**Supplementary Information, File 5**

OSL dating

**S5-1. Background**

Optically stimulated luminescence (OSL) is an absolute dating technique used to estimate the amount of time elapsed since mineral grains were last exposed to sunlight, or sufficient heat (Huntley et al., 1985; Aitken, 1998; Duller, 2004; Wintle, 2014; Roberts et al., 2015). Due to its ability to utilize grains of quartz (a ubiquitous mineral, readily available in archaeological deposits), and date to events beyond the span of radiocarbon dating, OSL dating has played a pivotal role in dating the Middle Stone Age (MSA) of South Africa (e.g., Jacobs et al., 2008a; Tribolo et al., 2016; Henshilwood et al., 2014). This method is based on the observation that once mineral grains are no longer exposed to light or elevated temperatures, there is a time-dependent increase in the amount of electrons which become trapped at defects in their crystal lattices. This absorption of energy and storage of trapped charge is due to the exposure of buried grains to an ionizing radiation flux from surrounding sediment, and cosmic rays. If a grain becomes exposed to light (e.g., through bioturbation) or sufficient heat (e.g., burning in immediate proximity) in antiquity, electrons will be released from their traps, and the OSL ‘clock’ is reset to zero. Similarly, if a grain is exposed to light in the laboratory (via optical stimulation), trapped electrons will be released, and a proportion will dissipate energy as photons (luminescence). Thus, the amount of luminescence emitted is proportional to the number of electrons trapped in the lattice. The ‘equivalent dose’ (De) refers to the laboratory estimate of the total radiation dose to which a mineral grain has been exposed since its last ‘resetting’ event, and is calculated by exposing grains to an intense light source, and measuring the amount of OSL produced (**Section S5-3**). The environmental dose rate refers to the rate at which the mineral grains were exposed to ionising radiation energy while buried, and is calculated using different methods (**Section S5-4**). In its simplest form, OSL ages are determined by dividing De by the dose rate, and are calculated directly in calendar years before present, so there is no need for subsequent calibration. The datum used for calculating the ages reported here is 2018.

**S5-2. Sample collection and preparation**

Five sediment samples were collected for OSL dating from square N850E321NW at Waterfall Bluff during the 2016 excavations. Plastic tubes were horizontally driven into cleaned surfaces of the eastern profile, from LBCS Sub-Aggregates Colton (CN312), the base of Kate (CN308), Otis (CN309), Kuka (CN310), and the lowermost SRCS Sub-Aggregate, Courtney (CN311). Additional sediment subsamples were collected from the hole of each OSL sample for laboratory measurements of the present-day moisture content of the sediment.

All samples were transported to the OSL Dating Laboratory at the University of Wollongong in Australia, and were opened and prepared under subdued red-light conditions following standard procedures (Wintle, 1997; Aitken, 1998). The first ~2 cm from each tube end was treated as potentially light-exposed, and carefully removed from the main sample. These ‘tube ends’ were utilized for laboratory measurements of the radioactivity present in the sediment. The remaining (light-safe) sample was prepared for quartz extraction. First, samples were wet-sieved to collect the 90–300 µm-diameter grain size fraction, from which carbonates were dissolved in 40% hydrochloric (HCI) acid, and organic matter oxidised in 32% hydrogen peroxide (H2O2) solution. Samples were wet-sieved again to isolate the 150–180 and 180–212 µm–diameter grain size fractions. Quartz, feldspar and heavy minerals were separated by density separation using sodium polytungstate (SPT) solutions diluted to densities of 2.62 g/cm3 and 2.7 g/cm3, respectively. Separated quartz grains were etched with 48% hydrofluoric (HF) acid for 40 min to remove the alpha-irradiated rinds and destroy any remaining feldspar. Samples were then rinsed in HCI acid to remove any precipitated fluorides, and dried and sieved again. Grains remaining on the 150 and 180 µm-diameter meshes were used for De determination.

