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Optically Stimulated Luminescence Dating

Andrew Murray^{1†}, Lee J. Arnold², Jan-Pieter Buylaert³, Guillaume Guérin^{4,5}, Jintang Qin^{6,7}, Ashok K. Singhvi⁸, Rachel Smedley⁹, Kristina J. Thomsen¹⁰

¹Department of Geoscience, Aarhus University, Høegh-Guldbergs Gade 2, DK-8000 Aarhus C, Denmark

²School of Physical Sciences, Environment Institute, Institute for Photonics and Advanced Sensing (IPAS), University of Adelaide, North Terrace Campus, Adelaide, SA 5005, Australia

³Department of Physics, Technical University of Denmark, DTU Risø Campus, DK-4000, Roskilde, Denmark

⁴IRAMAT-CRP2A, UMR 5060 CNRS - Université Bordeaux Montaigne, Maison de l'archéologie, Esplanade des Antilles, 33607 Pessac cedex, France, and Univ Rennes, CNRS, Géosciences Rennes, UMR 6118, 35000 Rennes, France

⁵Univ Rennes, CNRS, Géosciences Rennes, UMR 6118, 35000 Rennes, France

⁶State Key Laboratory of Earthquake Dynamics, Institute of Geology, China Earthquake Administration, Beijing 100029, China

⁷Xinjiang Pamir Intracontinental Subduction National Field Observation and Research Station, Beijing 100029, China

⁸AMOPH Division, Physical Research Laboratory, Navarangpura, Ahmedabad 380009, India

⁹Department of Geography and Planning, University of Liverpool, L69 7ZT, UK

¹⁰Department of Physics, Technical University of Denmark, DTU Risø Campus, DK-4000, Roskilde, Denmark

†Corresponding email: anmu@dtu.dk

Author contributions

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30

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41

42 **Competing Interests**

43 K.T. and J-P. B. declare a competing interest. They are both employees of the Technical University of
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45 declare no competing interest

46

47 **Abstract**

48 Optically Stimulated Luminescence (OSL) signals from quartz can be used to determine the timing of
49 deposition of sedimentary archives. OSL dating uses the accumulation of energy stored in a crystal
50 structure to measure time. This stored energy is absorbed from ionising radiation, and is released (reset)
51 by heat or daylight. The total energy (dose) absorbed since last resetting is measured using OSL, and
52 divided by the rate of storage (dose rate) to give the time elapsed from the last heating or daylight
53 exposure. In this Primer, we introduce the quartz OSL method and outline the signal resetting processes.
54 We describe the origins and quantification of the dose rate and describe the daylight-sensitive OSL
55 signal most appropriate to dose estimation. The most widely used dose measurement method is then
56 introduced, together with quality-control procedures. A broad set of geological and archaeological
57 studies are used to illustrate the wide range of potential applications, and we describe the challenges
58 arising from different deposition environments and summarize evidence for the precision and accuracy
59 of published ages. Uncertainties and minimum reporting are discussed together with methodological
60 limitations, particularly when applied to young and old sediments. Finally, we highlight the anticipated
61 future developments in the field.

62

63 [H1] Introduction

64 The surface of our planet preserves extensive sediment archives recording the history of our species
65 and its environment, but interpreting these archives depends on knowing when the sediments were
66 deposited. Luminescence dating is one of the main methods used to estimate the depositional age of
67 sediments over the last ~0.5 million years. This Primer is targeted at users of luminescence ages who
68 seek greater understanding of how ages are derived and how to interpret their reliability, and at students
69 and academics intending to implement the method in their work.

70 There are many methods for determining the deposition age of sediments and associated deposits¹, but
71 radiocarbon, cosmogenic radionuclide and luminescence dating are the most widely applicable to the
72 recent geological past. Other methods, such as U-series and Ar-Ar, are site and sample specific and can
73 only occasionally be employed. Radiocarbon dating was developed first, and it still dominates studies
74 in the Neolithic and more recent archaeology. In other time periods (especially beyond ~45 ka) and in
75 many geological applications, luminescence dating has proved to be the most useful. This method dates
76 the last time quartz (and feldspar) was heated or exposed to daylight. It can be applied to nearly all
77 sediments on the Earth's surface, and used to date material over a wide age range, from almost the last
78 decade to ~500 ka ago.

79 Luminescence is light resulting from the release of energy stored in a crystal structure. This signal can
80 be used for dating because the amount of stored energy increases with continuing exposure to natural
81 ionising radiation (and therefore increases with time), and because luminescence can be reset by heat
82 or daylight. As a result of its known sensitivity to heat, luminescence was first applied to the dating of
83 pottery and other ceramics, but its full potential only became apparent when it was realised that the
84 signal was also reset by daylight exposure². Initial studies employed thermoluminescence (TL), but
85 optically stimulated luminescence (OSL) rapidly took over following the first measurements on quartz
86 in 1985³. OSL can be more readily reset by daylight exposure; TL bleaches much more slowly, and is
87 never completely reset. Daylight exposure of sediment grains is ubiquitous during sediment erosion,
88 transport and deposition, and so OSL has the potential to date almost any surficial deposit. This direct
89 dating of the deposition of mineral grains also removes any ambiguity of association, such as occurs
90 with the radiocarbon dating of sediments using organic matter.

91 In nature, luminescence from quartz (and feldspar) arises from the dose absorbed from ionising
92 radiation through time. This radiation is emitted (**Fig. 1a**) primarily during the radioactive decay of ⁴⁰K,
93 and the ²³⁸U and ²³²Th decay series; it creates free electrons in the quartz crystal lattice, some of which
94 are then trapped for long periods in defects in the lattice structure (**Fig. 1b**). Stimulation with light or
95 heat (**Fig. 1c**) releases these trapped electrons, some of which give up their stored energy as photons;
96 this is luminescence **Fig. 2** illustrates how this luminescence depends on prior bleaching (here, natural

97 exposure to daylight), and on the dose rate . After sample collection and preparation, the dose rate is
98 derived from analyses of radionuclide concentrations, and the dose from OSL. In the laboratory, the
99 natural luminescence signal is first measured by illuminating a sub-sample with light (**Fig. 2a**), and this
100 signal is calibrated in terms of dose. The age in years is then calculated from:

$$101 \quad \text{Age [a]} = \frac{\text{Dose [Gy]}}{\text{Dose rate [Gy.a}^{-1}\text{]}} \quad \text{.....(1)}$$

102 This Primer focuses on OSL dating of sand-sized (63-300 μm) quartz grains, using blue (~470 nm) or
103 green (~530 nm) light stimulation and signal detection in the UV band (~340 nm). This choice is based
104 on the relative simplicity of this technique, the signal stability and the ubiquity of the mineral. Other
105 luminescence dating approaches use silt-sized quartz^{4, 5}, silt- and sand-sized feldspar⁶, and/or OSL
106 signals stimulated with violet⁷, or infra-red⁸ light. We outline the principles and the experimental
107 procedures most commonly used in deriving a quartz luminescence age, summarise methodological
108 challenges, and indicate how these are overcome in practice. We then discuss reproducibility and
109 accuracy, limitations, and finally our expectations for future developments.

110 **[H1] Experimentation**

111 Before a luminescence age is measured, various practical steps must be undertaken. First, the sample
112 must be collected without exposure to daylight – for example, from a wind-blown or fluvial sediment,
113 a deep-sea marine core, an archaeological pottery sherd or an anthropogenic cave deposit. In the
114 laboratory, the quartz grains are separated for measurement in darkroom conditions, because the signal
115 is sensitive to light. Any illumination used must be of a wavelength and intensity that minimises the
116 effect on the potential luminescence signal⁹. The measurement equipment includes a stimulation light
117 source, a source of ionising radiation, a heating system and a photon detector to count the photons
118 emitted by the luminescence process. All of these experimental issues are discussed below.

119 **[H2] Sampling**

120 The quartz-containing material - usually sediment - must be collected while avoiding daylight exposure
121 because OSL dates the last exposure of sand grains to daylight. In the field, the surface of the sediment,
122 or of the sedimentary sequence of interest, is first cleaned by scraping back¹⁰, which helps avoid the
123 light-exposed first few mm/cm of material. This also provides a clear view of the stratigraphy, so that
124 locations can be avoided that may have been affected by post-depositional disturbances, such as ice
125 wedges, animal burrows, roots, etc. At least the first few cm must be removed; if possible, it is better to
126 cut a section back by >1 m to avoid air-exposed material. This makes it more likely that the
127 oxidation/reduction conditions and moisture content of the sample are representative of the long-term
128 burial. These operations can be undertaken in full daylight, allowing photographs to be taken of the

129 sampling site and of samples before removal, thus providing a record of sampling locations and their
130 relationship to the general stratigraphy.

131 There are then two approaches to collecting a sediment sample from a cleaned terrestrial deposit. The
132 easiest is to hammer tubes (3-5 cm diameter of metal or plastic, depending on the degree of
133 consolidation of the deposit) into the section. This method is only suitable when the unit to be sampled
134 is of sufficient thickness that the tube will not cross sedimentological boundaries. If possible, the tube
135 should be at least 15 cm from any boundary, so that the γ dose rate gradient across the tube is small. A
136 crushed paper/plastic plug can be placed in the tube to support the sediment during insertion, reducing
137 the risk of collapse and the mixing of exposed and unexposed material. The tube must be filled
138 completely, either with sample or by packing with plastic, to ensure that the material cannot move and
139 mix during transport. In the laboratory, the outer ~2-3 cm of each end of the tube that have potentially
140 been light-exposed are set aside for radionuclide concentration measurements (to estimate the dose rate)
141 and for other sedimentological analyses (for example grain size, mineralogy, geochemistry, ancient
142 DNA, tephrochronology, etc.). The inner part of the tube is used for OSL measurements to estimate the
143 dose (see equation 1), and possibly for water content measurements (Box 1).

144 If the unit is too thin to be sampled with a tube, the second approach is to operate in the dark – either
145 under an opaque covering such as a tarpaulin or at night (moonlight has very low intensity and will not
146 significantly affect the OSL during the short sampling exposure¹¹). In both cases, a controlled light
147 source, such as a red LED or a headlamp emitting dim orange light, can be used. It is usual to collect
148 two parts for each sample: one for OSL analysis, used to estimate the dose, and the other for
149 radiochemical analysis - to calculate the dose rate. First, the previously cleaned surface is scraped back
150 again (~1 cm) as it is likely to have been exposed to daylight after the initial cleaning of the section; the
151 sediment removed during cleaning can be retained for radioelement concentrations measurements and
152 for any other complementary measurements. The second part of the sample, taken from further into the
153 section, must be protected from light exposure. Although a few hundred grams is usually sufficient for
154 later processing, the actual sample size depends on the concentration and the size distribution of quartz
155 grains. For instance, a carbonate-rich sandstone generally contains a smaller proportion of quartz grains
156 than a desert dune sand, and a marine/lake core usually yields fewer sand-sized grains... If a narrow-
157 grain-size fraction is to be targeted for single-grain OSL measurements, the amount of sediment
158 required may be greater than usual to ensure sufficient grains of the desired grain-size range are
159 recovered. This is in contrast to multi-grain aliquots, which can use a different and broader size range,
160 chosen to suit the sample.

161 Marine and lacustrine deposits are often sampled by coring. Although it is desirable to use opaque core
162 barrels, this is not always possible, and successful sampling has been undertaken on poorly-archived
163 light-exposed core material¹². Whenever possible, cores should be split under controlled lighting.
164 However, undessicated material can often be successfully sampled, even if the core was initially

165 processed in full daylight, by scrapping off a few mm of the light-exposed surficial material (this should
166 be retained for the dose rate measurement)^{13, 14}. It is important to recognise that the depth range that can
167 be treated as a single sample varies with core depth. For a given sedimentation rate, the depth interval
168 representing a period of time that is small compared to the age uncertainty increases with core depth.
169 For instance, consider an age uncertainty of 10%, and a sedimentation rate of 10 mm/ka. For sediment
170 that is 10 ka old (100 mm depth), 10 mm of sample depth is equivalent to the age uncertainty of 10%.
171 But at 100 ka (1000 mm), 10 mm of sample only represents 1% of the age; to achieve the same age
172 sampling resolution as at 10ka, a 100 ka old sample can be 100mm thick. This is important because
173 sample size is often a limiting factor in working with core material. For the same reason, vertical mixing
174 of the outer layer of the core by the core-barrel may be important in shallow material, but can often be
175 ignored in deeper, older material without impacting the age uncertainty.

176 If the target is ceramic material such as pottery, bricks, and tiles, or heated or unheated stones and
177 cobbles, all sampling can be undertaken in daylight, although direct sunlight should be avoided. This is
178 because, in the case of ceramics and heated stones, the potentially light-exposed surface of the sample
179 is discarded in the laboratory, while in the case of cobbles, light is attenuated so rapidly with depth that
180 a few minutes of daylight does not affect the average luminescence signal in the outer 1 mm¹⁵.
181 Nevertheless, some rocks (e.g quartzite) are more translucent than others, and more sensitive to light
182 exposure during sampling; it may be prudent to take extra care with such samples.

183 The presence of water reduces the dose rate and increases the calculated age. It is therefore important
184 to estimate the average water content of the deposit over the burial period. During sampling one should
185 consider the water content history of the site and collect samples on which to measure the present day
186 water content. A subsample of the OSL sample from inside the tube or from the radioelement sample
187 (see above) can be used for this, and for determining the saturated water contents. In order to measure
188 a reliable present-day water content, sealable bags should be used and the ends of the tubes made airtight
189 by sealing with plastic.

190 Finally, sampling is the occasion to collect information to measure or estimate the long-term average
191 overburden of a sample. This includes the nature of the overburden, such as a cave roof, sediment, etc.,
192 and the density and the thickness of the material covering the sample. The elevation of the site above
193 sea level and geographical coordinates (latitude, longitude) should also be recorded. These field
194 observations are used collectively to calculate the cosmic ray dose rate¹⁶, which typically contributes
195 ~10% to the total for shallow (<3 m) burial).. Furthermore, in cases where the sediment is heterogeneous
196 on a scale of 10-50 cm, for example because of the presence of rocks, sediment unconformity, boundary
197 between sediment and air or sediment and bedrock, etc., it may be necessary to measure in situ γ dose
198 rates (see below). It is also often appropriate to collect two or more separate dose rate samples, one
199 from sediment to be dated and one from the adjacent rock/sediment causing heterogeneity, in order to

200 measure the radionuclide concentrations in both components and model the γ dose rate (see Appendix
201 H in¹⁷).

202 [H2] Sample preparation

203 In the laboratory, the part of the sample intended for dose rate measurements is homogenised by
204 grinding, so that even the small samples required for radiochemical analyses (typically a few 100 mg)
205 are representative of the whole. Supplementary Table 1 lists typical methods for dose rate
206 determination, and the typical sample size required. Given the usually considerable resource investment
207 in sample collection, it is good practice to keep back a portion of the non-light-exposed material as a
208 substitute in case something goes wrong (mis-labelling, light exposure etc.) during sample preparation.
209 The remaining part of the sample intended for OSL measurements undergoes several treatment steps to
210 extract quartz grains (see **Fig. 3a**). The unexposed inner part of ceramics and heated stones is gently
211 crushed and either it or the sediment is wet sieved to obtain a specific grain-size range. Grain size is
212 important not only because of measurement practicalities, but also because both laboratory and
213 environmental dose rates are, to some degree, grain-size dependent¹⁷. The selection of a grain-size range
214 is driven by the abundance of that size range in the samples, and the practicality of chemically etching
215 the outer surface of the grains to remove the α -irradiated component. Typically, grains must not be
216 smaller than fine sand ($> 63 \mu\text{m}$) or there is a risk that chemical etching completely dissolves the sample
217 Generally, some choice is made within the range of 63-300 μm (for example 90-125 μm , 150-180 μm
218 or 180-250 μm); the 180-250 μm fraction is the most suitable for measurements using single-grain discs
219 with 300 μm diameter holes.

220 In all the steps outlined below, the sample should remain at or close to room temperature to avoid the
221 risk of thermal loss of the OSL signal, especially during the HF digestions steps. HF is a particularly
222 dangerous substance that can be lethal if used incorrectly. Because of this, the operator should carefully
223 familiarise themselves with all local safety procedures and regulations before using HF.

224 The selected size fraction is first treated with 10% HCl (aqueous, as are all solutions below) for at least
225 30 minutes, or until gas evolution ceases completely. This removes carbonates, which may impede the
226 reaction with HF. The sample is then treated with 30% H₂O₂ for up to a few days to remove finely
227 disseminated organics. To more easily disaggregate grains in fine-grained, clayey-silty sediment or
228 carbonate-rich sandstones, the bulk sediment can be soaked in water and treated with HCl and H₂O₂
229 before wet-sieving. After treating with HCl and H₂O₂ and sieving, a few gram(s) of sample should be
230 left - mainly a mixture of quartz, feldspar and sometimes heavy-mineral grains, all in the chosen grain-
231 size range. If only quartz is desired, the sample may then be placed directly in concentrated HF (40%).
232 Usually ~1 hour is sufficient to remove feldspar and etch the surface of the quartz grains (see below).
233 If the feldspar grains are also required, then prior mineral separation is necessary, usually via density
234 separation. K-feldspars, for example, are commonly used in other approaches to luminescence dating⁶.

