07 ± 0.19	1.09 ± 0.06
IN15	Coastal dune (Armitage et al., 2006)	5.35 ± 0.07	5.31 ± 0.06	0.99 ± 0.02
IN16	Coastal dune (Armitage et al., 2006)	2.34 ± 0.11	2.53 ± 0.12	1.08 ± 0.07
NG38	Shoreline (Armitage et al., 2015)	19.5 ± 0.2	20.3 ± 1.2	1.04 ± 0.06
NG39	Shoreline (Armitage et al., 2015)	19.1 ± 0.8	19.4 ± 0.5	1.02 ± 0.05
			Mean	**1.04 ± 0.02**

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