**S5-3. De determination and results**

OSL measurements were undertaken using two different automated Risø TL/OSL readers fitted with single-grain laser attachments (Risø 4 = DA-20, Risø 6 = DA-20 C/D). Samples were irradiated using calibrated 90Sr/90Y beta sources. Optical stimulation was achieved using an intense, green (532 nm) light from a 10 nW Nd:YVO4 solid-state diode-pumped laser (maximum power ~50 W cm-2), focussed onto a ~20 µm-diameter spot, at 90% power (Bøtter-Jensen et al., 2000). The resulting ultraviolet OSL emissions were then detected by an Electron Tubes Ltd. 9235QA (Risø 4) or 9107Q-AP-TTL-03 (Risø 6) photomultiplier tube, fitted with 7.5 mm thick Hoya U–340 filters.

Single-grain OSL measurements were undertaken on 180–212 µm-diameter quartz grains for all five samples. Grains were measured using standard single-grain aluminium discs drilled with 100 individual 300 x 300 μm holes, in which grains were individually placed (Bøtter-Jensen et al., 2000). Additional single-grain measurements on 150–180 µm-diameter quartz grains were required for samples CN312, CN308 and CN309, due to the luminescence sensitivity (brightness) of these quartz samples being very low. For these measurements, a 'pseudo' single-grain approach was taken to increase probability that a grain able to produce a statistically-significant OSL signal would be measured. It is estimated that ~2–3 grains were placed into each hole using this approach. The initial OSL measurements of the 180–212 µm-diameter grains for these samples showed that only ~13% of individual grains produce a statistically distinguishable OSL Tn signal. Demuro et al. (2013) have demonstrated that such an approach for De measurement is unlikely to produce any significant grain-hole averaging effects for samples characterised by low proportions (<30%) of luminescent grains. For such dim samples, this approach is useful in boosting the statistical significance of De determination, yet maintaining single-grain resolution.

The single-aliquot regenerative-dose (SAR) procedure (Galbraith et al., 1999; Murray & Wintle, 2000) used for measuring De in this study is outlined in **Table S5-1**. All luminescence data was processed and analysed using the R packages ‘numOSL’ (Peng & Li, 2017) and ‘Luminescence’ (Kreutzer et al., 2012, 2017). Single grains were optically stimulated by green (532 nm) light for 2 s at 125°C, and the net OSL signal was calculated using the first 0.2 s, after subtracting an estimated (‘late’) background count rate from the last 0.3 s of stimulation (**Figure S5-1a**). Sensitivity-corrected OSL signals (LX/TX) were determined, and dose-response curves (DRCs) fitted for each grain (**Figure S5-1b**). All DRCs were fitted using a general-order kinetics (GOK) function (Guralnik et al., 2015), which has been demonstrated to be effective for fitting a wide range of DRCs (Peng & Li, 2017). De values were estimated by interpolating the sensitivity-corrected natural signals (LN/TN) onto their corresponding DRCs. The uncertainty on this estimate for each grain was determined from photon counting statistics, instrument irreproducibility estimates (1.3% or 2.5%, depending on which Risø reader was used), and curve fitting uncertainties, determined by Monte Carlo simulation (Duller, 2007).

Dose recovery experiments (Roberts et al., 1998, 1999; Galbraith et al., 1999) were conducted on bleached 180–212 and 150–180 µm-diameter quartz grains from A2SE-1-308 to determine the optimum preheat (PH) temperatures for the Waterfall Bluff single-grain OSL SAR procedure. The most accurate De results were obtained using 180°C for 10 s (PH1) and 180°C for 5 s (PH2), which gave a measured/given dose ratio consistent with unity (0.98 ± 0.03) (**Figure S5-2**).