235 ¹⁸. To improve recovery when density separating quartz from feldspar, surface coatings and attached
236 clay grains can be removed using dilute HF (10%). The density separation step normally uses an
237 aqueous heavy liquid such as lithium heteropolytungstate. This is much safer to use than the more
238 traditional organic liquids, and it is also much more convenient, because it can be readily
239 diluted/evaporated to any density between 1 and $\sim 3 \text{ g.cm}^{-3}$. A density of 2.70 g.cm^{-3} allows any heavy
240 minerals to sink; 2.62 g.cm^{-3} allows a quartz-rich fraction to sink; 2.58 g.cm^{-3} allows a Na-rich feldspar
241 fraction to sink and leaves a K-rich feldspar fraction floating. Some laboratories use only a single 2.58
242 g.cm^{-3} step and rely on the later HF step to remove any denser feldspars that remain in the quartz-rich
243 fraction. Any heavy minerals that persist after the HF treatment rarely, if ever, make a significant
244 contribution to the quartz luminescence signal. Magnetic separation is also used to separate quartz and
245 feldspar grains, instead of density separation¹⁹.

246 Finally, treating the quartz extract with concentrated HF (40%) for 1 hour removes any remaining
247 feldspar. This treatment also etches $\sim 10 \mu\text{m}$ from the outer surface of the quartz grains, thus removing
248 most of the quartz layer exposed to α particles from the surrounding sedimentary matrix during burial
249 (see **Fig. 1**). The internal α activity of quartz is very small and this etching step significantly simplifies
250 the dosimetry calculations. Some laboratories then soak the sample in 10% HCl for several hours to
251 remove any fluorides that might have precipitated on the grain surfaces. Although there is no evidence
252 that such amorphous precipitates are a significant source of contamination in quartz OSL, some fluoride
253 residues can be of sufficient thickness to significantly attenuate the OSL signal. After a final wash, it
254 may then be desirable to re-sieve the samples to define the minimum grain size during β -irradiation and,
255 for single grain measurements, to minimise the risk of multiple grains in each hole in the sample holder.
256 For the measurement of multi-grain aliquots, a few mg of quartz may be placed loose in cups, or adhered
257 as a monolayer to stainless steel discs using silicone oil. The quartz is then tested for signal purity using
258 infrared stimulation (see Box 3). If it fails this test, the HF etching stage can be repeated, although the
259 extra mass loss has a small additional effect on the average natural β dose to the grains.

260 [H2] Dose rate fundamentals

261 The dose rate term in equation 1 is derived from the flux of ionising radiation originating with both
262 natural radioactivity and cosmic rays. For sand-sized quartz grains, the dose rate is dominated by ^{40}K ,
263 which emits β particles and γ rays²⁰, and members of the ^{238}U and ^{232}Th decay series
264 (α, β, γ ¹⁷; Supplementary Figures 2 and 3). The abundance of these radionuclides in sediments depends
265 on the source rock and on differential transport processes related to weathering, erosion, transport and
266 deposition. Some of these processes are physical, such as sorting by size and/or density, while others
267 separate nuclides as a result of their geochemical properties²¹. The dose rate contribution from cosmic
268 rays is usually small, and depends on latitude, altitude and burial depth¹⁶ (Supplementary Figure 1).

269 Direct measurement of the dose rate is complex, particularly because the various radiations penetrate
270 matter differently; in sediments, α particles travel distances of up to $\sim 20 \mu\text{m}$, β particles up to $\sim 3 \text{ mm}$
271 and γ rays up to $\sim 50 \text{ cm}$ (**Fig. 1a**). Fortunately, in many cases the key dosimetric concept of an infinite
272 matrix can be used to circumvent this difficulty^{17, 22}; in an infinite matrix all the energy emitted in the
273 medium must also be absorbed in the medium, provided that the medium is homogeneous over
274 dimensions significantly larger than the distance travelled by the relevant radiation. Thus, one only
275 needs to know the energy emission rate to know the dose rate. The infinite matrix has different
276 dimensions depending on the radiation: for γ rays it is a body of sediment $>0.75 \text{ m}$ in diameter, and for
277 β particles it is only about 5 mm in diameter.

278 The rate of energy emission in an infinite matrix is calculated from the concentrations of all the relevant
279 radionuclides. The activity per unit mass [Bq.kg^{-1}] of a certain nuclide can be estimated directly, by for
280 example alpha counting or γ spectrometry (see Supplementary Table 1), but very often only the mass
281 concentrations of K and the series parents U and Th are measured using, for example, inductively
282 coupled plasma mass spectrometry. In order to deduce the activity concentrations of other radionuclides
283 in the U and Th decay series, the condition known as secular equilibrium is often assumed. In some
284 cases this assumption is invalid, because members of the decay series have been separated by their
285 differential geochemistries^{21, 23}, and a more detailed knowledge of the state of equilibrium is required.
286 The latter may or may not be time-dependent, depending on whether the system is open or closed to
287 radionuclide migration.

288 The lifetime-averaged water content of the sediment is also important (see Box 1). Water absorbs energy
289 but since it does not usually contain significant radioactivity, it does not emit energy. The net effect is
290 to reduce the dose rate.

291 [H3] In situ γ dose rate measurements

292 In cases where sediment is heterogeneous on the scale of the penetration distance of γ rays ($\sim 30\text{-}50 \text{ cm}$,
293 see Figure 1a) the infinite matrix γ dose rate may not be representative of the γ dose rate to which the
294 sample was exposed during burial. These cases include poorly sorted sediment, sediment including
295 pebble and cobbles, or sediment adjacent to a rock wall. As an alternative, the γ dose rate may be
296 measured in situ at the sample location using, for instance, luminescent aluminium oxide ($\text{Al}_2\text{O}_3\text{:C}$)
297 pellets. Aluminium oxide is very similar to quartz in terms of dose absorption characteristics, and
298 doping with carbon gives this material a very sensitive luminescence response to dose²⁴. In practice,
299 pellets are left buried in an aluminium tube for 6-12 months, after which the absorbed dose is sufficient
300 to induce a measurable OSL signal^{25,26}. The aluminium tube has a $\sim 2\text{-}3 \text{ mm}$ wall thickness to prevent
301 beta particles from reaching the pellets. This directly-measured dose rate is the sum of γ and cosmic
302 dose rates, as affected by escape of ^{222}Rn and the water content of the sediment over the period of
303 measurement. Escape of the noble gas ^{222}Rn can be important because the decay of this radionuclide

304 gives rise to about 60% of the beat dose rate from the ^{238}U series, and 98% of the gamma dose rate (see
305 Table S2b). In contrast to dose rates derived from radionuclide concentrations, correcting in situ dose
306 rates for past variations in ^{222}Rn escape or water content requires some independent knowledge of the
307 effect on the calibration of the dosimeter of such variations, and this information is not usually available.
308 The other common way of determining in situ γ dose rates involves the use of a field γ spectrometer,
309 typically with a NaI(Tl) or LaBr probe. Calibration involves measurements in infinite media of known
310 radionuclide concentrations²⁷. Data analysis may consist of estimating effective K, U and Th
311 concentrations using the ‘windows technique’ (see Appendix L in¹⁷), and then converting these
312 concentrations into effective infinite matrix dose rates. The concentration of K is based on the emission
313 rate of the γ ray peak at 1461 keV, of U on the 1764 keV peak and of Th on the 2615 keV²⁸.
314 Alternatively, the proportionality between total γ dose rate and the count rate above a certain energy is
315 used in the ‘threshold’ technique^{29,30}. The advantage of this approach lies in the inclusion of a greater
316 portion of the recorded spectrum to estimate the γ dose rate, thereby reducing the uncertainties for a
317 given counting time (typically several minutes), but this approach does not give any nuclide-specific
318 information. It is also difficult to correct such threshold data for past variations in ^{222}Rn escape or water
319 content without independent knowledge of the U, Th and K contributions to the total.

320

321 [H2] Measuring the Equivalent dose (D_e)

322 The instrumentation for determining the dose absorbed during burial and its equivalent in terms of
323 producing luminescence includes a luminescence detector, a stimulation light source, a source of
324 ionising radiation and a heating system. **Fig. 3b** summarises the relationship of these four components
325 with respect to the quartz grains to be measured.

326 The main challenge in designing such a system is instrument sensitivity. The precise measurement of
327 an absorbed dose depends both on the sensitivity of the sample - equivalent to the number of photons
328 emitted per unit mass and per unit absorbed dose [$\text{kg}^{-1}\cdot\text{Gy}^{-1}$], and on the detection efficiency - the
329 fraction of the emitted photons that are actually detected by the instrument. The relationship of the
330 resulting signal to the instrument background noise must also be considered. Detection efficiency
331 depends on the light path, the quantum efficiency of the photon detector, and the wavelength and
332 intensity of the stimulation light source. The latter are important because the more rapidly the charge is
333 released or detrapped (Figure 1c), the stronger the initial luminescence signal compared to background
334 noise. For a given detection wavelength, the electron detrapping rate depends on the wavelength-
335 dependent photoionization cross-section³¹ and the power density of the stimulation light at the sample.

336 The luminescence detector consists of a photon detector and detection filter(s). To precisely measure
337 weak luminescence signals, a highly sensitive light detector with a low background count rate, such as
338 a photomultiplier, and good light collection are essential. The latter requires that the solid angle

339 subtended by the detector surface at the sample be as large as possible – this is usually achieved by
340 minimising the distance between sample and detector or by focussing through lenses. In OSL, the
341 luminescence signal is emitted as a consequence of optical stimulation, which is $\sim 10^{15}$ times more
342 intense than the OSL signal itself³². Thus appropriate detection filters are necessary, both to separate
343 the luminescence from the stimulation light and to define the spectral detection window.

344

345 [H3] Photon detector

346 Quartz has a relatively broad OSL emission peaking at ~ 360 nm, with a full width half maximum (
347 FWHM) of ~ 80 nm (see Supplementary Figure 6). Photomultiplier tubes (PMT) operating in photon
348 counting mode and sensitive in the blue/UV range are most commonly used for detecting quartz OSL,
349 because of their high sensitivity, large dynamic range and low dark count rate. A large signal-to-noise
350 ratio is particularly important when measuring insensitive samples and/or samples having absorbed
351 small radiation doses, such as young samples. The quantum efficiency of the PMT is typically up to
352 ~ 30 % depending on the wavelength of the luminescence. However, PMTs do not provide information
353 on the spatial variation of the emitted luminescence. In applications where such spatial information is
354 of importance, such as rock surface luminescence dating, electron-multiplier charge-coupled devices
355 (EMCCD) can be used but they are generally less sensitive than PMTs (see examples in³³⁻³⁵).

356

357 [H3] Detection filters

358 The use of detection filters is important to define the wavelength window of interest and to separate the
359 emitted luminescence from scattered stimulation light. In quartz OSL dating, the emission is usually
360 detected through a filter with a peak transmission at 340 nm and a FWHM of 80 nm (Supplementary
361 Figure 7). The main emission from quartz is centred on 365 nm and, in principle, a significant
362 improvement in light detection is available by choosing a longer wavelength detection filter closer to
363 the main emission of quartz. However, moving the detection window closer to the typical stimulation
364 wavelength of 470 nm also gives an increase in background signal resulting from breakthrough of the
365 stimulation light, and hence does not necessarily improve the signal-to-noise ratio³⁶.

366

367 [H3] Stimulation light source

368 The electron detrapping rate depends both on the intensity and the wavelength of the optical stimulation
369 light^{37, 38} (see Supplementary Figure 8); the more intense and shorter the wavelength, the stronger the
370 OSL signal per unit time. For most applications, optical stimulation is at a constant light intensity
371 (continuous wave mode), but modulating the intensity of the stimulation light, for example in linear
372 modulated OSL³⁹, can be used to visually separate the signals from the different traps involved in OSL
373 production. In pulsed OSL [POSL], pulsing the stimulation light and only detecting in between short

374 ~50 μs bursts of stimulation light can help to separate quartz and feldspar OSL signals instrumentally⁴⁰.
375 POSL is also used to gain insight into luminescence recombination processes⁴¹.

376 Optical stimulation of quartz can be achieved using any visible light source such as filtered halogen
377 lamp systems³⁶, lasers^{34, 42, 43}, light emitting diodes (LEDs)³² and laser diodes^{34, 44}. In most quartz multi-
378 grain OSL dating applications, blue (450-470 nm) or green (~525 nm) LEDs are used, with power
379 densities at the sample position ranging between ~40 and 100 mW/cm^2 . These result in the decay of the
380 fast OSL component within a few seconds (see below). As LEDs have a broad emission at FWHM ~30
381 nm, it is often necessary to stimulate the luminescence through cut-off filters to ensure an effective
382 separation of stimulation light and detection windows⁴⁵ (see Supplementary Figure 7). Shorter
383 wavelength light sources such as 405 nm laser diodes⁷ can be used to access deeper traps insensitive to
384 blue light. In single-grain applications, stimulation by a green 532 nm solid state laser providing a power
385 density of ~50 W/cm^2 is common⁴⁶.

386 [H3] Source of ionising radiation

387 The measured natural OSL signal must be calibrated in terms of absorbed dose. As different sub-
388 samples/aliquots of quartz can have very different luminescence sensitivities, it is necessary to calibrate
389 aliquots individually. This calibration is performed by comparing the natural OSL signal with that
390 arising from a known laboratory dose given to each aliquot (see below and Figure 4). For convenience,
391 a β source containing the artificial radionuclide ⁹⁰Sr with a half-life of 28.8 years and its short-lived
392 progeny ⁹⁰Y is usually used to deliver the laboratory dose {Aitken, 1985 #359} {Andersen, 2003 #434}.
393 This combination of emitters has a maximum β energy of 2.27 MeV and a mean energy of ~600 keV,
394 which makes it relatively easy to shield sufficiently for it to be safe to use.

395 The laboratory dose rate to a quartz sample depends on the activity of the source, the source/sample
396 distance, sample grain size, and the substrate material on which the grains sit. Because of this, the source
397 must be calibrated for each irradiation geometry, particularly for different substrates and grain-size
398 ranges. The calibration is usually based on a portion of quartz grains that has been given a known dose
399 using a calibrated γ source. An appropriate measurement protocol is used to determine the duration of
400 β irradiation that provides a sensitivity-normalised luminescence intensity equal to that induced by the
401 reference γ dose⁴⁷ (see below). Some laboratories provide sensitive quartz that has already absorbed a
402 well-known γ dose⁴⁷, which allows those without access to a calibrated γ source to calibrate their own
403 β source as required.

404 [H3] Heating system

405 Finally, to obtain an accurate D_e , it is important to perform reproducible thermal pre-treatments and to
406 control the temperature of the aliquot during optical stimulation. OSL dating systems usually heat up to
407 700 °C, controlled by user-defined linear heating rates typically ranging between 0.01 and 10 °C/s. This
408 heating capacity allows for flexibility in application, particularly when measuring TL. In routine quartz

OSL measurements, temperatures above 280 °C are rarely used and heating rates in the range of 2 to 5 °C/s are typical.

[H3] Instrument reproducibility

Very little has been published on formally testing the stability and reproducibility of the various components of an OSL measurement system, but there has been some examination of the overall reproducibility of the measurement of an OSL signal. After allowing for systematic changes in sensitivity in one automated system, repeated cycles of dosing, preheating and OSL measurement using multi-grain aliquots gave a standard deviation of ~1% in the resulting OSL signals⁴⁸.

[H1] Results

[H2] Deriving the dose rate

Radionuclide concentrations vary considerably from one location to another⁴⁹ (Supplementary Figure 4 a-e) because of changes in grain size and mineralogy, with the result that sample-specific dose rates are required. Because well-sorted sands tend to be lower in radioactivity than clays or silts, the effective age range of the technique can often be extended by targeting sandy sediments - the lower the dose rate, the greater the dating range. Radionuclide concentrations within quartz grains are very low⁵⁰, and the dose rate to such grains is usually derived almost entirely from their surrounding environment. Sand-sized quartz grains (usually 63 - 300 µm) are etched in concentrated HF during sample preparation to remove the outer 10-20 µm layer^{17, 19}, which reduces the external alpha dose rate contribution to negligible proportions. The effective total dose rate to well-sorted sands is typically ~1-2 Gy.ka⁻¹ (Supplementary Table 3), although this value can vary considerably⁴⁹. In dry sediment, typically ~60% of the dose rate is from β particles and ~30% is from γ rays; the remaining ~5-10% contribution is from cosmic radiation. The contribution from ⁴⁰K alone may be ~40-50% of the total (Supplementary Figure 3), although this is variable (Supplementary Figure 4e,f) because of the considerable range of relative activity concentrations in natural sediments.

Depending on how the concentrations of radionuclides have been derived, they may be expressed as activity concentration A [Bq·kg⁻¹] or mass concentration C₀ [g·kg⁻¹]. The first step in deriving the infinite matrix dose rate is to convert mass concentrations into activity concentrations. Then the atom concentration per unit mass of sediment N [kg⁻¹] for a particular nuclide is calculated using

$$N = (C_0/m_a) \cdot N_A \quad (2)$$

where m_a is the atomic mass (for our purposes with units [g·mol⁻¹]) and N_A is Avogadro's number [mol⁻¹]. Then the activity concentration is given by

$$A = N \cdot \lambda \quad (3)$$

441 where λ is the relevant decay constant [s^{-1}]. Measured concentrations may be reported by external
442 analysts in ppm [$\mu g \cdot g^{-1}$] for U and Th, and percentage [% , 10 $mg \cdot g^{-1}$] for K; these must first be converted
443 to [$g \cdot kg^{-1}$]. In the case of K, the mass concentration of ^{40}K is then derived using the isotopic abundance
444 (0.0117%) before using equations (2) and (3).

445 Finally, for each nuclide and type of emission (α, β, γ), the infinite matrix dose rate is given by the
446 product $A \cdot \bar{E}$, where \bar{E} is the average energy emitted per disintegration usually given in MeV (1
447 MeV = 1.6×10^{-13} J , see [The National Nuclear Data Center](#)). Once the infinite dose rate for each nuclide
448 and each radiation type has been derived, they are summed over all radionuclides to give the infinite
449 matrix dose rates for each type of radiation, as in the example given in Supplementary Table 3.
450 Relevant conversion factors ($Gy \cdot ka^{-1}$)/($Bq \cdot kg^{-1}$) are summarised in Supplementary Table 2, and are
451 regularly compiled and updated⁵¹.