A total of 10,900 single grain measurements were undertaken for De determination in this study. Aberrant grains were excluded from final De calculations using well-established rejection criteria (e.g., Jacobs et al., 2006a) (**Table S5-2**). The vast majority of grains (84%) were rejected on the basis of signal brightness, either because their test dose signals (TN) were not statistically distinguishable following a laboratory dose of ~9 Gy (Criteria 1, 73%), or TN signals were imprecisely known (Criteria 2, 11%). The 180–212 µm-diameter quartz grains measured for each sample show a clear trend – grain signal brightness significantly decreases from the uppermost Sub-Agg that was OSL dated (SRCS Courtney) to the lowermost (LBCS Colton). This is likely related to the intensity of the amount of burning present in the respective Sub-Aggs. Exposure of quartz to high temperatures is associated with an increase in OSL sensitivity, which results in an increase in the amount of luminescence generated in response to one unit of dose (Chen et al., 2001; Pagonis et al., 2010). This has been previously observed in association with intensive burning at, for example, Blombos Cave (Jacobs et al., 2006b), Diepkloof (Jacobs et al., 2008a), Sibudu (Jacobs et al., 2008b) and Pinnacle Point 13B (Jacobs, 2010).

De values of accepted grains from each sample are displayed as radial plots (**Figure S5-3**). Overdispersion (OD) values refer to the relative spread in De remaining after measurement uncertainties are accounted for (Galbraith et al., 2005). Using the central age model (CAM) (Galbraith et al. 1999), individual De values were combined to give a weighted mean De for each sample. The 3-parameter minimum age model (MAM) (Galbraith et al. 1999) was used in addition for samples CN310, CN309, CN308 and CN312 to calculate the minimum De value for each sample. Before running the MAM we added (in quadrature) an additional OD value of 15% to the measurement error of individual De values. An OD value of 15% was chosen based on the OD value obtained during dose recovery experiments on CN308 (**Fig. S5-2**). Due to the sensitivity of the MAM to low-dose outliers, two intrusive younger grains (both with De values <20 Gy) were excluded during the calculation of this statistical model for sample CN312.

The number of usable grains obtained in the initial analyses of the 180–212 µm-diameter quartz grains from samples CN311 and CN310 is deemed appropriate for final De estimation. Those obtained for samples CN309, CN308 and CN312 were considered insufficient for reliable De calculation. The CAM, MAM and OSL age results we present in the main textcombines De values obtained from both 180–212 and 150-180 µm-diameter quartz grains for these samples. Comparison of the weighted mean De values obtained for each of these grain size fractions from each sample is provided in **Table S5-3**.

**S5-4. Dose rate determination and results**

The manner dosimetry has been measured in this study assumes consistent conditions over the burial period. An internal alpha dose rate of 0.033 ± 0.008 Gy/ka has been assumed for all samples, based on previous measurements of HF-etched South African quartz (Jacobs, 2004). Beta and gamma dose rates were estimated using two independent methods: a) a combination of GM-25-5 beta counting (GMBC) (Bøtter-Jensen & Mejdahl, 1988) and thick-source alpha counting (TSAC), and 2) inductively-coupled plasma optical emission spectrometry (ICP-OES) for determination of K and ICP-mass spectrometry (MS) for determination of U and Th. GMBC and TSAC were carried out on the same samples at the OSL Dating Laboratory at the University of Wollongong, and ICP-OES/MS at Genalysis Intertek Laboratory in Perth, Australia. Comparisons between the measured elemental concentrations (K, Th and U) obtained using both approaches are provided in **Table S5-4**.

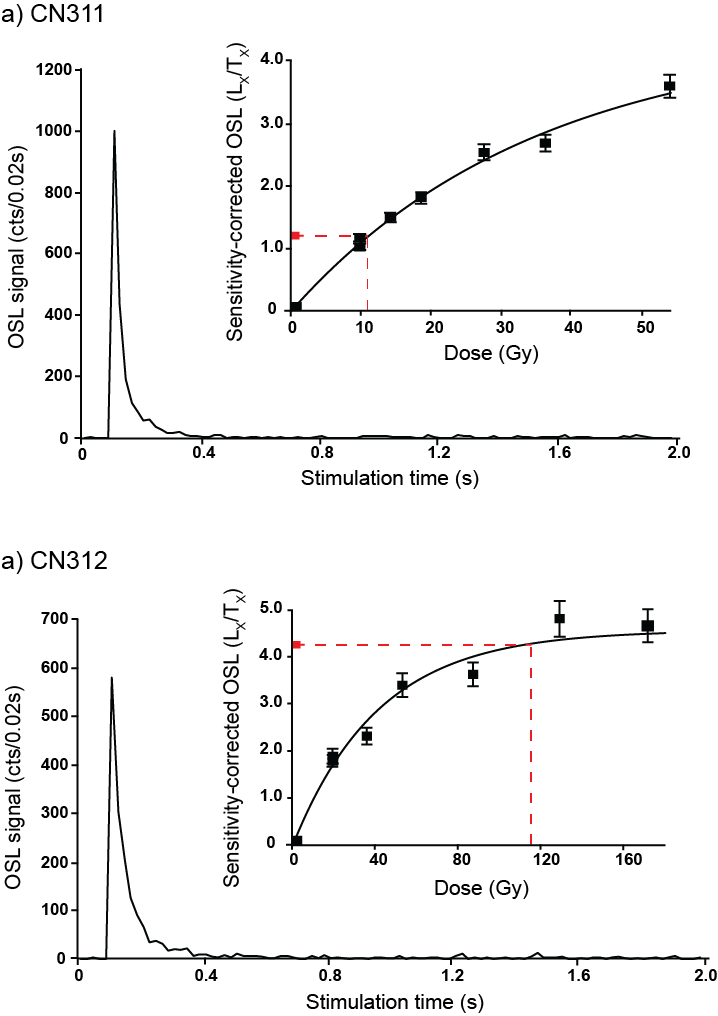
Beta and gamma dose rate values for the chronology presented in the main textare derived from the ICP-MS/OES results. Allowance was made for the effect of moisture content (Aitken, 1985; Nathan & Manz, 2008), grain size (Mejdahl, 1979; Brennan, 2003) and HF etching (Bell & Zimmerman, 1978) on beta-dose attenuation for all samples. Laboratory-derived gamma dose rates are not ideal, because they assume that the subsample collected and measured is representative of the ~30 cm gamma sphere surrounding the OSL sample, in its original location. The use of an *in situ* gamma spectrometer, unavailable at time of sampling, would have been preferable. The best we can do at present is model the gamma dose rates for each sample by taking into account known heterogeneities within the ~30 cm diameter sphere around sample location. To do this, we used the K, Th and U concentrations and field water content measured from each dosimetry sample to characterise the wet fractional gamma contributions from each respective SubAgg, given the location of each OSL sample. Because LBCS Lily was not sampled for OSL dating, we used average elemental concentrations and water content values derived from LBCS Kuka above, and LBCS Otis below. No OSL samples were taken above SRCS Courtney, or below LBCS Colton. Due to this, we could only project the known elemental concentrations and water content of SRCS Courtney for those SubAggs above, and those of Colton, below. Elemental concentrations were converted into gamma dose rates (Gy/ka) using the dose rate conversion factors of Guérin et al. (2011), and corrected for sample moisture content (Nathan & Mauz, 2008). These modelled gamma dose rates are used in the Waterfall Bluffchronology and are presented in **Table 3**.

Cosmic-ray dose rates were also taken into account, which was adjusted for the site altitude, geomagnetic latitude, the thickness of sediment and rock overburden (Prescott & Hutton, 1994), and the cos2φ-zenith angular dependence of cosmic rays (Smith et al., 1997). We used the 3D GIS developed for Waterfall Bluff as an aid to obtain accurate estimates of the parameters input into the equations for estimating cosmic-ray dose rates (Fisher et al., 2015).