452 The example spreadsheet (Supplementary Data) attached to this primer allows the reader to work
453 through the calculation of dose rates using the infinite matrix assumption starting from concentrations
454 of K, U and Th. Such calculations provide estimates of the total amount of energy emitted (and thus
455 absorbed) in the sediment per unit time, regardless of the heterogeneity in the radionuclide
456 concentrations, and thus in the radiation field around each grain. They thus give the arithmetic average
457 dose rate to the grains of interest. Such heterogeneity in dose rate is to be expected in almost all sandy
458 deposits (ref Mayya et al., 2006, RM, Guérin et al., 2015, QG). In these circumstances it can be shown
459 mathematically that an accurate age can only be obtained by using the average absorbed dose in
460 equation 1 (ref Guérin et al., 2017, QG). This problem of the most appropriate dose estimation model
461 to use in the age equation is considered further below.

462 When dose rates vary in time and/or space, the infinite matrix assumption is not met⁵². A heterogeneous
463 medium results in a heterogeneous radiation field and thus leads to different doses absorbed by different
464 quartz grains. Small-scale heterogeneity on the μm - mm scale affects radiation travelling only short
465 distances, such as α and β particles. For example, K-rich feldspar grains in a quartz-dominated sand
466 provide localized high concentrations of ^{40}K and different β dose rates to different quartz grains
467 (Supplementary Figure 5b^{53, 54}). Larger scale heterogeneity on the cm scale also affects γ dose rates; if
468 limestone blocks, low in ^{40}K , ^{238}U and ^{232}Th , are embedded in a higher activity sediment, the γ dose
469 rates to sand grains within a few tens of cm of the blocks will be lower than those to grains distant from
470 the blocks (see Supplementary Figure 5a). The radiation field may also vary with time, for example as
471 radioactive material is liberated and exported by reducing groundwater or the hydrolysis of feldspar, or
472 as non-radioactive material is imported into the deposit with infiltrating rainwater or groundwater,
473 leading to, for instance, the precipitation of carbonates or silicates⁵⁵.

474 [H2] Deriving the equivalent dose D_e

475 Estimation of the dose absorbed during burial (termed the natural-, burial- or palaeo-dose) lies at the
476 heart of any luminescence age, and reliable dose estimation depends on the OSL signal used. The
477 luminescence signal from quartz is complex, but with some exceptions⁵⁶ there is evidence that the part
478 of the signal known as the fast-component derives from a single type of trap, which appears to empty
479 according to first-order kinetics, if held at an appropriate temperature⁵⁷. As a result, this fast component
480 is largely independent of the other components⁵⁸, and because it follows first-order kinetics, the fast-
481 component consistently shows a sample- and dose-independent exponential decay shape with
482 stimulation time (Fig.4c inset)^{58, 59}. Importantly, only the luminescence characteristics of the fast-
483 component (particularly stability and light sensitivity) are sufficiently well-known to be used in a
484 reliable estimation of natural dose. The fast-component signal is stable (over $\sim 10^8$ years)⁶⁰ and is also
485 most readily reset by daylight, making it most likely to be sufficiently reset in nature. It can also reset
486 by heating⁶⁰. Heating prior to burial increases the sensitivity of the fast-component to dose^{59, 61-63},
487 allowing the measurement of much smaller doses. In particular, ceramics only a few years/decades old
488 can often be dated, and this can also apply to naturally heated sediments such as a lava flow. Although
489 the fast-component OSL signal increases with laboratory dose often up to many hundreds of Gy, natural
490 doses tend to be underestimated above ~ 150 -250 Gy. This effective dose limit is often well below the
491 largest dose that can be measured under laboratory conditions, and the reason for this discrepancy is
492 not well understood⁶⁴⁻⁶⁶. When considered together with the value of the dose rate, it is this upper limit
493 to the usable dose that defines the maximum age of the method. This maximum is typically ~ 150 ka,
494 although ages of >1 Ma have been reported in unusually low dose rate environments⁶⁷⁻⁶⁹. In unusually
495 high dose rate environments, the upper age limit will be correspondingly lower. The simplest way to
496 confirm the presence of the fast-component is to compare the initial rate of signal loss during stimulation
497 with that of a standard sample known to be fast-component dominated (**Fig. 4c** inset using quartz
498 from⁴⁷) or to use the ratio of the initial intensity during stimulation to the intensity remaining later in
499 the stimulation period^{70, 71}.

500 Experimentally, it is important to ensure that the fast-component dominates the numerical estimates of
501 OSL used in dose calculations to avoid contributions from slower components with different bleaching
502 and thermal stability characteristics, and to avoid possible contamination by feldspar OSL. Such signal
503 separation can be achieved by component fitting^{37, 72} using a sum of exponential decay terms. In
504 addition, separation can also be improved by stimulating with green light instead of blue because the
505 difference between optical cross-sections for release of charge from the fast and slower components is
506 greater for longer wavelengths⁷³. Nevertheless, slower components can also be represented by
507 independent decaying exponential curves, and there is an order of magnitude difference in decay rate
508 between the fast and slower components when stimulating with blue light⁵⁹. For most multi-grain
509 applications where the stimulation power is the same from aliquot to aliquot, this difference in decay

510 rates allows the use of simple summation, called early background subtraction^{74, 75}, to discriminate
511 against the slower components. In early background subtraction, a signal proportional to the intensity
512 of the fast-component is calculated by subtracting the luminescence observed in the period after the
513 beginning of stimulation from the initial luminescence observed at the beginning of the stimulation
514 period. The signal difference is effectively zero in the absence of any fast-component, because the
515 medium and slower components do not decay significantly in the time between the two summation
516 periods.

517 Natural quartz has very heterogeneous luminescence and only a few % of grains give a significant fast-
518 component signal⁷⁶. Thus there are considerable advantages in measuring aliquots containing hundreds
519 to thousands of grains. This approach is only recommended if it is unlikely that useful information is
520 contained in the distribution of doses recorded by different grains, such as that resulting from
521 incomplete resetting or post depositional mixing (this reservation does not apply to dose rate
522 heterogeneity⁷⁷). Luminescence is averaged over many grains when working with such multi-grain
523 aliquots, and the Central Limits theorem suggests that these average OSL signals will be normally
524 distributed.

525 [H3] Dose estimation using the Single Aliquot Regenerative (SAR) dose procedure

526 In regeneration methods, the dose absorbed by an aliquot of quartz grains is estimated by first measuring
527 the natural luminescence resulting from the natural dose. Luminescence signals are then regenerated in
528 the same aliquot by various known laboratory doses given after laboratory resetting. These regenerated
529 signals are then used to construct a curve of luminescence response to dose. Finally, the natural
530 luminescence signal is interpolated onto the laboratory dose response to obtain an estimate of the natural
531 dose. However, sensitivity changes from measurement to measurement have been observed for any
532 regeneration of the signal in the laboratory, rendering interpolation inaccurate. Although such changes
533 can arise from several sources, all are expressed as changes in the sample response to dose.

534 The Single Aliquot Regenerative (SAR) dose procedure⁷⁸ (see **Box 2** and **Fig. 4**) successfully
535 overcomes this problem by using the luminescence response to a fixed test dose to correct for sensitivity
536 changes. SAR is now the most widely used dose estimation method in retrospective dosimetry including
537 dating. It is particularly useful because all the measurements needed for a single dose estimate are made
538 on one aliquot, which can be as small as an individual grain. SAR is thus one of only two procedures
539 that have been used to determine distributions of dose in quartz grains within a sample (the other
540 procedure being the Single Aliquot Added Dose – SAAD –⁷⁹). This sensitivity-change correction, and
541 the assumptions implicit in its use, are discussed using the simple model given in **Box 2**. The most
542 important outcome of this model is that the sensitivity-corrected luminescence signal is independent of
543 the measurement cycle, which is confirmed experimentally (**Fig. 5a**, data obtained using the
544 measurement sequence of **Fig. 5b**)⁷⁸.

545 The model in **Box 2** suggests that the luminescence response is unlikely to follow simple exponential
546 saturation. Nevertheless, it is common practice for interpolation to fit the response of L_i/T_i to dose with
547 an expression of the form:

$$548 \quad \frac{L_i}{T_i} = \frac{L_S}{T_S} \cdot (1 - e^{-\frac{D}{D_c}}) \dots (6)$$

549 where the constant $\frac{L_S}{T_S}$ is the sensitivity-corrected light level at saturation, and the constant D_c is
550 commonly described as the characteristic saturation dose. D_c may be referred to as D_0 in the literature,
551 but here we reserve this for the natural dose in a SAR sequence (see **Box 2**). D_c is only truly constant
552 (and equal to $1/b$ in b) if $\chi(D)$ in **Box 2** is constant, which is unusual. Fortunately, interpolation of the
553 natural signal onto a curve built from the regenerated signals ensures that the resulting dose is almost
554 independent of models of luminescence response to dose, in marked contrast to a result obtained by
555 extrapolation.

556 The signals resulting from the use of the typical SAR measurement sequence (**Fig. 5b**) with a single
557 multi-grain aliquot of quartz extracted from a heated brick are shown in **Fig. 4a-c**, and with quartz from
558 an unheated sediment in Figure 4d-f. The values for L_i and T_i were derived by summing the signals
559 observed in the first 0.32 s (~25 mJ) of stimulation and subtracting the next 0.32 s as background. These
560 measurements are discussed below.

561 In the first SAR cycle ($i=0$), D_0 was absorbed during burial and the natural signal is measured. This
562 natural OSL signal (L_0) from the brick quartz is shown as a dashed horizontal line in **Fig. 4b**, together
563 with the following test dose response (T_0) in **Fig. 4a** (solid blue symbol plotted at 0 Gy). At the end of
564 the cycle, the aliquot is bleached to ensure it does not contain any residual signal (step 7 in **Fig. 5b**),
565 before returning to Step 1 for the second cycle ($i=1$, $D_1=0.5$ Gy); and the second cycle signals (L_1 and
566 T_1) are then measured. Repeating the measurement cycle continues with values of D_i increasing by 0.5
567 Gy steps until $D_{11}=10.5$ Gy. In the following cycle, $D_{12} = 0$ Gy to give L_{12} (downwards open triangle in
568 **Fig. 4b**) and T_{12} in **Fig. 4a**. The 2.5 Gy point is then repeated (L_{13} - open square in **Fig. 4b**, T_{13}), and
569 finally a repeat measurement (L_{14} - also open square - and T_{14}) of the response to a 10.5 Gy regenerative
570 dose. **Fig. 4c** shows the resulting sensitivity-corrected dose response curve obtained by dividing each
571 value of L_i by the corresponding T_i . The intercept of the natural signal, L_0/T_0 , on the smooth curve
572 passing through the regenerated L_i/T_i data is 3.8 Gy. Without the sensitivity correction, an incorrect
573 value of 1.8Gy would have been obtained (**Fig. 4b**).

574 The same process was repeated for the sedimentary quartz aliquot (**Fig. 4d-f**), but with different values
575 of D_i , and additional repeated dose points. In this case, the corrected dose estimate is 25 Gy in contrast
576 to a value of 14 Gy without sensitivity correction.

577 It is important to note that the SAR procedure determines the laboratory β dose (as delivered by a
578 relatively simple unscattered β spectrum) that produces the same luminescence response as the natural
579 dose (delivered by complex heavily-scattered α , β , γ and cosmic ray spectra). This matching laboratory
580 dose may not be identical to the natural dose and is therefore termed the equivalent dose D_e owing to
581 the different luminescence efficiencies of different radiations, especially alpha, cosmic rays and low
582 energy scattered electrons and photons^{17, 77, 80}.

583 [H3] Testing the applicability of a SAR protocol

584 SAR is an empirical procedure supported by modelling⁸¹. Reliability can never be assumed a priori and,
585 as far as possible, its applicability should be tested on each sample. In routine application, measurements
586 of the recycling ratio and recuperation are usually built into any individual SAR dose measurement.

587 A recycling ratio is derived from a repeated measurement of at least one dose point (see open symbols
588 in Figure 4, $D \neq 0$ Gy), to test the accuracy of the sensitivity correction. The recycling ratio is the ratio
589 of the second regenerated value of L/T to the first ($L_2/T_2:L_1/T_1$) and should be close to unity.

590 Recuperation is measured by determining L/T for $D = 0$ Gy (see open symbols in Figure 4 for
591 regeneration dose of 0 Gy). This measurement determines the carry-over of any residual signal from
592 the previous measurement cycle, and it should be small compared to (L_n/T_n). In some samples,
593 recuperation may be significant (>5%), and a further optional step added⁸² to the original SAR
594 procedure (step 7 in Figure 5b) can reduce it. In very young samples, seemingly significant recuperation
595 (>>5%) is often ignored without apparently compromising the accuracy of the ages⁸³.

596 Measuring the recycling ratio and recuperation tests the reproducibility of the sensitivity-corrected
597 luminescence response to doses given in the laboratory. In addition, evaluating the importance of
598 thermal pretreatment, thermal transfer and test dose size, and the dose recovery ratio all contribute to
599 the evaluation of the applicability of a SAR protocol.

600 Thermal pretreatment or preheating before luminescence measurement is required⁸⁴ for accurate
601 estimation of the D_e . The thermal dependence of D_e arises largely because of changes in the probability
602 of luminescent recombination⁸⁵, and because of the presence of less-light-sensitive, thermally-shallow
603 unstable traps, which give TL peaks at 110, 180 and 220°C. Charges released from these traps may be
604 recaptured by the deeper fast-component trap and identified with a thermoluminescence peak at 325
605 °C^{57, 86}. Alternatively, charge released during stimulation from the fast-component trap may be retained
606 by these shallow traps in a process known as optical transfer. Optical transfer processes are of different
607 significance in measurement following burial compared to measurement after laboratory dosing. For
608 instance, the shallow trap at 110 °C is unstable at environmental temperatures and so remains essentially
609 empty during burial, whereas deeper traps will retain some charge. In contrast, all traps fill during

610 irradiation on a laboratory timescale. Both optical transfer processes affect the intensity of the fast-
611 component signal; after irradiation in the laboratory, the shallow trap at 110 °C must be emptied by
612 preheating or else the intensity of the regenerated fast-component signals will be affected and result in
613 a significant error in D_e ⁸⁷. To this end, preheating is mandatory (step 2 in **Box 2**) because it helps isolate
614 the fast-component by emptying other less-light-sensitive traps that deplete more slowly but contribute
615 to the OSL signal. Preheating also speeds up the transfer of charge after laboratory irradiation,
616 simulating the effect of storage time: it both empties shallow traps and transfers some charge to the fast-
617 component trap, as similarly happens during burial. Optical transfer from the 110 °C trap is also why
618 stimulation is usually carried out at a temperature sufficient to prevent the accumulation of charge in
619 the 110 °C trap. In practice, this stimulation temperature is usually 125 °C⁵⁷.

620 However, thermal pretreatment also has undesirable effects: the S and/or χ (see Equation 4 in **Box 2**)
621 appropriate to the natural dose may respond differently to preheating compared with the S and/or χ
622 appropriate to a laboratory dose. In a process known as thermal transfer, preheating may also induce a
623 fast-component signal in an otherwise well-bleached modern sample through charge transfer from light-
624 insensitive shallow traps into a previously empty OSL trap. One way of testing for the significance of
625 all the above-mentioned effects is to look for a plateau in D_e estimates following preheating to various
626 temperatures. This analysis enables determining the range over which the estimated dose is independent
627 of preheat temperature (**Fig. 6a**). The sample should also be preheated immediately before measurement
628 of T_i because the test-dose response can help track the sensitivity of the fast-component. This second
629 preheat step (Step 5, ‘cut heat’ in **Fig. 5b**) must be above 130 °C to empty the 110 °C trap, and is usually
630 at least 20 °C below the regeneration preheat temperature in practice.

631 Thermal transfer of charge to the fast-component trap from low-temperature light-insensitive traps can
632 make a well-bleached sample, with negligible charge in the fast-component trap at the time of burial,
633 appear to be incompletely bleached. These low-temperature traps are sufficiently stable in nature to
634 retain a significant trapped charge population, and because they are light-insensitive they are therefore
635 difficult to empty before burial. As a result, they act as charge reservoirs. Thermal transfer can move
636 sufficient charge from these reservoirs into the fast-component trap to result in an overestimation of D_e .
637 In general, this phenomenon is unlikely to be of significance in older samples, but in young samples,
638 where there is only a small amount of trapped charge in the OSL trap from the most recent burial period,
639 it can be of considerable importance. The potential significance can best be examined by bleaching a
640 set of aliquots in the laboratory either by artificial or daylight stimulation, followed by a preheat plateau
641 test as described above. The apparent dose at low preheat temperatures such as 160°C should be
642 negligible (**Fig. 6b**). However, the apparent dose increases as the preheat temperature is increased and
643 charge is increasingly transferred to the OSL trap. The choice of maximum acceptable preheat
644 temperature is then made by comparison with the D_e ^{88, 89}.

645 Any dependence of D_e on test dose size can be similarly examined to find a test dose range over which
646 the D_e is constant. The OSL from a small test dose can be affected by thermal transfer, especially if the
647 preceding regeneration dose was large, and too large a test dose may lead to dose underestimation⁷⁸. In
648 general, it seems that a test dose of ~30% of the natural dose is a good compromise. However, a value
649 equal to or larger than the D_e may be necessary for young or insensitive samples to ensure a measurable
650 test dose signal.