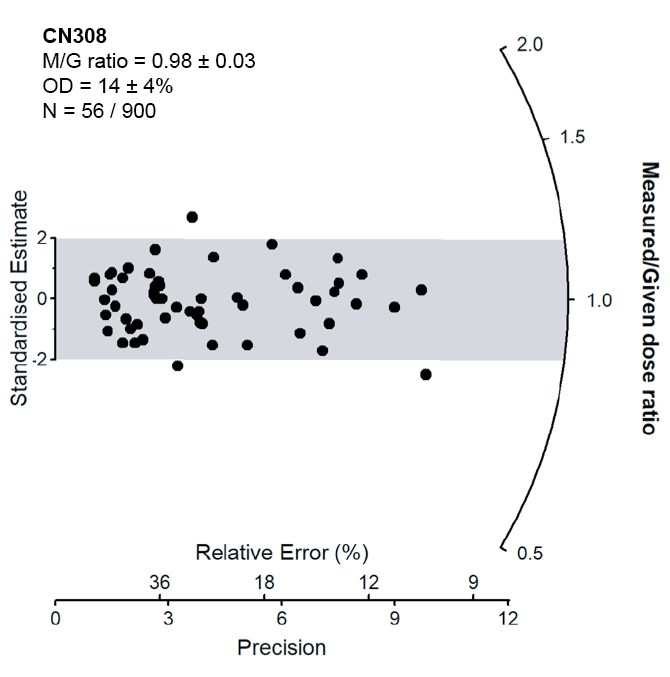
The sensitivity of beta dose rate, gamma dose rate and final OSL age to a) the method used for beta and gamma dose rate estimation, and b) whether or not gamma sphere modelling was employed is insignificant and presented in **Table S5-5** and **S5-6**. The quadratic sum of all known and estimated sources of systematic and random error are represented as the uncertainty attached to each total dose rate. These include a relative uncertainty of 25% (at 1σ) in sample moisture content estimates (made using measured field values) to accommodate likely variations over the burial period.

|  |  |  |
| --- | --- | --- |
| **Step** | **Treatment** | **Signal** |
| 1 | Dose (natural or laboratory) |  |
| 2 | Preheat 1 (180ºC for 10 s) |  |
| 3 | Single-grain OSL stimulation (125ºC for 2 s) | LN or LX |
| 4 | Test dose (~9 Gy) |  |
| 5 | Preheat 2 (180ºC for 5 s) |  |
| 6 | Single-grain OSL stimulation (125ºC for 2 s) | TN or TX |
| 7 | Repeat measurement cycle for different |  |
|  | sized regenerative doses |  |
|  |  |  |

**Table S5-1. Single-grain SAR OSL procedure used for the Waterfall Bluff samples.** For each sample analysed, this measurement cycle was repeated for the natural dose, 4–6 different regenerative doses, a 0-Gy dose to monitor recuperation, as well as a duplicate regenerative dose (equivalent to the first regenerative dose after the natural dose measurement) to check the adequacy of the test-dose sensitivity correction. At the end of each SAR sequence, a check was undertaken on possible contamination of feldspar using the OSL IR depletion-ratio test (Duller, 2003).



**Figure S5-1.** Representative natural signal decay curves (a) and representative dose response curves for one grain from sample CN311, and another from CN312.