651 Although all the tests described above are important to a greater or lesser degree, the dose recovery
652 ratio⁹⁰ provides the single most complete test of SAR performance for any given sample. It can be used
653 to assess whether a specific SAR protocol can accurately determine a known dose administered when
654 the sample is in a condition as close as possible to that when it was initially reset before burial. To
655 achieve this, any natural trapped charge population in an aliquot is first removed by artificial or daylight
656 exposure without any thermal treatment before the aliquot is given a known laboratory dose. While the
657 given laboratory dose is typically similar to the D_e , young samples may necessitate a larger dose for the
658 precision required. The given dose is then measured using the chosen SAR protocol. The dose recovery
659 ratio is the ratio of the measured dose to the given dose and should have a value close to unity (**Fig.**
660 **6c,d**). The given dose must be administered before any pretreatment (especially before any preheating)
661 other than light exposure to distinguish the dose recovery ratio from the recycling ratio.

662 Although usually reported as a dose recovery ratio, it is important to realise that the measured quantity
663 is sensitivity-corrected luminescence (L/T), not dose. At low doses, in the more linear part of the dose
664 response curve, dose behaves similarly to sensitivity-corrected OSL (**Fig. 7**, black OSL distribution at
665 20% of saturation). In contrast, at high doses where the response curve becomes significantly sub-linear,
666 even small uncertainties in L/T are magnified by interpolation and dose uncertainties can become
667 unbounded. For a normal OSL distribution with a relative standard deviation (RSD) of 5% centred on
668 86% of saturation ($2xD_e$, **Fig. 7**, blue data), almost all signals can be interpolated onto the growth curve.
669 However, the resulting dose distribution is significantly asymmetrical and the RSD is ~20% of the mean
670 dose or 4 times the measurement uncertainty on L/T . In contrast, for an OSL signal with 5% RSD
671 centred on 95% of saturation ($3xD_e$), ~15% of aliquots cannot be interpolated onto the dose response
672 curve and the RSD of the apparent dose distribution is ~30% of the mean (**Fig. 7**, green data).

673 Alternatively in a dose recovery measurement, if the regenerative dose D_1 is chosen equal to the given
674 dose D_0 then the recovery ratio is more usefully expressed as the luminescence ratio $(L_0/T_0)/(L_1/T_1)$.
675 This OSL ratio is a more sensitive measure for the reliability of a chosen protocol because the
676 dispersions in the OSL distributions will always be \leq those in the dose distributions. The OSL ratio also
677 avoids the incorrect rejection of values when measuring close to saturation. In true saturation, the
678 average luminescence ratio should be unity, with 50% of observations (for a normal distribution) lying
679 above unity⁹¹ (**Fig. 7**, red data). However, luminescence ratios above unity cannot be interpolated to

680 give doses, and so must be ignored if a dose ratio is reported. This incorrectly biases dose estimates to
681 low values (**Fig. 7**), and produces an inaccurate dose distribution with pronounced asymmetry and a
682 standard deviation of ~30%. As a result, any dose recovery measurement close to saturation is biased
683 and accompanied by a risk of incorrectly accepting/rejecting a sample as suitable for dating.

684 The dose recovery ratio or luminescence recovery ratio is an average sample descriptor, which cannot
685 be applied to the individual aliquots used for dose measurement. The reason for this is that the given
686 dose must be administered before any preheating, but samples must be preheated before measurement
687 of the natural signal. The dose recovery ratio describes the suitability of the chosen SAR protocol to
688 measure a known laboratory dose given to a particular sample prior to any thermal treatment, but it does
689 not establish whether the chosen SAR protocol will measure a dose equivalent to the natural dose
690 accurately; this remains an assumption. To this end, Guérin et al.⁹² were unable to establish a correlation
691 between dose recovery ratios and the ratio of OSL ages to independent ages. In the final analysis, the
692 accuracy of OSL ages can only be tested by dating material of known age.

693 [H3] Aliquot specific rejection criteria

694

695 Once a suitable SAR protocol has been identified, it is used to measure the D_e in many aliquots. Not all
696 measurements may be of equal value, and criteria based on recycling ratios, recuperation, absence of
697 effect of prior infra-red stimulation on OSL (known as OSL-IR depletion ratios, see **Box 3**⁹³) have all
698 been suggested to identify less reliable values⁷⁸. However, the use of any such criteria should be
699 justified. By normalising doses from each individual aliquot to the sample mean, the correlation
700 between recycling ratio, recuperation and OSL-IR depletion ratio, and D_e can be examined across a set
701 of samples, each with a different average D_e ^{91, 94}. This correlation is done for 54 loess samples in **Fig.**
702 **8a,b**, and for these samples at least, there is no justification for aliquot-specific rejection based on either
703 poor recycling ratios or significant OSL-IR depletion ratios. Perhaps surprisingly, this is not unusual⁹⁵⁻
704 ⁹⁹. To this end, data should only be considered for rejection if there is evidence to support that doing so
705 improves the average dose estimate. However, identifying appropriate rejection thresholds to identify
706 and reject individual aliquots from a single sample may not always be possible. Experimentally, the
707 aliquot-to-aliquot over-dispersion in these measurables can be considerable, such that in comparison
708 the individual estimates of uncertainty on each aliquot are often small.

709 Quartz dose response curves vary in curvature even within a sample, and some aliquots saturate at
710 lower doses than others¹⁰⁰⁻¹⁰⁴; this is particularly true for individual grains. For older samples with
711 larger natural doses or when larger given doses are used in dose recovery tests, some aliquots cannot
712 be used to measure the dose of interest because D_e in Equation 6 is too small. In the worst case, said
713 aliquots that cannot be used will not give estimates of D_e . In practice, this leads to unbounded
714 estimates of D_e and/or uncertainty for a number of aliquots, and discarding these values leads to bias.

715 To avoid this biasing of the D_e distribution towards low dose estimates, aliquots can be sorted by their
716 ability to record large doses or by increasing D_e values (**Fig. 8c**), with aliquots only with increasingly
717 larger D_e values selected^{91, 94, 105-107}. As D_e increases, the fraction of aliquots in saturation becomes
718 negligible and the estimate of the central tendency of the equivalent dose or given dose distribution
719 becomes stable.

720 **[H1] Applications**

721 This primer is not intended as a comprehensive review of the OSL application literature, as several such
722 reviews are already available¹⁰⁸⁻¹¹⁴. Here, we present illustrative examples of actual and potential
723 applications. Many of these applications were pioneered using thermoluminescence and **IRSL** on both
724 quartz and feldspar, and include the dating of pottery¹¹⁵, loess¹¹⁶, colluvium^{117, 118}, archaeological
725 sediments¹¹⁹, periglacial features¹²⁰, fluvial, marine and aeolian systems¹²¹⁻¹²³, direct dating of tectonic
726 faults¹²⁴, soils¹²⁵ and glacier ice¹²⁶.

727 **[H2] Quartz OSL in archaeo- and geochronology**

728 Quartz grains must give a measurable fast-component OSL signal in response to ionising radiation for
729 reliable dating. Unfortunately, not all sources of quartz have these characteristics. Igneous rocks are the
730 main source of quartz grains in sedimentary deposits, but grains extracted directly from primary rock
731 do not usually show significant luminescence sensitivity^{63, 127}. Consequently, sediments in mountain
732 catchments dominated by primary rocks rarely give a measureable fast-component signal⁶³. Sensitivity
733 seems to develop during repeated cycles of erosion, transport and deposition¹²⁸ and laboratory studies
734 support this inference^{63, 129, 130}. Thus repeatedly reworked quartz grains, such as those in large-scale low-
735 gradient river systems or in deserts, almost always have a strong luminescence response to dose. Once
736 sensitivity has been confirmed, any dating application must also consider: the reliability of the chosen
737 SAR protocol; the degree of resetting by daylight or heat before burial; and the dose rate during burial.
738 Below we review some illustrative dating applications in this context, and **Table 1** summarises different
739 events that can be constrained in time using quartz OSL.

740 In fired or naturally-heated samples, the luminescence signal follows **Arrhenius** kinetics, where
741 exponential depletion depends on both time and temperature¹⁷. Typically, heating for one hour at 250 °C
742 or one minute at 300 °C removes 99% of the fast-component OSL⁵⁷. Such heating conditions are
743 commonly exceeded in the firing of pottery, ceramic bricks and tiles^{131, 132}, in heated stones^{133, 134} and
744 lithics¹³⁵, in sediment heated under archaeological hearths¹³⁶, and in contact with lava flows¹³⁷.

745 Local temperatures can also be raised a few hundred degrees by frictional heating associated with
746 formation of fault gouges during tectonic movement, or dike injection due to earthquakes, completely

747 resetting OSL¹³⁸⁻¹⁴⁰. Lightning strikes can cause localised melting in dry sand, creating fulgurites and
748 resetting nearby grains¹⁴¹.

749 Since daylight exposure is ubiquitous (Figure 1), most sedimentary settings can be dated using OSL
750 (Figure 9). The bleaching rate depends on both the daylight flux and spectrum^{31, 142}. Under water, the
751 depth and suspended particle load reduce photon flux, especially at shorter wavelengths, and turbulence
752 carries particles into and out of the illuminated zone; all these processes reduce the bleaching rate. Sub-
753 aerial transport is thus more effective at resetting OSL than sub-aqueous. Nevertheless, dating is often
754 possible in completely dark sub-glacial, cave, deep lake¹⁴³ and deep ocean environments^{5, 144} because
755 the sediments were sufficiently exposed to daylight before last entrainment.

756 [H2] Landscape evolution and past environments

757 The rapidity of daylight bleaching implies that usually only the most recent transport and deposition
758 event is dated using OSL. Applications in landscape evolution and palaeo-environmental studies are
759 numerous (**Table 1**) and have resulted in major advances in understanding a variety of geomorphic
760 processes, including thresholds, leads and lags¹⁴⁵⁻¹⁴⁸. OSL dating of a wide range of terrestrial sequences
761 has also demonstrated the interrupted nature of terrestrial records^{95, 149}. Many deposits record only short
762 and sub-millennial periods of time, often when the climate is changing rapidly from one relatively stable
763 state to another¹⁵⁰⁻¹⁵³. These results have raised questions about the validity of past land-sea and land-
764 ice correlations that were usually based on the assumption of continuous deposition in the absence of
765 numerical chronologies¹⁵⁴⁻¹⁵⁶. OSL dating has changed our view of the Weichselian ice limits in
766 northern Eurasia (QUEEN project^{157, 158}) and the retreat of the British-Irish ice sheet (BRITICE-
767 CHRONO project^{159, 160}). It has also provided one of the foundations of the INQUA Dunes Atlas, a
768 global chronological database on periods of inland or continental sand dune accumulation and
769 stabilization^{161, 162}. Dating of frost wedges has demonstrated landscape stability over an entire glacial
770 cycle¹⁶³ and constrained palaeotemperatures. In addition, OSL dating of sediment covering faults has
771 enabled the constraining of the timing of past earthquakes¹⁶⁴⁻¹⁶⁷.

772
773 OSL is also reset by daylight exposure during the vertical reworking of grains by bioturbation (including
774 the formation of biogenic structures such as termite mounds, **Fig. 9e**), pedoturbation and cryoturbation
775 ¹⁶⁸⁻¹⁷³. These additional reset mechanisms have led to new research possibilities of relevance to the
776 Anthropocene (Table 1), particularly in understanding critical zone processes and rates of sediment
777 turnover^{174, 175}. In a palaeosol developed in a sediment, turbation continually reset grains during soil
778 development and ceased when the surface was buried. Thus grains in the subsoil below the mixing zone
779 are expected to record the time the sediment was deposited, and the now buried topsoil records the time
780 soil formation stopped¹⁷².

781

782 All of these processes are climate-driven, or at least climate-dependent. Establishing the absolute timing
783 and rates of change of such landscape development is thus key to identifying periods of climate change,
784 and to establishing the correlations between these local changes and the global climate record^{95, 176, 177}.
785 Luminescence is particularly useful among the techniques that can be used to establish such
786 chronologies because it targets the silt and sand grains, which are present in all landscapes.

787 [H2] Archaeology and migration studies

788 Quartz OSL dating has made significant contributions to Holocene archaeology, including dating of
789 Neolithic settlements^{178, 179}, Archaic mound complexes in the Americas¹⁸⁰, Bronze and Iron Age
790 middens¹⁸¹, rock art^{182, 183}, medieval bricks¹⁸⁴, mortar¹⁸⁵ and stone surfaces¹⁸⁶⁻¹⁸⁸. On longer time scales,
791 the diverse sedimentary environments dated using OSL also served as habitats for past human and
792 faunal populations. As a result, quartz OSL is the preferred dating tool in many anthropological and
793 palaeontological applications (**Table 1**). This preference holds despite the greater precision of other
794 techniques such as argon-argon or U series dating. In contrast to OSL dating, argon-argon or U series
795 dating are very restricted in application (for example, argon-argon dating targets infrequent volcanic
796 **ejecta**), and even if applicable, usually only give a small number of ages in a section.

797 OSL dating has made major contributions to debates on the timing of human evolution and migration
798 and on changes in palaeoecology, especially for events beyond the limit of radiocarbon dating (~45 ka).
799 Important results include timelines for Out-of-Africa dispersals of *Homo sapiens*¹⁸⁹⁻¹⁹⁴, the earliest
800 known human settlement of Australia¹⁹⁵⁻¹⁹⁸ and possibly of the Americas^{199, 200}, occupation dynamics of
801 Neanderthals and Denisovans^{201, 202}, key evolutionary, behavioural and cultural turning points across
802 different human species²⁰³⁻²⁰⁶, and the late Pleistocene megafaunal extinctions in Australia and the
803 Americas²⁰⁷⁻²¹⁰.

804 [H1] Reproducibility and data deposition

805 Complexities in OSL dating arise from four main factors: limited net daylight flux causing some grains
806 to not completely reset at deposition; different OSL signals from different grains in response to the same
807 radiation, heating and optical stimulation; different radiation fluxes to different grains because of
808 heterogeneity in mineralogy, grain size and burial matrix; and open system deposits leading to time-
809 dependent radiation fluxes because of post-depositional mixing, geochemical and diagenetic processes,
810 and variable water contents.

811 Limited net daylight flux leads to heterogeneity in the apparent D_e . Different OSL signals from different
812 grains lead to heterogeneity in the apparent D_e , and both different radiation fluxes to different grains
813 and open system deposits lead to heterogeneity in dose rate and thus in natural dose. All of these factors
814 affect the ease with which OSL dating can be applied in different settings (summarised in **Fig. 10**).

815 [H2] Heterogeneity in D_e

816 The major factors affecting the complexity of determining D_e are discussed below. Their relative
817 significance is summarised in **Fig. 10a** shows that, for example, aeolian sediments are likely to show
818 low complexity (furthest from the centre), because they tend to be well bleached (red band) and have
819 good luminescence characteristics (dark blue band), both with low variability (width of the band). In
820 contrast, glacial sediments are likely to show high complexity (close to origin), because they tend to be
821 poorly bleached and have poor luminescence characteristics with considerable variability (width of red
822 and dark blue bands). Bleaching becomes progressively less important and contributes less to variability
823 as the burial age increases. The complexity arising from bleaching therefore decreases with age,
824 whereas complexity from luminescence characteristics is at a maximum for oldest sediments, mainly
825 because of dose saturation effects, and a minimum for medium age material, where signals are strong
826 and the saturation effects are small.

827 The nature of grain transport by wind or water implies that every grain is exposed to a different daylight
828 flux and spectrum. Thus, for short transport duration under attenuated daylight, the latent OSL signals
829 from different grains are reset to different degrees, and only when the exposure is of sufficient duration
830 will all grains be fully reset. Field environment and age are the major factors affecting the completeness
831 of bleaching, and they dictate the position and width of the red band in **Fig. 10a**. In general, given the
832 wavelength-specific attenuation in a water column, and the wavelength dependence of bleaching, the
833 grain-to-grain variability of bleaching in water-lain sediments is greater than in aeolian sediments.
834 Nevertheless, some flood deposits are surprisingly well-bleached^{211, 212} suggesting that significant
835 bleaching occurred before fluvial entrainment or sub-aerially during low flow.

836 If all grains are fully bleached and the dose rate is homogeneous, the D_e distribution of a sample should
837 be centred on a single value with dispersion accounted for by random measurement uncertainties^{48, 213,}
838 ²¹⁴. Unfortunately, this is rarely, if ever, the case. Additional dispersion in D_e originates from differences
839 in the behaviour of OSL signals from different grains, and this compromises both the accuracy and
840 precision of the chosen SAR protocol (see the dark blue band in Figure 10a, which represents intrinsic
841 sources of dispersion). These difficulties arise because the luminescence from different grains responds
842 differently to radiation, heating and optical stimulation^{91, 104, 106, 215, 216} or because of different charge
843 transfer properties and different OSL signal compositions^{59, 217-219}. In addition, the process of
844 interpolation onto the dose response curve (**Fig. 7**) will give rise to an asymmetric dose distribution
845 with an increased dose-dependent dispersion compared to that of the OSL distribution. At the limit, a
846 fully saturated sample gives an incorrect, apparently bounded, dose distribution (**Fig. 7**), with the
847 potential to be misinterpreted as a poorly bleached distribution or as mixture of a finite dose with a
848 saturated sample.

849 In addition to complications arising from these intrinsic factors, D_e dispersion also arises from extrinsic
850 sources such as variations in bleaching, mixing, and dose rate. When dispersion in D_e is significant,
851 complex statistical analysis may be necessary^{77, 213, 220-225} to extract the dose component of interest. It is
852 not straightforward to differentiate between intrinsic and extrinsic sources of dispersion because we do
853 not have a way of independently measuring the extrinsic component. As a result, any dispersion greater
854 than that determined in laboratory experiments is simply assumed to be extrinsic⁵³. However, there are
855 cases where this assumption is incorrect⁹⁴.

856 Although the size of the apparent residual dose remaining after bleaching is independent of the
857 subsequent burial time, this burial timing affects the residual dose by affecting ‘bleaching’ (see red band
858 in Burial age in **Fig. 10a**). There are three approaches to investigating whether a sample is likely to have
859 been sufficiently bleached at deposition: first, modern analogue samples²²⁶ provide site-specific
860 evaluations of bleaching conditions^{111, 227, 228}, which assume that the net effects of transport and
861 deposition on bleaching have not changed through time. In general, modern analogues have not
862 completed their transport at the time of collection and because most recently deposited sediment is held
863 in ephemeral storage such as point bars, it is likely to be reworked again before final deposition in long-
864 term storage. Thus modern analogues will tend to underestimate the degree of bleaching available in
865 the environment²²⁶. The second approach involves examining the self-consistency of OSL dating
866 through measurement of different minerals with different luminescence signals. For example, paired
867 dating comparisons of quartz OSL and K-rich feldspar IRSL or post-IR IRSL, where the two feldspar
868 signals are optically reset at different rates, can be used to confirm that all quartz grains in a sample
869 were completely bleached prior to deposition²²⁹⁻²³¹. Finally, the third method uses single-grain OSL
870 dating to examine heterogeneity in D_e in sediments from environments with likely limited daylight
871 exposure or post-depositional alteration^{114, 232}. In such cases, heterogeneity in radiation dose rate at the
872 individual grain level must also be considered (see below).

873 Unfortunately, analytical models based on the widely-used Central Age Model (Galbraith et al., 1999;
874 such as the Minimum Age Model (Ref. Galbraith et al. 1999) and Finite Mixture Model (Roberts et
875 al., 2000)) do not take this dose rate heterogeneity into account and so risk producing biased estimates
876 of D_e (Guérin et al., 2017, QG).

877 This can be illustrated by considering a hypothetical sample in which the distribution of dose-rates
878 dominates the natural dispersion. Suppose this to be a well-bleached, well-behaved sample in which the
879 uncertainty on the measurement of dose is negligibly small (e.g. 1%). All grains must by definition
880 record the same age. Suppose this sample is 10 ka old, and that the environmental dose rate is made up
881 of two equal but spatially separated (at the grain scale) contributions, one of 1Gy.k^{-1} , and one of 10
882 Gy.k^{-1} ; we would measure an average dose rate of 5.5Gy.k^{-1} . Now consider the dose - on average,
883 50% of the grains absorb a burial dose of 10 Gy, and 50% a dose of 100 Gy. If these two doses could
884 be measured individually, each with some similar overall uncertainty, then the unweighted average dose
885 would be 55 Gy, the geometric mean 31.6 Gy and the weighted average 10.9 Gy. Only the average dose

886 gives the correct age of $55/5.5 = 10$ ka; the geometric mean age is $31.6/5.5=5.7$ ka, and the weighted
887 data give $10.9/5.5=2.0$ ka.

888 Of course such simple bi-modal distributions do not exist in nature, but the point remains – when only
889 an average dose rate is known, and the dose rate is a major factor in dispersion, the dose must also be
890 an average, or the age estimate is likely to be biased. This is particularly true of large aliquots, where
891 the dose is in any case averaged as a result of the large number of grains giving light in each aliquot,
892 and analytical uncertainties on dose estimates from each aliquot are very much smaller than the data
893 dispersion. This point is made in Guerin et al. (2017), where the implications for the analysis of dose
894 distributions are discussed in detail.

895

896 Site stratigraphy and grain sorting are of considerable importance to reliable OSL dating (**Fig. 10a**).
897 Sediments affected by significant post-depositional mixing via, for example, bioturbation, freeze/thaw
898 cycles, tectonic activity, or cultivation should be avoided as these processes can lead to significant
899 dispersion in D_e values, but sampling potentially-mixed sediments may be unavoidable at times. In
900 some cases this mixing is obvious but not always, and the presence of distinguishable components in
901 small-aliquot or single-grain dose distributions has been used to infer mixing^{233, 234}. These dose
902 populations can be modelled as a mixture of discrete components using the Finite Mixture Model²³⁵,
903 and typically the dominant component is chosen to provide the deposition age^{234, 236-239}. However, such
904 analyses can imply unrealistic degrees of physical mixing between non-adjacent layers even in deposits
905 where mixing can safely be ruled out⁹⁴, suggesting that the distributions identified by dose mixing
906 models may not necessarily arise from physical mixing²⁴⁰. Together with the inability to derive
907 component-specific dose rates for mixed sediments (see below), this calls into question the application
908 of dose mixing models for dating purposes

909

910 [H2] Heterogeneity in dose rate

911 The major factors affecting dose rate complexity are discussed below, and their relative importance is
912 summarised in **Fig. 10b**.

913 Dose rate evaluations become demanding when the infinite matrix assumption is no longer applicable
914 ⁵² (red end of the ‘Dimension of deposit’ bar in **Fig. 10b**). Sand-sized quartz grains have a scattered β
915 spectrum range comparable to that in sediments; the larger the grain, the higher the attenuation of the
916 external β dose rate (Supplementary Table 4). In addition, the distribution of radionuclides at the grain
917 scale in sediments is spatially heterogeneous with variations in both internal and surface-bound
918 radioactivity. Thus ‘hotspots’ of higher radioactivity, such as K-feldspar and zircons, or ‘cold spots’ of
919 lower radioactivity, such as carbonate nodules, result in significant variations in the β dose rate to
920 individual grains. This spatial variability can be observed^{135, 241, 242}, modelled and, on average, accounted
921 for^{53, 54, 243}, but the association of spatially-varying β doses with individual grains from which a D_e has

922 been estimated is currently not possible. Such variability is usually lower in a well-sorted deposit (green
923 end of ‘Sorting’ in **Fig. 10b**).

924 Heterogeneities on a scale of tens of cm affect the γ ray dose rate, which may result from the proximity
925 of a sample to a large rock fragment of a different mineralogy, or from interfaces between different
926 stratigraphic units (see Appendix H in¹⁷). While in many cases these heterogeneities can be avoided,
927 the choice of sampling location is often restricted in archaeological sites. In that case, the γ dose rate to
928 samples collected close to stratigraphic interfaces may require modelling²⁴⁴ or in situ γ dose rate
929 measurement using luminescence dosimeters or field γ spectrometers^{25-28, 30, 245, 246}.

930 Water content is particularly important (see **Box 1**) as it reduces the dose rate compared to a dry matrix¹⁷,
931 ²⁴⁷, and a 1% change in water mass fraction typically results in a 1% change in apparent OSL age. Thus,
932 a low value for ‘Saturated water content’ (**Fig.10b**) reduces the possible range of water contents during
933 burial, and therefore reduces the dependence on assumptions concerning ‘Fraction of saturation’ of
934 water content (**Fig. 10b**). Whatever the saturated water content, if it is likely that for the entire burial
935 period the sample has either been dry, for example desert sand, or fully saturated, for example below
936 the water table, then uncertainties arising from assumptions are much reduced. The least desirable
937 condition is a sample in a temperate climate vadose zone, where the average fraction of saturation
938 throughout the burial period is poorly defined (see red centre of band in **Fig. 10b**). In such cases, there
939 may be no alternative to assuming 50% of saturation with some appropriately large uncertainty on that
940 value.

941 The dose rate is also affected by temporal changes^{55, 248}. For instance, post-depositional changes in
942 burial depth can be important for shallow burial (see red end of ‘Overburden thickness’ in **Fig. 10b**).
943 Changes due to erosion or deposition increase complexity by affecting both γ and cosmic ray dose
944 rates¹⁶, but are likely to be less important for deep burial (see green end of band in **Fig. 10b**). Density
945 changes due to porosity alone do not change dose rates because the density of air is too low to absorb
946 significant amounts of energy. However, density changes arising from time-dependent carbonate
947 infilling/leaching of pore space, or oxidation of organic material, change the effective dose rates and
948 the saturated water content (**Fig. 10b**)^{249, 250}, which directly and indirectly increases complexity (see
949 Supplementary Tables 5-6 for correction factors).

950 The dose rate may also change because radionuclides are imported/exported from a volume of sediment.
951 Radionuclide concentrations are affected by water movement, which may import specific radionuclides
952 into secondary minerals or leach them by reduction and/or hydrolysis with time, particularly ⁴⁰K, ²³⁸U,
953 and ²²⁶Ra (a shorter-lived member of the ²³⁸U decay series^{21, 23}). This import/export leads to
954 disequilibrium in the ²³⁸U decay series and possibly time-dependent dose rates²⁵¹. In addition, the 3.8
955 day half-life of the noble gas ²²²Rn (²³⁸U series) allows migration through sediment pores before decay.

956 Diffusion dominates for partial pore-filling by water, but bulk transport can occur if water moves
957 through a deposit²⁵², with losses of 50% at 5 m depth in sand having been reported²⁵³. Although the
958 post-²²²Rn part of the ²³⁸U decay series contributes the majority (>75 %) of the β and γ dose rate from
959 the ²³⁸U series (Supplementary Figure 3), for a typical sandy sediment containing 20 Bq.kg⁻¹ ²³⁸U and
960 ²³²Th and 300 Bq.kg⁻¹ ⁴⁰K (1.5 and 5 ppm, and 1% respectively), the contribution to the total dose rate
961 is <20%. A combination of thermal ionisation mass spectrometry and high-resolution γ spectrometry
962 can be used to describe the state of equilibrium of, for example, the ²³⁸U decay series, but in most cases,
963 disequilibria have rather small effects on the OSL age.

964 Finally, any significant ‘post depositional mixing’ (see red end of bar in **Fig. 10b**) increases the
965 challenge of determining a time averaged dose rate. In a mixed deposit, grains will have absorbed dose
966 in potentially different dose rate environments before mixing, but only the mixed dose rate is
967 measurable today. Without knowledge of the dose rates in the source deposits contributing to the
968 mixture and of the timing of the mixing, reconstruction of an average dose rate is uncertain.

969 In summary, dose rates at the grain level are likely to be variable during burial. This variability must be
970 taken into account when using the age equation, and accurate ages should be calculated using a model
971 which delivers average estimates of the absorbed doses during burial (Guérin et al., 2017, QG).

972 [H2] Checking reproducibility

973 One way to reduce the impact of the various methodological issues discussed above is through internal
974 and external checks on reproducibility. These may, for example, involve collecting a suite of OSL
975 samples targeting different types of sediments preserved in clear stratigraphic relationships, or by
976 collecting replicate samples from the same horizons^{95, 99, 198}. Although resource-intensive, multiple-
977 technique and multiple-sample OSL dating strategies are recommended in all dating applications^{95-97,}
978 105, 135, 198, 201, 231.

979 Statistical uncertainties associated with individual laboratory measurements -conventionally given at
980 68% confidence intervals in luminescence dating- are typically 2-3%, both on natural dose
981 measurements involving OSL and on dose rates (as determined from γ spectrometry). These random
982 uncertainties can be reduced by increasing the number of measurements, but this requires a considerable
983 increase in instrument time per sample and is often impractical. Studies involving high-density sampling
984 from large stratigraphic sections frequently report over-dispersed ages, where the ages are more variable
985 than expected from analytical uncertainties alone (see **Fig. 11a**⁹⁵). This suggests that reductions in
986 measurement uncertainty would be of limited benefit. Note that Figure 11a does not include the
987 systematic uncertainties common to all the ages in the section, and that the error bars represent random
988 uncertainties only.

989 The stated uncertainties on calibration standards are also ~2-3%, both for the γ -irradiated quartz⁴⁷ used
990 to calibrate laboratory β sources and for the geochemical standards used for the calibration of equipment
991 used in dose rate determination²⁵⁴. Together, these contributions result in typical total calculated
992 uncertainties (random and systematic) on an OSL age ranging 5-10% (Supplementary Table 7).
993 Currently, 5% is considered a minimum uncertainty for a sample in an ideal setting and >10% for
994 unfavourable cases involving, for instance, low luminescence sensitivity or saturation of OSL with dose.

995 Perhaps because OSL dating is a relatively young method, with the present methodologies dating back
996 to the 2000s and most laboratories small with fully committed instrumentation, the community has yet
997 to develop a routine of inter-laboratory comparisons. The only exception is an exercise in which 26
998 different laboratories dated a beach-ridge sand selected for its good OSL behaviour²⁵⁵. The dispersion
999 in the 26 ages (~18%) significantly exceeded the uncertainties on individual ages as estimated by the
1000 various participants. Some of the dispersion arose from different assumptions: for example, the use of
1001 a 'typical' internal dose rate to quartz by some, while others ignored this possible contribution. In this
1002 study, it is clear that the community, as a whole, underestimated the uncertainties they assigned to
1003 individual age estimates.

1004 In addition, laboratories make use of either their own age calculation software or one of the more widely
1005 available packages, such as DRAC²⁵⁶, RLuminescence²⁵⁷ or numOSL²⁵⁸. These software all include
1006 underlying, often dissimilar, default assumptions, and a rigorous comparison of these packages is highly
1007 desirable but not yet established.

1008 Until recently most articles containing luminescence ages presented new data with a variable degree of
1009 detail. However, publications addressing larger scale questions and requiring meta-analyses of
1010 published datasets are becoming more frequent. It is thus increasingly important that all published OSL
1011 ages are accompanied by underlying data of sufficient detail to allow the reader to reproduce the age
1012 calculations and reliably compare ages from different laboratories. An example of the minimum sample
1013 specific information necessary for such calculations is given in **Table 2**. There should also be text
1014 describing the experimental parameters and the results of any additional performance tests, especially
1015 dose recovery experiments. We hope that all journals, even those without specific interests in the
1016 technicalities of dating, will encourage authors to publish such information.

1017 **[H1] Limitations and optimizations**

1018 The problems encountered in quartz OSL dating often depend on the age of the sample. The ability to
1019 date young samples with small doses depends on the sensitivity of the luminescence signal to radiation.
1020 For less sensitive quartz, it can be difficult to detect a measurable fast-component signal above the
1021 instrument's background and slower decaying OSL signals. Thus dating young desert dunes is much

1022 more likely than dating young mountain-valley slope deposits (see **Fig. 10a**). Also, thermal transfer can
1023 usually be avoided by a low temperature preheat (**Fig. 6b**).

1024 For older sediments, it may take weeks of instrument time to collect the data required for a single age,
1025 which is a major restriction for laboratories with limited instrumentation. Standardised dose-response
1026 curves are sometimes suggested to reduce this measurement time¹⁰⁰. For large suites of samples with
1027 similar properties such as, for example, a loess section, the average responses of multi-grain aliquots of
1028 quartz follow a common dose-response curve as demonstrated by examining a sub-set of the suite²⁵⁹.
1029 In such cases, individual L_n/T_n values can be interpolated on to this common dose-response curve to
1030 determine individual D_e . This approach is potentially useful for younger, homogeneous samples within
1031 the linear part of the dose-response curve^{100, 260, 261}. However, there can be significant reproducibility
1032 issues with less homogeneous material, and particularly as the dose-response curve approaches
1033 saturation.

1034 More generally, the apparent behaviour as the laboratory dose response curve approaches saturation
1035 may be misleading. The controls on luminescence saturation with dose are poorly understood. The
1036 luminescence from individual grains and sometimes from multi-grain aliquots can saturate at very
1037 different doses, continue to grow without apparent saturation, or even decrease at high doses^{101, 262}.
1038 However, quartz often underestimates the true age at large doses (150-250 Gy)^{64, 66, 263} and it seems
1039 likely that at these large doses the laboratory dose response curve does not reflect the natural dose curve
1040^{65, 264, 265}. One way to address this very important question is to investigate natural dose response curves
1041 using homogeneous material from sections with a well-known age model, such as some well-studied
1042 loess sections^{65, 266}. New measurement strategies and statistical tools have also been proposed with the
1043 aim of increasing the range of single-grain OSL^{267, 268}.

1044 [H2] Overall accuracy

1045 The overall performance of sand-sized quartz OSL to date samples from a variety of deposition
1046 environments, each with independent age control, has been evaluated by others^{89, 108, 269}. These data sets
1047 are combined in **Fig. 11b**, for 249 ages there is now a considerable body of data suggesting that OSL
1048 dating is accurate over a time span of more than four orders of magnitude (**Fig. 11b**), and that the
1049 precision and accuracy are largely in the range originally predicted.

1050 [H1] Outlook

1051 Over the last twenty years, dating based on the quartz fast-component OSL signal has become an
1052 important tool in the geochronology of the past 500 ka. The method is still undergoing rapid
1053 development and improvement and exciting new areas of application are waiting to be explored.

1054 The origins of intrinsic over-dispersion in D_e , both at the multi- and single-grain level^{148, 232}, begs further
1055 investigation. An improved understanding and quantification of these intrinsic sources would, in turn,
1056 enable more reliable quantification and interpretation²⁴² of the contribution to dispersion from extrinsic
1057 sources, such as incomplete bleaching and mixing. Limits on the upper dose range of the OSL signal
1058 are also poorly understood. Laboratory dose response curves often increase to large doses (hundreds of
1059 Gy), but natural doses either do not increase above ~200 Gy, or if they do, they often appear to be an
1060 underestimate^{66, 263, 270}. An improved understanding of the fast-component dose response curve at high
1061 doses would increase confidence in older ages and help to extend the datable range. Other quartz signals
1062 such as Violet Stimulated Luminescence^{7, 271} and Thermally Transferred OSL²⁷²⁻²⁷⁴ also have the
1063 potential to date older material, but both require further research.