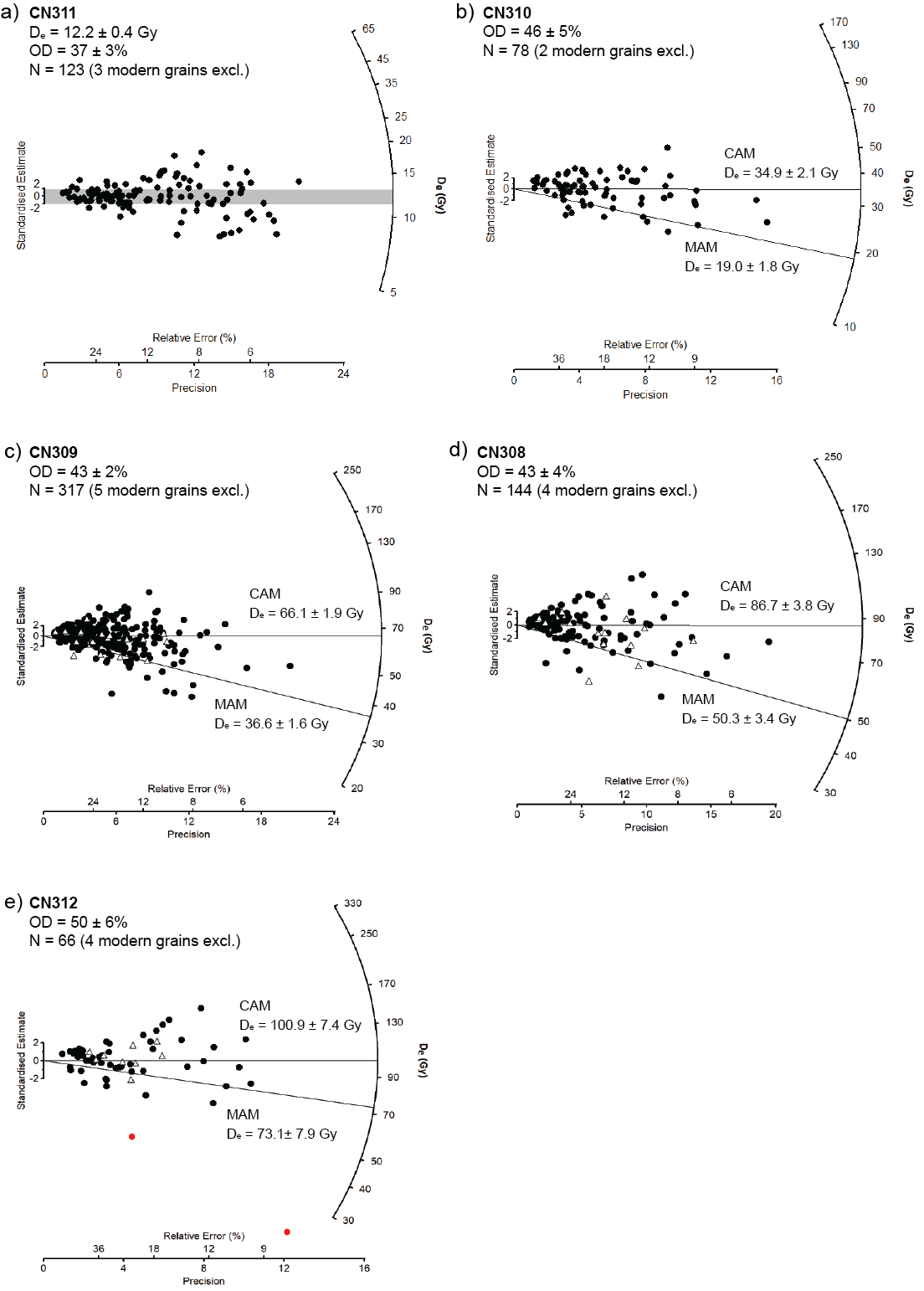


**Figure S5-2.** **Single-grain OSL dose recovery tests undertaken on 150-180 µm-diameter quartz grains from CN308 using the measurement procedure outlined in** **Table S1.** A surrogate natural dose of ~95 Gy was administered. The individual measured/given dose ratios are displayed as a radial plot. Weighted mean ratio and overdispersion (OD) value were calculated using the central age model (CAM) (Galbraith et al., 1999). The grey bar is centred on the CAM weighted mean ratio.

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **OSL sample code (CN** | **No. of measured grains** | **1. TN signal <3σ BG** | **2. TN error >25%** | **3. Recuperation ratio >5%** | **4. Poor recycling ratio** | **5. IR depleted** | **6. FOM exceeds 10%** | **7. De calculation by extrapolation** | **8. Saturated grains** | **9. Modern grains** | **Sum of rejected grains** | **Accepted individual De values** | **% of grains used for final De calc.** |
| 311 | 1000 | 580 | 123 | 9 | 40 | 35 | 86 | 0 | 1 | 3 | **877** | **123** | **12.3** |
| 310 | 1000 | 689 | 111 | 3 | 28 | 17 | 63 | 4 | 5 | 2 | **922** | **78** | **7.8** |
| 309 | 1000 | 871 | 59 | 1 | 15 | 11 | 17 | 1 | 2 | 4 | **981** | **19** | **1.9** |
| 309**\*** | 2000 | 1185 | 196 | 8 | 77 | 61 | 107 | 37 | 30 | 1 | **1702** | **298** | **14.9** |
| 308 | 1000 | 895 | 49 | 2 | 7 | 7 | 13 | 6 | 2 | 4 | **985** | **15** | **1.5** |
| 308**\*** | 2000 | 1400 | 290 | 3 | 36 | 21 | 69 | 23 | 29 | 0 | **1871** | **129** | **6.5** |
| 312 | 1000 | 882 | 74 | 1 | 5 | 7 | 16 | 1 | 3 | 2 | **991** | **9** | **0.9** |
| 312**\*** | 1900 | 1468 | 247 | 6 | 25 | 27 | 40 | 13 | 15 | 2 | **1843** | **57** | **3.0** |
| ***Total*** | *10,900* | *7970* | *1149* | *33* | *233* | *186* | *411* | *85* | *87* | *18* | ***10,172*** | ***728*** | ***6.7*** |

**Table S5-2. Number of single-grain measurements rejected, measured and accepted for final De.** Analysis was undertaken on 1000 individual 180-212 µm-diameter quartz grains for each sample. Samples which had additional analyses undertaken on their 150-180 µm-diameter quartz grain fraction are marked with (**\***). Grains were rejected from final De estimations if they failed one or more of the following criteria.

1. Initial TN signal <3σ above corresponding background count.
2. Relative standard error on TN >25%.
3. Recuperation ratio >5%.
4. Recycling ratio inconsistent with unity at 2σ.
5. OSL IR depletion-ratio more than 2σ below unity.
6. Figure-of-merit (FOM) value for LX/TX ratio >10%.
7. De value calculated by extrapolation of dose-response curve beyond the largest regenerative dose.
8. LN/TN ratio statistically consistent with or higher than the saturation limit of the dose-response curve.
9. De values below 1 Gy.



**Figure S5-3.** **Single grain quartz** **De values obtained for all Waterfall Bluff OSL samples.** Filled-in circles indicate individual De values obtained from sand-sized quartz. For samples CN311 and CN310 (S5-3a and b) these were measured solely from quartz grains 180-212 µm in diameter. For CN309, CN308 and CN312 (S5-3c-e) these were measured primarily on 150-180 µm-diameter quartz. In the case of the latter, measurements undertaken on 180-212 µm-diameter quartz are indicated by open triangles. The grey bar in S5-3a is centred on the weighted mean De value using the CAM. The two red data points in S5-3e indicate younger intrusive grains that were excluded for MAM calculations.

|  |  |  |  |
| --- | --- | --- | --- |
| **OSL sample code (CN)** | **No. of accepted grains (180-212 µm/150-180 µm)** | **CAM De (180–212 µm) (Gy)** | **CAM De (150-180 µm) (Gy)** |
| 309 | 19 / 298 | 58.15 ± 6.15 | 66.63 ± 2.0 |
| 308 | 15 / 129 | 76.81 ± 10.59 | 88.17 ± 4.05 |
| 312 | 9 / 57 | 111.00 ± 11.06 | 99.28 ± 8.27 |

**Table S5-3. Weighted mean De results obtained for 180–212 and 150-180 µm-diameter quartz grains from samples CN309, CN308 and CN312.** Weighted mean De values were calculated using the CAM (Galbraith et al. 1999).

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Sample (CN)** | **ICP-MS/OES** | | | **TSAC + GMBC** | | |
| **K (%)** | **Th (ppm)** | **U (ppm)** | **K (%)** | **Th (ppm)** | **U (ppm)** |
| 311 | 0.4306 | 4.03 | 0.86 | 0.4268 | 2.28 | 1.00 |
| 310 | 0.8098 | 7 | 1.39 | 0.8142 | 4.95 | 2.60 |
| 309 | 0.8463 | 8.5 | 1.44 | 0.8742 | 7.24 | 2.10 |
| 308 | 0.8827 | 8.46 | 1.77 | 0.9345 | 5.69 | 2.43 |
| 312 | 0.8996 | 8.46 | 2.25 | 0.9249 | 7.39 | 2.32 |