1064 Accurate calibration of laboratory radiation sources is an active research area^{47, 275} and a regular
1065 programme of radiation source comparisons is desirable.

1066 Addressing these issues should help improve luminescence age accuracy to the probable minimum of
1067 ~5%. As the demand for OSL dating continues to grow, the demonstrated benefits of a high stratigraphic
1068 age density to site interpretation^{148, 198, 276-278} will only increase this demand further.

1069 Single grain dating has demonstrated its potential for dating incompletely bleached deposits^{213, 225, 279,}
1070 ²⁸⁰ but inherent dosimetry uncertainties likely prevent the accurate dating of deposits subject to
1071 significant post-depositional mixing⁷⁷. For unmixed deposits, the ability to determine dose rates specific
1072 to the individual grains giving a D_e would likely produce a step change in interpretation and application.

1073 Field measurements of OSL have been demonstrated²⁸¹⁻²⁸³ and the ability to determine approximate
1074 ages 'on site' would be valuable. Although there is no technical impediment, calibrating the signal in
1075 terms of dose requires a portable ionising radiation source, which has regulatory barriers to overcome.

1076 Direct dating of buried rock surfaces is rapidly growing^{127, 284}, although quartz applications are limited
1077 to sandstones and quartzite clasts because of sensitivity. One advantage of direct dating is that a rock
1078 surface records its history of daylight exposure, which eliminates the uncertainty in whether it was
1079 sufficiently bleached^{187, 188, 285, 286}. The potential applications in both geology for cobble fans, moraines,
1080 scree/talus, and rock falls, and in archaeology for megaliths, field boundaries, buildings, and lithics, are
1081 considerable. Interest in the inverse problem of dating the length of daylight exposure of a rock surface
1082 is just beginning^{127, 287} and currently requires a known-age calibration surface^{15, 287}. However, the
1083 potential of this method is considerable, especially in archaeology.

1084 Finally there is considerable opportunity to use the signal remaining after daylight exposure to
1085 investigate geomorphological processes, such as the relative importance of supra/sub-glacial
1086 transport²⁸⁸ or fluvial residence time²⁸⁹.

1087 OSL dating of quartz continues to expand its fields of application beyond those mentioned in this
1088 Primer. The method continues to develop, and will undoubtedly contribute further to our understanding
1089 of Earth surface processes.

1090

1091

1092 Glossary

1093 (Some definitions are specific to the context of this Primer. Where appropriate, units are given in
1094 square brackets, e.g. [s^{-1}])

1095 The **Arrhenius** equation states that, for an electron to escape a trap, it must first acquire a minimum
1096 amount of energy, called the activation energy E_a . Then the rate of thermal eviction of trapped
1097 electrons is given by $Ae^{-\frac{E_a}{k_B T}}$. The ‘frequency factor’ A can be seen as the number of attempts to
1098 escape made by the trapped electron, and $e^{-\frac{E_a}{k_B T}}$ as the probability that any given attempt will result in
1099 an escape at a particular absolute temperature T . k_B is Boltzmann’s constant.

1100 **α particles:** ionising radiation in the form of energetic helium nuclei produced by nuclear decay.

1101 **Anthropocene:** a proposed geological epoch covering the period of significant impact on Earth’s
1102 geology and ecosystems by humans.

1103 **Ar-Ar (or K-Ar) dating:** a dating technique based on the decay of ^{40}K to ^{40}Ar .

1104 **β particles:** ionising radiation in the form of energetic electrons produced by nuclear decay.

1105 **bioturbation:** after initial deposition, the reworking of sediment by biota (plants, animals, etc).

1106 **Bq:** Becquerel, a nuclear disintegration per second. [s^{-1}]

1107 **cal BP:** calibrated ^{14}C years before present (referred to AD1950). Only used for ^{14}C ages, after
1108 conversion to calendar years by calibration. [a]

1109 **Central Limits theorem:** states that the sum of independent random variables tends toward a normal
1110 distribution, even when the original variables themselves are not normally distributed

1111 **cosmogenic radionuclide dating:** any one of several methods to date length of exposure of minerals
1112 at the surface (or sometimes length of burial) using the build-up or decay of radioactivity induced by
1113 cosmic rays.

1114 **coversand:** a continuous layer of sand deposited by wind, often in a coastal environment.

1115 **crotoquina:** An animal burrow that has been filled with material from overlying sediments.

1116 **cryoturbation:** the reworking of sediment after initial deposition by the freeze/thaw cycle.

1117 **dark count rate:** the count rate from a photon detector completely obscured from light. [s^{-1}]

1118 **dose:** total amount of energy absorbed per unit mass from ionising radiation. [Gy]

1119 **dose rate:** the rate of energy absorption from ionising radiation during burial

1120 **D_c (or D_o (letter o)):** a constant used to describe the curvature of the luminescence response to dose,
1121 usually in an expression of the form $Y=A(1-\exp(-D/D_c))$. [Gy]

1122 **D_e :** equivalent dose, the laboratory dose that induces the same luminescence response as the natural
1123 dose. [Gy]

1124 **ejecta:** material thrown out by volcanic eruption.

1125 **fast-component:** the part of the signal that decays most rapidly during stimulation

1126 **feldspar:** a group of K-, Na- and Ca-rich aluminosilicate minerals, makes up ~40% of the Earth's
1127 crust.

1128 **frost wedges:** cracks formed in frozen ground, often filled with sand; this preserves them in the
1129 sedimentary record.

1130 **FWHM:** Full Width at Half Maximum, a numerical description of the width of a peak in data or
1131 response.

1132 **γ rays:** ionising radiation in the form of energetic photons produced by nuclear decay.

1133 **Gy:** Gray, energy absorbed per unit mass from ionising radiation. 1 Gy = 1 Joule/kg [J.kg⁻¹].

1134 **igneous rock:** formed by the solidification (crystallisation) of magma or lava.

1135 **IRSL:** Infra-red stimulated luminescence

1136 **ka:** thousands of years. [a]

1137 **λ :** lambda, the nuclide-specific probability of a nuclear decay. [s⁻¹]

1138 **leads and lags:** in climate science, the difference in timing between changes in two correlated climate
1139 phenomena (e.g. build-up of global ice and changes in regional dust deposition).

1140 **loess:** wind-blown dust (predominantly silt-sized (4-63 μ m) grains)

1141 **luminescence signal:** the luminescence response, L_i , to any dose, divided by the response, T_i , to a
1142 subsequent fixed test-dose, i.e. L_i/T_i

1143 **midden:** in archaeology, a refuse pile of domestic waste.

1144 **Neolithic:** the most recent division of the Stone Age (last ~12 ka), and associated with the
1145 development of farming.

1146 **optically stimulated luminescence – OSL:** the luminescence signal observed when stored charge is
1147 stimulated (evicted from traps) by visible or near-visible light.

1148 **overburden:** the material covering some geological feature of interest.

1149 **palaeosol:** a fossil soil (i.e. one no longer forming); usually found buried within a sequence of
1150 sedimentary units.

1151 **pedoturbation:** the reworking of sediment after initial deposition by the geochemical and biological
1152 processes of soil formation.

1153 **primary rock:** any rock that formed by solidification (crystallisation) from the melt.

1154 **Quantum efficiency:** how many photons are required to give a detected event, and the effectiveness
1155 of photon to electron conversion

1156 **quartz:** the most common natural form of the compound SiO₂, makes up about 20% of the Earth's
1157 crust.

1158 **radiocarbon dating (or ¹⁴C dating):** a dating technique based on the decay of ¹⁴C. Can only be
1159 applied to material of organic origin.

1160 **Reset:** in the case of luminescence, set to zero or 'bleached'

1161 **secular equilibrium:** where the production and decay rates of each nuclide in a decay chain are almost
1162 identical.

1163 **sedimentological boundaries:** strictly, distinct breaks (unconformities) between bodies of sediment.
1164 Often used more informally to describe gradational boundaries.

1165 **SAAD - Single Aliquot Added Dose:** a single aliquot procedure for determining D_e , by adding doses
1166 to the natural dose and measuring the growth of the resulting luminescence signal.

1167 **SAR - Single Aliquot Regenerative dose:** a single aliquot procedure for determining D_e , based on
1168 resetting the luminescence signal to zero between each measurement of luminescence, whether
1169 resulting from natural or laboratory doses

1170 **$\tau_{1/2}$ - half life:** The period of time taken for 50% of a particular radionuclide to undergo radioactive
1171 decay and change into another element. [s]

1172 **thermoluminescence:** the thermally stimulated luminescence signal observed when a sample is
1173 heated to evict stored charge from traps.

1174 **threshold:** In climate science, the point at which a small change in a physical or chemical parameter
1175 (e.g. global temperature) can lead to a tipping point in outcome and a shift to a new equilibrium state
1176 of range of average conditions (e.g. massive loss of global ice mass)

1177 **vadose zone:** in a porous body (e.g. of sediment), that part lying between the ground surface and the
1178 water table.

1179

1180 **Figures captions**

1181 **Figure 1: The origins of the luminescence dating signal in quartz.** (a) energy is absorbed (dose) as
1182 α and β particles and γ rays emitted during the decay of naturally occurring radionuclides (especially
1183 ^{238}U and ^{232}Th series, ^{40}K) penetrate quartz grains. The different ranges of the various emissions are
1184 indicated, relative to typical grain dimensions. Cosmic rays also contribute to the dose. (Redrawn
1185 from²⁹⁰) (b) Energy from ionising radiation is absorbed by an electron (e^- , filled blue circle), which
1186 migrates through the grain until it is trapped at a defect in the lattice structure (electron trap). The
1187 corresponding 'hole' (h^+ , literally the place where an electron should be, unfilled orange circle) also
1188 migrates until it is trapped in a different kind of defect (hole trap). This process stores energy. (c)
1189 Shining blue light on the crystal gives the trapped electron sufficient energy to escape the trap (optical
1190 stimulation). When it then recombines with the hole, it releases the trapped energy as a more energetic
1191 UV photon (optically stimulated luminescence).

1192 **Figure 2: The relationship of the erosion, transport and sedimentation cycle to the acquisition**
1193 **and loss of a luminescence dating signal** (a) any existing energy stored in the quartz crystal structure
1194 is released by daylight exposure (i,iii,v) during different erosion and transport events, by wind, water,
1195 ice, gravity or turbation (see (b)). These periods of resetting are interspersed with periods of burial
1196 (ii,iv,vi), during which dose is absorbed from natural radiation (Figure 1). Sufficient net daylight
1197 exposure completely removes the previously stored energy (e.g. during i and v), otherwise it is only
1198 partially reset (iii). This energy builds up again during the most recent burial (vi), until the quartz sand
1199 grains are sampled (vii) and taken to the laboratory. After cleaning, they are then exposed to blue light,
1200 and the luminescence signal acquired during last burial is measured. (b) illustrates the various transport
1201 agencies that can lead to daylight exposure of mineral grains before final burial (gravity acts as an
1202 independent transport agency only on high gradients).

1203

1204 **Figure 3: Sample preparation and instrumentation for OSL measurement.** (a) The laboratory steps
1205 used to extract quartz grains from a tube of sediment. Note the alternative processing routes, depending
1206 on whether feldspar is also required. All these steps should be performed under subdued lighting
1207 conditions, using a wavelength and intensity that avoids significant effect on the trapped charge
1208 population⁹. Q: quartz, H: heavy minerals, F: feldspar, NF: sodium-rich feldspar, KF: potassium-rich
1209 feldspar. The combined water drops [💧] and fan symbol [🌀] indicate thorough washing and drying.
1210 (b) The main components in an OSL measurement system. After irradiation in nature or in the
1211 laboratory, the quartz is heated to the desired temperature, and the stimulation light turned on. The
1212 photons making up the optically stimulated luminescence pass through the detection filter to the photon
1213 detector, to give the time dependent signal shown at the top.

1214 **Figure 4: Using SAR to determine the equivalent dose, D_e .** (a) T_i and (b) L_i plotted against dose for
1215 an aliquot of heated brick quartz; some T_i are labelled to indicate measurement order. (c) Sensitivity-
1216 corrected (L_i/T_i) growth curve obtained by dividing data from (b) by data from (a); inset, stimulation
1217 curves for natural signal and calibration quartz⁴⁷. (d) - (f) same as (a)-(c) but for quartz extracted from
1218 sediment. (Heated brick - 173701; monastery floor, Sweden. Sediment - 175405; Kalahari Desert,
1219 Namibia.) Open triangles show the response to $D=0$ Gy. Other open symbols indicate repeated dose
1220 measurements. Note that without sensitivity correction the repeated values of L (in b,e) are inconsistent
1221 with earlier measurements at the same dose; correcting for sensitivity change by dividing by T_i corrects
1222 this (in c,f). The interpolated doses (' D_e ' in b,e and D_e in c,f) and dose response curve shapes are also
1223 significantly different between the uncorrected and sensitivity-corrected data.

1224 **Figure 5: Correcting for sensitivity changes using the luminescence response to a test dose.** (a)
1225 The proportional relationship between L_i and T_i for a large aliquot (containing hundreds of grains) of
1226 180-250 μm quartz extracted from a fired brick (filled red circles, 19 cycles) and a quartz aliquot from
1227 a desert sediment (filled blue circles, 42 cycles), determined using the SAR sequence in (b). The natural
1228 signals (L_0/T_0) are shown as open circles. Same samples as in Figure 4; sediment: $D_i=44.4$ Gy, $D_T=5.9$
1229 Gy; fired brick: $D_i=3.6$ Gy, $D_T=1.0$ Gy. Both datasets are consistent with straight lines (dashed) going
1230 through the origin, indicating that the ratio L_i/T_i is constant for each sample. This is despite the
1231 considerable sensitivity change recorded by T_i (also shown inset as a function of measurement cycle,
1232 normalized to first test dose measurement). As is common, the heated quartz displays a greater
1233 sensitivity change during SAR measurements than the quartz extracted from sediment. (b) Typical SAR
1234 measurement sequence. In the first cycle, $i=0$ and D_0 is the natural dose, or the given dose in a dose
1235 recovery experiment. After completing a cycle (step 6 or 7), the sequence returns to step 1 as required.

1236 **Figure 6: Dependence of D_e and dose recovery ratio on thermal pretreatment** (see text for details).
1237 (a) Preheat plateaus for sand-sized quartz extracted from loess (D38141⁹⁵, coversand (OS-B⁹⁶) and
1238 heated sediment from a charcoal kiln (GLL-184301²⁹¹). For all three samples the D_e is independent of
1239 preheat temperature over a wide temperature interval (dashed line). (b) Thermal transfer plateaus for
1240 sand-sized quartz extracted from a fluvial sand (GLL-160301²⁹²) and a beach ridge (OSL43²⁹³). Quartz
1241 aliquots were bleached at room temperature twice using blue light stimulation to remove the natural
1242 OSL signal before measurement of the thermally transferred dose. The effect of thermal transfer
1243 becomes visible for preheats $\geq 200^\circ\text{C}$. (c) Dose recovery plateau for sand-sized quartz extracted from
1244 Chinese loess. The measured to given dose ratio is insensitive to preheat temperature over a wide
1245 temperature range. (d) Histogram of dose recovery results measured with a preheat temperature of
1246 260°C . Adapted from⁹⁹.

1247 **Figure 7: Effect of sublinear dose response on apparent dose distributions** (based on²⁹⁴). Monte-
1248 Carlo-modelled normalised OSL distributions shown on vertical axis, centred on 20% (black), 86%

1249 (blue), 95% (green) and 100% (red) of saturation, each assumed normal with a relative standard
1250 deviation of 5%. The resulting interpolations onto a dose response curve ($D_c=45$ Gy, solid pink) are
1251 given on the dose axis, with medians connected using intersecting lines. Each dose median is also shown
1252 in Gy, followed by the ratio of the median to the expected dose and the % of results giving a non-finite
1253 dose in parentheses; the arithmetic means and geometric means are all within 3% of these medians.
1254 Note that the dose median shown for 100% is the apparent value, ignoring non-intersecting OSL signals.
1255 The true median lies at infinity.

1256

1257 **Figure 8: Testing aliquot rejection criteria.** The relationship between D_e and recycling ratio (a) or
1258 OSL IR depletion ratio (b) for large aliquots (containing many hundreds of grains) of 90-180 μm quartz
1259 extracted from Chinese loess. Upper figures show the individual aliquot D_e values (normalized to the
1260 average D_e of each sample) as a function of recycling ratio and OSL IR depletion ratio. Lower figures
1261 show the recycling and OSL IR depletion ratios as histograms with mean ratios plotted as blue and red
1262 dashed lines, respectively. From the upper figures it can be seen that there is no relationship between
1263 the measured D_e and the recycling or OSL IR depletion ratio; rejection of aliquots based on recycling
1264 or OSL IR depletion ratios is not justified. Figure adapted from⁹⁵. (c) Effect of D_c filtering on grains
1265 with different D_c . The dose recovery data (red data, given dose 170 Gy) shows the effect on the dose
1266 recovery ratio of only accepting results from aliquots with increasingly larger D_c values. These data
1267 suggest that only those grains with $D_c > 125$ Gy give accurate results (red data). Even with this
1268 minimum D_c , ~3% of the accepted grains still give measured doses in saturation (black data). Sample
1269 from Covalejos cave (Northern Spain)²⁹⁵

1270 **Figure 9: Opportunities for bleaching of the OSL signal in different environments.** (a) Aeolian
1271 transport during dust storms (Pavliha-istockphoto.com). (b) fluvial transport (Marek Uliasz
1272 race.fit2paddle.com). (c) gravity-driven debris fans (unknown) (d) sediment transported by ice (Michael
1273 Hambrey - Glaciers online). (e) bioturbation - termite mounds (ourterritory.com).