**Table S5-4. Measured elemental concentrations obtained using ICP-MS/OES and a combination of GMBC and TSAC.**

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Sample**  **(CN)** | **ICP-MS/OES** | | | | | **GMBC + TSAC** | | | | |
| **β Dr (Gy/ka)** | **γ Dr (Gy/ka)** | | **Age (ka)** | | **β Dr (Gy/ka)** | **γ Dr (Gy/ka)** | | **Age (ka)** | |
| **Unmodelled** | **Modelled** | **γ Dr unmodelled** | **γ Dr modelled** | **Unmodelled** | **Modelled** | **γ Dr unmodelled** | **γ Dr modelled** |
| 311 | 0.397 ± 0.024 | 0.319 ± 0.020 | 0.429 ± 0.021 | 15.2 ± 0.9 | 13.3 ± 0.7 | 0.375 ± 0.026 | 0.263 ± 0.021 | 0.401 ± 0.021 | 16.8 ± 1.1 | 14.1 ± 0.8 |
| 310 | 0.849 ± 0.031 | 0.656 ± 0.026 | 0.578 ± 0.026 | 11.9 ± 1.2\*  22.0 ± 1.5 | 12.5 ± 1.2\*  23.1 ± 1.6 | 0.950 ± 0.035 | 0.692 ± 0.037 | 0.589 ± 0.027 | 11.0 ± 1.1\*  20.2 ± 1.4 | 11.7 ± 1.2\*  21.5 ±1.5 |
| 309 | 0.877 ± 0.037 | 0.701 ± 0.031 | 0.695 ± 0.030 | 22.0 ± 1.3\* | 22.1 ± 1.3\* | 0.942 ± 0.040 | 0.719 ± 0.047 | 0.700 ± 0.031 | 21.0 ± 1.3\* | 21.2 ± 1.2\* |
| 39.8 ± 1.8 | 39.9 ± 1.8 | 37.9 ± 1.9 | 38.3 ± 1.8 |
| 308 | 0.989 ± 0.035 | 0.780 ± 0.031 | 0.740 ± 0.033 | 27.2 ± 2.0\* | 27.8 ± 2.1\* | 1.038 ± 0.037 | 0.735 ± 0.039 | 0.721 ± 0.033 | 27.1 ± 2.1\* | 27.3 ± 2.1\* |
| 46.9 ± 2.6 | 47.9 ± 2.7 | 46.7 ± 2.6 | 47.1 ± 2.6 |
| 312 | 1.058 ± 0.038 | 0.834 ± 0.033 | 0.808 ± 0.035 | 37.1 ± 4.2\* | 37.6 ± 4.2\* | 1.058 ± 0.037 | 0.799 ± 0.048 | 0.775 ± 0.034 | 37.7 ± 4.3\* | 38.2 ± 4.3\* |
| 51.1 ± 4.1 | 51.8 ± 4.2 | 52.1 ± 4.3 | 52.7 ± 4.2 |

**Table S5-5. Comparison of variations in beta dose rate, gamma dose rate and final OSL age due to a) approach used for beta and gamma dose rate estimation, and b) the employment of gamma sphere modelling.** The CAMwas employed for all age calculations, except for those demarcated (\*), whereby the MAM was used.

|  |  |  |  |
| --- | --- | --- | --- |
| **Sample**  **(CN)** | **β Dr (Gy/ka)** | **γ Dr (Gy/ka)** | |
| **Unmodelled** | **Modelled** |
| 311 | 1.06 ± 0.04 | 1.21 ± 0.04 | 1.07 ± 0.03 |
| 310 | 0.89 ± 0.04 | 0.95 ± 0.04 | 0.98 ± 0.04 |
| 309 | 0.93 ± 0.05 | 0.97 ± 0.05 | 0.99 ± 0.04 |
| 308 | 0.95 ± 0.05 | 1.06 ± 0.05 | 1.03 ± 0.05 |
| 312 | 1.00 ± 0.05 | 1.04 ± 0.06 | 1.04 ± 0.05 |
| *Average ratio* | *0.97 ± 0.05* | *1.05 ± 0.04* | *1.02 ± 0.04* |

**Table S5-6.** Ratios of beta dose rates and gamma dose rates (unmodelled and modelled) obtained through calculations using two different approaches: ICP-MS/OES and TSAC + GMBC.

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