1274 **Figure 10: Sources of complexity in estimations of dose and dose rate.** (a) The likely complexities
1275 in D_e determination arising from luminescence characteristics (dark blue band) and bleaching (red
1276 band), expressed qualitatively in terms of field observables and expected age (see [H1] “Reproducibility
1277 and data deposition” for details). These observables and estimates are shown on the 10 radial axes,
1278 grouped according to Depositional environment and implied transport mechanism (very light blue),
1279 Sedimentology (light blue) and the expected Burial age of the samples (medium blue). “Young” refers
1280 to quartz grains with expected D_e values of less than ~10 Gy, “Old” to quartz grains with D_e values of
1281 larger than ~130 Gy. Each axis is scaled from most complex (centre of the figure) to least complex
1282 (outer edge of figure). The likely variability of each feature is shown by the band width on a particular

1283 axis. There is no reason to expect a correlation between luminescence characteristics and
1284 sorting/stratification, and so there is no dark blue band in that portion of the figure. (b) Bar chart
1285 showing qualitatively the relative influences of different sedimentary characteristics on dose rate
1286 complexity. Each bar depicts an observable sedimentary characteristic (y-axis) that can be evaluated at
1287 the time of OSL sample collection or during sample preparation (white labels – present at the time of
1288 deposition; black labels – post-depositional alteration). Each sedimentary characteristic varies from low
1289 to high (x-axis), and the gradational colouring indicates the degree of beta, γ and cosmic dose rate
1290 complexity (in both space and time) expected for a given category value.

1291 **Figure 11: Accuracy and precision in quartz OSL dating.** (a) Large aliquot 63-90 μm quartz OSL
1292 ages plotted as a function of depth for a section of Chinese loess encompassing last glacial loess and
1293 Holocene soil. Uncertainties are random only. A Bayesian model (with 1σ error band) is also shown
1294 illustrating how higher precision can be achieved. Ages excluded from modelling are from samples
1295 deliberately taken within **crotoquina**. Figure modified from SI in⁹⁵. (b) Comparison of OSL ages
1296 determined using quartz from well-bleached samples with independent age control (n = number of
1297 samples, followed by the % of samples within $\pm 2\sigma$ of the line of unit slope)^{89, 108, 269}.

1299 **[bH1] Box 1: Water content estimation**

1300 Constraining the average water content of a deposit is important to the accuracy of a luminescence age,
1301 because a 1% increase/decrease in lifetime average water content typically leads to a 1%
1302 increase/decrease in derived age^a. Well-sorted sands have a small variation in porosity, from fully
1303 (~38%) to loosely (46%) compacted²⁹⁶, and because the grain surfaces are in physical contact (in
1304 contrast to for example, clay-rich deposits) it is very difficult to over-compress sand by increased
1305 overburden pressure. When fully saturated with water, 42% porosity corresponds to 21% by weight
1306 water content. A saturation water content can be readily estimated using for example, the syringe
1307 method^b because this packing density is reproducible, in a sandy deposit. If the sediment was below the
1308 ground-water table throughout the burial period, this is the lifetime average water content. Otherwise,
1309 the fraction of saturation during the burial period must be estimated from an understanding of the local
1310 hydrology (drainage, precipitation, etc.) and sediment characteristics²⁹⁷. The observed water content,
1311 such as the water content in a field sample measured by drying a known weight of wet material, can
1312 often be taken as a lower limit to lifetime water content because typical sampling sites (cleaned sections,
1313 gully walls, river or road cuts etc.) are usually significantly drier than corresponding material a few
1314 metres deeper into the section. In the worst case, where no further constraints can be placed on water
1315 content history, the mid-point between field and saturated water content can be chosen, and an estimate
1316 of uncertainty adopted, equal to 25% of the range. Then all possible values are covered by ± 2 times the
1317 uncertainty. For example, a measured water content of, say, 9% (cf. saturation water content of 21%)
1318 would give a mean lifetime water content of $15 \pm 3\%$ by weight, implying an overall contribution to the
1319 uncertainty on the age of $\sim 3\%$ ²⁹⁸. This is not large compared to the expected minimum uncertainty of
1320 $\sim 5\%$ (uncertainties added in quadrature, see **Table 7**), and so is not of major concern. Similar arguments
1321 can be used to constrain the effects of the infilling of porosity by carbonate.

1322
1323 The water content of a ceramic buried in a sediment is more complicated. The porosity of a ceramic
1324 will differ from that of the sediment matrix, but it is readily measured by simply saturating the ceramic
1325 in water, weighing, drying and weighing again. Then it is assumed that the fraction of saturation that
1326 applies to the sediment matrix also applies to the ceramic¹⁷. The lifetime average water content
1327 estimated for the matrix is applied to the γ dose rate, and that for the ceramic to the beta dose rate.

1328
1329 ^aThe age calculation spreadsheet attached to this paper allows the testing of the sensitivity of the ages to various parameters.

1330 ^bA saturation water content can be measured for sand by adding sediment and excess water to say, a pre-weighed 100 ml
1331 syringe and applying pressure to remove the excess water. The point at which the syringe plunger stops moving is well-
1332 defined, and increasing the pressure does not extrude more water. The syringe and wet sand are weighed to obtain the
1333 saturated weight, and then placed in an oven and dried to obtain the dry weight: the ratio of the weight loss of the saturated
1334 sample during drying to the dry weight is the saturation water content.

1336 [bH1] Box 2: The Single Aliquot Regenerative (SAR) dose procedure

1337 Assuming first order filling of an electron trap during irradiation, the subsequent luminescence signal
 1338 can then be expressed as (based on⁶³):

1339
$$L(D) = S_j \cdot \chi(D) \cdot N_0 \cdot (1 - e^{-b \cdot D}) \quad \dots\dots\dots(4)$$

1340 where L is the number of luminescence photons, S and χ describe the dependence of luminescence
 1341 sensitivity (here photons per recombining electron) on measurement number, j, and dose, D,
 1342 respectively, N₀ is an upper limit to the number of trapped electrons (determined by the trap density),
 1343 and b is a constant describing the rate of filling of the electron trap per unit dose. If the OSL trap is fully
 1344 emptied during measurement, then T(D_T), the response to a subsequent test dose D_T can be written as:

1345
$$T(D_T) = S_{j+1} \cdot \chi(D_T) \cdot N_0 \cdot (1 - e^{-b \cdot D_T})$$

1346 Thus, if D_T is now fixed

1347
$$\frac{L(D)}{T} = \frac{S_j}{S_{j+1}} \cdot K \cdot \chi(D) \cdot (1 - e^{-b \cdot D}) \quad \dots\dots\dots(5)$$

1348 where K = 1/($\chi(D_T) \cdot (1 - e^{-b \cdot D_T})$) is a constant. Equation 5 represents the luminescence response to
 1349 dosing and measurement in any one SAR cycle. If the ratio S_j/S_{j+1} is constant, the sensitivity-corrected
 1350 luminescence signal (L/T) is independent of SAR cycle, and for any given dose, D, takes on a unique
 1351 value. This result has been confirmed by numerical simulations using a more complex model that takes
 1352 into account competing electron trapping and recombination processes⁸¹, and by experiment (**Fig. 5a**).

1353 In practice, the sensitivity-corrected luminescence from the 'i'th SAR cycle is usually identified as L_i/T_i.
 1354 The first SAR cycle is denoted by i=0 because it is different from all later cycles, in that the dose, D₀,
 1355 was absorbed before any subsequent laboratory pretreatment such as preheating. If D₀ was absorbed in
 1356 nature, then it is often referred to as D_n (the measured value of D_n is D_e). In all later cycles (i=1, k), the
 1357 aliquot has been preheated and light exposed before absorbing a dose D_i. Note also that this formulation
 1358 assumes that the probability of trapping electrons in this system does not change with repeated dosing
 1359 and emptying (b is independent of j and i), and does not change from natural irradiation to laboratory
 1360 irradiation, assumptions fundamental to any added or regenerated dose procedure.

1361 Although it is the exponentially saturating population of the electron trap (Equation 4) that records the
 1362 chronometric information, it is important to realise that the resulting sensitivity-corrected luminescence
 1363 response curve (Equation 5) does not mirror the growth of the electron trap population. The
 1364 luminescence response curve may not be a simple saturating exponential and thus may not plateau at
 1365 high doses; it can continue to increase, or even decrease, with dose, all depending on the behaviour of
 1366 $\chi(D)$.

1367

1368 [bH3] Box 3: Testing for quartz purity

1369 SAR dose measurements are most successful if the quartz OSL is dominated by the fast-component.
1370 One important step in ensuring this is to remove any other minerals likely to give luminescence. In
1371 practice, this means ensuring that all feldspar has been successfully separated in the sample preparation
1372 steps described above. Unfortunately, the chemical pretreatment intended to remove feldspar
1373 contamination is not always completely successful^{299, 300}. The reasons for this are unclear, but
1374 presumably involve occluded feldspar grains, or feldspathic-like impurities within individual quartz
1375 grains. Feldspar impurities is a problem because feldspar is sensitive to the wavelengths used to
1376 stimulate quartz (usually 450 to 530 nm), and so can contaminate the quartz OSL signal. Luminescence
1377 from feldspar impurities is also significantly unstable, and much more difficult to bleach. If additional
1378 etching with HF does not remove residual signal contamination, then instrumental separation must be
1379 considered. For stimulation with blue or green wavelengths, the decay rate of the feldspar signal is
1380 considerably slower than that of the quartz fast-component, and so the use of the early background
1381 subtraction approach reduces the importance of any feldspar contamination. But feldspar, in contrast to
1382 quartz⁵⁹, is sensitive to infrared (IR) stimulation. This characteristic can be used to test for, and to
1383 reduce, contamination of the quartz OSL by feldspar OSL; this is done by inserting an IR stimulation
1384 immediately prior to the measurement of L_i (but not T_i) in an additional recycling ratio test⁹³. If the
1385 resulting $(L_i/T_i)/(L_1/T_1)$ recycling ratio (often referred to as the *OSL IR depletion ratio* in the literature)
1386 is indistinguishable from unity, then feldspar OSL does not contribute significantly to the quartz signal.
1387 In samples where the feldspar contamination remains of significance even after additional etching with
1388 HF, it can be reduced by inserting the IR stimulation step in front of every L and T measurement³⁰¹,
1389 although such a ‘double SAR’ approach is expensive in measurement time. There are also other
1390 instrumental methods for minimising any feldspar contribution, but these are not in routine use^{40, 302}.

1391

1 **Tables**

2 **Table 1** Synthesis of different dating applications and events that can be constrained using quartz OSL. Columns 4-5 indicate the general
 3 suitability of OSL dating in different geological settings and key practical consideration that can influence OSL dating reliability. (Suitability
 4 classification: O = Optimal; R = routine with appropriate methodological considerations; S = sub-optimal; C = challenging; N = not
 5 recommended; Nv = novel or not widely used)

6

Application type	Event being dated	Signal resetting agent	Suitability and age interpretation	Practical considerations
Aeolian (wind- transported)	Formation of desert and coastal dunes ^{83, 146, 161, 261, 303-311}	Daylight	O; direct dating	Usually well-reset by sub-aerial exposure. Dune fields reflect transitional climates from arid to humid to allow sand movement and subsequent trapping. OSL dates last movement, and can be used to derive dune aggradation and migration rates and desert albedo changes. Bioturbation and pedogenesis processes can affect low-cohesion, near- surface dune deposits. Significant beta-dose heterogeneity possible in low radioactivity dune deposits and/or coarse grained sand dunes. Does not significantly affect average behaviour.
	Loess accumulation ^{95, 99, 104, 148, 312-321}	Daylight	O; direct dating	Dating of gypsum dunes by targeting embedded quartz grains. Fine-grain deposits with limited sand-sized quartz. Coarse silt can be used, but feldspar contamination may be difficult to remove.

Fluvial, alluvial, beach (high-energy water transport)	Terrace deposition and abandonment ^{106, 269, 322-324}	Daylight	R-C; direct dating of fluvial deposition; indirect dating of floodplain abandonment	In all cases, both sub-aerial and sub-aqueous daylight exposure likely for fluvial sediment transported downstream through many reworking cycles, with intervening periods of direct daylight exposure during low flow. Thus well-sorted distinct sand lenses/layers derived from low energy, shallow water environments are most likely to be well-bleached; they also provide a simplified dosimetry.	
	Channel migration ^{325, 326}	Daylight	R-C; direct dating of channel deposition; indirect dating of channel erosion		
	Channel cutting and filling; floodplain aggradation and alluvial fan formation ³²⁷	Daylight	R-C; direct dating of filling events, indirect dating of cutting or incision events		
	Palaeoflood deposits ^{211, 212, 328}	Daylight	R-C: direct dating of flood events		Bleaching during flood unlikely, but prior bleaching of sediments before flood entrainment seems common.
	Beach formation associated with sea level changes ^{217, 293, 329-331}	Daylight	O-C; direct dating		Usually well reset by sub-aerial and sub-aqueous bleaching. High carbonate contents common in low-latitude beach deposits, may lead to complexity (time-dependent dose rates) if carbonate content changes with time.
	Tsunami deposits ³³²⁻³³⁴	Daylight	S-N; direct dating	Bleaching unlikely during event but sediments entrained in shallow water likely already well-bleached.	
Lacustrine and offshore-marine	Lake level variations ³³⁵⁻³⁴⁰	Daylight	S-C; direct dating of lake full phases (sedimentation);	Sampling by coring usually precludes <i>in situ</i> γ dose rate measurement. Organic-rich, low-energy lakes may have limited coarse-grain quartz yields. Time-dependent dose rates can arise from both U-series	

(low-energy water transport)			indirect dating of lake drying phases (non-deposition)	disequilibria and sediment compaction/dewatering associated with accumulation of overburden (especially true for gyttja).
	Formation of lake-margin deposits (deltas, lake shoreline features) ^{143, 151, 152, 341-343}	Daylight	R; direct dating	Used to date lake high stands. Marginal deposits may be more accessible and suitable than lake bed deposits (in terms of grain size, bleaching history). Sub-aerial and sub-aqueous bleaching likely for beach deposits.
	Ocean floor sedimentation ^{5, 13, 14, 144, 344, 345}	Daylight	R-C; direct dating	Bleaching sub-aerial (dust) or sub-aqueous (fluvial input). Time-dependent dose rates due to U-series disequilibria may increase complexity, especially in the presence of high carbonate content. High water content reduces dose rate and extends age range. Usually very low coarse-grained quartz content.
Glacigenic (ice-related)	Moraine / till formation ^{157, 346-348}	Daylight	R-C; direct dating of ice margin	For all glacial sediments, low OSL signal sensitivity is common in primary ice-eroded material. Limited/no daylight exposure possible. If sediment unexposed to daylight during the last transport, the OSL age will relate to an earlier transport event, but dose rate may have changed.
	Proglacial outwash plain activation ^{157, 159, 349, 350}	Daylight	R; direct dating of outwash plain; indirect dating of ice margin	Transport distance from ice-margin important for bleaching.

	Glaciofluvial channel activation ^{150, 351, 352}	Daylight	R; direct dating of channel activation; indirect dating of ice margin	Event-related activation may lead to limited daylight exposure.
	Glaciolacustrine sedimentation ³⁵³	Daylight	R-S; direct dating of proglacial lake sedimentation.	Sediment deposited in ice-marginal turbid lakes may experience little/no daylight exposure; OSL age may therefore reflect previous transport events.
	Glacioaeolian deposition ^{163, 209, 354-356}	Daylight	O; indirect dating of (peri)-glacial processes	See “sand dune formation” and “loess accumulation” above.
Colluvium, solifluction sheets, debris flows (gravitational mass movements)	Slope failures, formation and progradation ³⁵⁷⁻³⁶⁰	Daylight	SR-C; direct dating	Limited transport distances and/or daylight exposure durations can increase the likelihood for heterogeneous bleaching. Poorly sorted deposits may suffer from spatially heterogeneous radiation fields.
Archaeology, palaeontology, palaeoanthropology (including karst deposits)	Site depositional history ^{97, 105-107, 193, 194, 198, 202, 361-363}	Daylight	O-R; direct dating	Covers a wide-range of depositional contexts (see above for details). Open-air archaeological sites have better bleaching conditions and fewer post-depositional complexities than caves and rock shelters. Complexity from syn-depositional mixing and contamination by older generations of pre-existing deposits may arise in cave settings.
	Internment of autochthonous fossils / cultural materials in sediments ^{201, 207, 208, 210, 364}	Daylight	O-R; indirect dating but close association with dating event	Multiple cycles of human occupation may cause post-depositional disturbance (digging, burials, fire pits).

	Internment of allochthonous fossils / cultural materials in sediments ^{230, 365}	Daylight	O-R; indirect dating and poor association with dating event	OSL provides minimum age for fossils and archaeological material. Animal disturbance of host sediments is possible in dens and pitfall traps.
	<i>In situ</i> production of cultural materials, including debitage production ^{97, 135, 136, 366}	Heat (lithics, fireplaces) Daylight	R, C, Nv; direct dating	Stones (fireplace, pot-boilers) usually straightforward if heated sufficiently to fully reset. Heated lithics with no stratigraphic context (museum specimens) will have poorly constrained γ dose rates.
	Heating of pottery, bricks, tiles. Manufacturing of mortars ^{131, 132, 367-370}	Heat, Daylight (mortars)	O; direct	Straightforward but extraction of sufficient coarse quartz may not always be possible in pottery. Surrounding sediment/brick should be collected for γ dose rate estimation
Soil and regolith processes	Formation of soil A horizons ^{125, 169, 172}	Daylight	C, Nv; direct dating	Bleaching by turbation ongoing until soil surface buried. Age usually reflects time of burial. Old soils well-bleached, young soils usually incompletely bleached. Scatter in D_e can be interpreted in terms of turbation rates.
	Sediment mixing between layers ^{170, 240, 371}	Daylight	C-N; direct dating	Single-grain studies may provide insight into grain movement histories. Reliable dating not usually possible, but identification of mixing may be possible.
	Soil creep rates ^{168, 372}	Daylight	C, Nv; direct dating	Reliable interpretation of apparent ages challenging.
	Formation of pedogenic and biogenic structures (carbonate nodules, termite mounds, root casts) ^{171, 173}	Daylight	C-N, Nv; direct dating	Careful sampling important to isolate individual formation events. Problems of interpretation similar to those for soil creep rates. Carbonate-rich structures may involve time-dependent dose rates.

Neo-tectonics and volcanic histories	Occurrence of palaeo-earthquakes ^{164-167, 373-375}	Daylight before/after seismic events	R-C; indirect dating of seismic event itself but close stratigraphic association	Sediments associated with palaeo-earthquakes often experienced limited transport (and so daylight exposure), particularly colluvium, debris flows or locally intruded deposits. Targeting stable palaeo-surfaces can improve dating reliability.
	Offset of geomorphic surfaces due to fault movement ³⁷⁶⁻³⁸¹	Daylight before and after faulting event	R-C; indirect dating of faulting event itself but close stratigraphic association	Limited transport distances and daylight exposure if sampling fluvial, colluvium, debris flows or locally intruded deposits. Rock surface dating may be applicable.
	Dating fault gouge and dike intrusions ¹³⁸	Frictional heating	R-C; direct dating of faulting or seismic event	Sand-sized quartz grains may not be present in gouge. Gouge may be very thin, making sampling challenging
	Volcanic eruptions – dating proximaldeposits ³⁸²⁻³⁸⁴	Heating by magma	R-C; indirect dating of volcanic event itself but close stratigraphic association	Quartz from volcanic sources usually insensitive, weak fast-component. But quartz from contact-heated sediments likely to have improved OSL sensitivity.
	Volcanic eruptions – dating distal deposits ^{104, 351, 354, 364, 385, 386}	Daylight	R-C; indirect dating of volcanic event itself but close stratigraphic association	Sampling above and below discrete tephra horizons constrains ash fall events. Sampling of deposits that host cryptotephra is also possible.
Burial dating of rock surfaces	Construction of megaliths, cairns, stone walls, etc. Dating erratics,	Daylight	R-C, Nv; direct dating	Only likely applicable to sandstones and some quartzite clasts because of poor quartz OSL sensitivity in primary rocks (e.g. granites).

	scree slopes, rockfalls, cobble beaches and beach ridges ^{187, 188, 284}			
Exposure dating of rock surfaces	Construction of megaliths, cairns, stone walls, etc. Rock art dating. Dating erratics, scree slopes, rockfalls, cobble beaches and beach ridges ^{15, 187, 287}	Daylight	C, Nv; direct dating	Known-age exposure samples needed for calibration. Only likely applicable to sandstones and some quartzite clasts because of poor quartz OSL sensitivity in primary rocks (e.g. granites).
Thermochronometry	Bedrock exhumation ^{387, 388}	Cooling below closure temperatures of 30-40°C	C, Nv; direct dating	Signal saturation restricts applications to rapidly exhumed settings or elevated temperature settings. Combination of quartz OSL with other signals (TL, IRSL, post-IR IRSL) may offer more versatility. Only likely applicable to sandstones and some quartzite clasts because of poor quartz OSL sensitivity in primary rocks (e.g. granites).
Palaeo-lightning events	Fulgurite formation ¹⁴¹	Heating of trapped quartz grains	O-R, Nv; direct dating	Target quartz grains occluded in the fused matrix.

7

8

9 **Table 2.** Example tables containing suggested minimum sample-specific information that should be presented in a publication containing quartz OSL
 10 luminescence ages. All uncertainties expressed at 1σ confidence level and contain both random and systematic components. More general characteristics
 11 (especially dose recovery) are also required to assess reliability, and can be summarised in associated text.

12

13 a)

Lab.code	^{238}U , Bq.kg $^{-1}$	^{226}Ra , Bq.kg $^{-1}$	^{232}Th , Bq.kg $^{-1}$	^{40}K , Bq.kg $^{-1}$	*beta dose rate, Gy.ka $^{-1}$	* γ dose rate, Gy.ka $^{-1}$
1	51 \pm 12	46.0 \pm 1.2	61.0 \pm 1.2	562 \pm 20	2.29 \pm 0.06	1.48 \pm 0.04
2	72 \pm 12	48.2 \pm 1.1	60.6 \pm 1.2	566 \pm 16	2.32 \pm 0.05	1.50 \pm 0.04
3	58 \pm 9	45.2 \pm 0.9	60.2 \pm 1.0	581 \pm 13	2.33 \pm 0.05	1.48 \pm 0.04
4	46 \pm 8	43.8 \pm 0.8	32.2 \pm 0.8	158 \pm 8	1.05 \pm 0.04	0.81 \pm 0.04

Note: * dry infinite matrix dose rates

14

15 b)

Lab.code	Site	Depth, cm	w.c. %	OSL D_e , Gy	(n_r) (n_a)	Total dose rate, Gy/ka	OSL age, ka
1	Test site-A	65	16	95 \pm 4	1 23	3.16 \pm 0.16	30.0 \pm 2.0
2	Test site-B	65	16	73 \pm 4	0 12	3.19 \pm 0.16	22.9 \pm 1.7
3	Test site-C	65	16	107 \pm 6	0 24	3.20 \pm 0.16	33.5 \pm 2.5
4	Test site-C	30	11	43 \pm 2	1 35	1.79 \pm 0.09	23.9 \pm 1.7

Note: (a) Mean grain size 215 μm . (b) Site altitude 0 km. (c) w.c.: water content. (d) n_r , n_a : number of rejected and accepted aliquots, respectively.

16

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Supplementary Information

(Section numbering corresponds to the relevant section number in the main text)

2. Experimentation

(a) Sampling

Luminescence dating obviously starts in the field, where the sample containing quartz grains - usually sediment - must be collected. Since OSL dates the last exposure of sediment to daylight, one must avoid light exposure during sampling. The surface of the sediment, or of the sedimentary sequence of interest, is first cleaned by scraping back. This is not only to avoid the light exposed first few mm of the exposure, but also to provide a clear view of the stratigraphy and so avoid locations that may have been affected by post-depositional disturbances (ice wedges, animal burrows, roots, etc.). At least the first few cm must be removed; it is better to cut a section back by > 1 m to avoid air-exposed material and so to take samples where oxidation/reduction conditions and moisture content are more likely to be representative of the long term burial (see Section 2.1 on dose rates). All these operations can be done in full daylight and taking photographs of sampling site (overview) and sampling locations (detail) is recommended.

There are then two approaches to collecting a sediment sample: the simplest is to hammer tubes (metal or plastic, depending on the deposit) into the cleaned section; these are often intended for one-time use, but may be reusable [1]. This method is only suitable when the unit to be sampled is of sufficient thickness that the tube will not cross sedimentological boundaries. Ideally the gamma dose rate gradient across the tube should also be small; if possible, the tube should be at least 15 cm from any boundary. A plug of wadded paper or crumpled plastic bag should be placed in the tube, at the end closest to the section. This will support the sediment as it enters the tube and reduce the risk of it collapsing and mixing exposed and unexposed material. The tube must be filled completely, either with sample or by packing with plastic, to ensure that the material cannot move and mix during transport. In the laboratory the potentially light-exposed outer ~ 2 -3 cm of each end of the tube is retained for radionuclide concentration measurements, and any other sedimentological analyses (e.g. grain size, mineralogy, geochemistry, ancient DNA, tephrochronology, etc.). The inner part of the tube is used for OSL measurements, and possibly for water content measurements.

If the unit is too thin to be sampled with a tube, then the second approach is to operate in the dark – either at night (moonlight is very low intensity, and will not significantly affect the OSL during the

short sampling exposure, see [2]), or under an opaque covering such as a tarpaulin. In both cases, it is safe to use a controlled light source, such as headlamp emitting dim orange light, or a red LED (e.g. bicycle rear light). It is usual to collect two parts for each sample: one mainly for OSL analysis and the other mainly for radiochemical analysis. The previously cleaned surface is first scraped to remove a further cm or so. This part is likely to have been exposed to daylight after the cleaning of the section, and is not suitable for OSL measurement; it is removed and retained for measurement of the radioelement concentrations and for any complementary measurements. Since exposure to light is unimportant for such measurements, this part of the sample may be collected in transparent bags. The second part of the sample, taken from further into the section, must be protected from light exposure, either by packing into metal containers, or fully opaque plastic bags. Usually a few hundred grams is sufficient – but that of course depends on the concentration and grain-size distribution of quartz grains in the sample. A carbonate-rich sandstone will generally yield less quartz than a desert sand, and a marine/lake core fewer sand-sized grains than a beach sand. If a narrow-grain-size fraction is to be targeted (e.g. for single-grain OSL), the amount of sediment required may be greater than usual.

If the target is ceramic material (pottery, bricks, tiles) or cobbles, heated stones etc., all sampling can be undertaken in daylight, although direct sunlight should be avoided. This is because, in the case of ceramics and heated stones, the potentially light-exposed surface of the sample is discarded in the laboratory, and in the case of cobbles, light is attenuated so rapidly that a few minutes of daylight does not affect the average luminescence signal in the outer 1 mm.

During sampling one must also consider the water content history of the site and the collection of (separate) samples for the present day water content. A subsample of the OSL samples (from inside tube) or the transparent bags (see above) can be used to determine this and the saturated water content (see in section 2.1.2 below). In order to retain as much moisture as possible sealable bags should be used and the ends of the tubes covered with plastic and sealed with tape.

Finally, sampling is the occasion to collect information relevant for calculating cosmic dose rates (Figure S1) by measuring or estimating the overburden of a sample. This includes the nature of the overburden (e.g. cave roof, sediment, etc.), the density and above all the thickness of the material covering every sample. The elevation of the site above sea level and geographical coordinates (latitude, longitude) should also be recorded. These field observations are used to calculate the cosmic dose rate [3]. Furthermore, in some cases (see section S2.1 below) where the sediment is heterogeneous on a scale of 10-50 cm (presence of rocks, sediment unconformity, boundary between sediment and air, or sediment and bedrock, etc.) it may be necessary to measure in situ gamma dose rates. It is often also possible to collect two separate dose rate samples (one from sediment to be dated and one from the adjacent rock/sediment causing heterogeneity), measure the radionuclide concentrations for both samples and then model the gamma dose rate (see Appendix H in [4]).

(b) Sample preparation

In the laboratory, that part of the sample intended for dose rate measurements is homogenised (usually by grinding) so that even a small sample of less than few grams is representative of the whole. This part may be from the ends of a sampling tube, or the outer few mm of a ceramic or heated stone. Section S2.1 lists typical methods for dose rate determination. It is good practice to keep a portion of the non-light-exposed material as back-up in case something goes wrong in sample preparation. The remaining part of the sample intended for OSL measurements undergoes several treatment steps to extract quartz grains (see Figure S2). The unexposed inner part of ceramics and heated stones is gently crushed. Then the crushed material, or the sediment, is wet sieved - a constrained grain size fraction is of importance not only because of measurement practicalities, but also because both laboratory and environmental dose rates are, to some degree, grain-size dependent – see section S2.1 below. The factors driving the selection of grain size are: (i) the abundance of that fraction in the samples; (ii) the ability to chemically etch the outer surface of the grains to remove the alpha-irradiated component (typically not smaller than fine sand, i.e. $> 63 \mu\text{m}$ – grains must be large enough, or else nothing is left after the HF treatment described below); (iii) ease of manipulation of the grains. Generally, some choice is made within the range 63-300 μm (e.g. 90-125 μm or 180-250 μm); the fraction 150-180 μm is most suitable for measurements using single-grain discs with e.g. $\varnothing 200 \mu\text{m}$ holes.

The sample should remain at or close to room temperature in all the steps outlined below, to avoid the risk of thermal fading of the OSL signal; this applies particularly to the HF digestions steps. The selected size fraction is treated with 10% HCl for at least 30 minutes (until no reaction is visible) to remove carbonates - these may impede the reaction with HF - and with 10% H₂O₂ (for up to a few days) to remove finely disseminated organics. After HCl and H₂O₂ treatments, the grain fractions are washed with water (usually 2-3 times). Note that it is usually easier to soak more compact fine-grained (clayey-silty) sediment in water and treat with HCl, H₂O₂ first, to disaggregate the grains, before wet sieving. This is also true for carbonate-rich sandstones (e.g. aeolianites).

After sieving and treating with HCl and H₂O₂, there should be at least a few gram(s) of sample, at this point mainly a mixture of quartz, feldspar and possibly heavy-mineral grains in the chosen grain size fraction. If only quartz is desired, the sample may be placed directly in concentrated HF (40%); usually ~1 hour is sufficient to remove feldspar and etch the surface of the quartz grains (see below). If the feldspar grains are also required, then prior mineral separation is necessary (K-feldspars in particular are commonly used in other approaches to luminescence dating). Prior to mineral separation the mixed quartz/feldspar fraction can be etched for up to 40 min in 10% HF to remove surface coatings (e.g. iron oxides) and attached clay grains, washed in 10% HCl for ~20 mins, and then with water and dried. This step improves recovery in the following separation step. Density separation using aqueous heavy liquids (e.g. lithium heteropolytungstate, or LST) is a safe and convenient

approach; a density of 2.70 g.cm^{-3} allows the heavy minerals to sink; 2.62 g.cm^{-3} allows a quartz-rich fraction to sink; 2.58 g.cm^{-3} allows a Na-rich feldspar fraction to sink, and floats mainly K-rich feldspar grains. Some laboratories use only a single 2.58 g.cm^{-3} step, and rely on HF to remove any denser feldspars that remain in the quartz-rich fraction. Any heavy minerals surviving the HF treatment rarely, if ever, make a significant contribution to the quartz luminescence signal. Magnetic separation is also used to separate quartz and feldspar grains [5].

Finally, treatment with concentrated HF (40%) for 1 hour removes any remaining feldspar, and $\sim 10 \mu\text{m}$ from the outer surface of the quartz grains, thus removing most of the quartz layer exposed to alpha particles from the surrounding sedimentary matrix during burial. The internal alpha activity of quartz is very small, and so this step significantly simplifies the dosimetry calculations. Some laboratories then soak the sample in 10% HCl for several hours to remove any fluorides that might have precipitated on the grain surfaces, because some fluoride salts are luminescent. Although there is no evidence that these amorphous precipitates are in fact a significant source of contamination in quartz OSL, some fluoride residues can be of sufficient thickness to significantly attenuate the OSL signal. Finally, the samples are washed (typically 3 times) with water and dried; it may be desirable then to sieve the samples again, to define the minimum grain size during beta irradiation, and, for single grain measurements, to avoid the risk of multiple grains in each hole in the sample holder. The quartz is then tested for signal purity (Section S2.2.1(a)); if it fails this test, the HF etch can be repeated (the additional mass loss will have a small effect on the average natural beta dose to the grains).

2.1. Dose Rate

2.1.1. Basic concepts

(a) Dose rate calculation using the infinite matrix assumption.

Various methods for determining the dose rate are summarised in Table S1. Concentrations of radionuclides may be expressed in activity concentrations [Bq.kg^{-1}] or mass concentrations. To calculate the activity concentration from a mass concentration (C_0 , [g.kg^{-1}]) measurement, the atom concentration per unit mass of sediment (N , [kg^{-1}]) is first calculated using $N=C_0 [\text{g.kg}^{-1}] / Z$ (atomic weight [g.mol^{-1}]) $\times N_A$ (Avogadro's number [mol^{-1}]). N is then multiplied by the relevant decay constant (λ , [s^{-1}]) to give the radionuclide activity concentration. Measured concentrations may be reported in ppm [$\mu\text{g.g}^{-1}$] for U and Th, and percentage [%] for K; these must first be converted to [g.kg^{-1}]. In the case of K, the mass concentration of ^{40}K must then be derived using the isotopic abundance (0.0117%).

Finally, for each nuclide, multiplying the activity concentration by the average energy emitted per disintegration (see e.g. <https://www.nndc.bnl.gov/nudat2/> given in MeV, $1 \text{ MeV}=1.6\times 10^{-13} \text{ J}$;) gives

what is known as the infinite matrix dose rate. Relevant conversion factors ($C_0 \times N_A \times \lambda / Z$) (summarised in Table S2) are regularly compiled and updated (e.g. [6]). Table S3 uses these data to derive infinite matrix dose rates from a sand containing representative concentrations of ^{40}K , ^{238}U and ^{232}Th . Figure S3 shows the ^{238}U and ^{232}Th radioactive decay chains, and Figure S4 groups radionuclides according to their potential mobility in sediment, and presents the relative contribution of each group to the total dose rate, assuming the relative activity concentrations of Table 3. Figure S5 summarises the potential variability in ^{40}K , ^{238}U and ^{232}Th concentrations, and their relative contributions to dose rates. Figure S6b) illustrates how variable beta dose rates can be at the 100 μm scale.

(b) In situ gamma dose rate measurements

In some cases, sediment is heterogeneous at the scale of the penetration distance of gamma rays (~30-50 cm, see Figure S6a), e.g. if it is poorly sorted, and includes pebble and cobbles, or is adjacent to a rock wall. Then the infinite matrix gamma dose rate may not be representative of the gamma dose rate to which the sample was exposed during burial. As an alternative, the gamma dose rate may be measured in situ, at the sample location, e.g. with luminescent $\text{Al}_2\text{O}_3:\text{C}$ pellets. Aluminium oxide is very similar to quartz in terms of dose absorption characteristics, and doping with carbon gives this material a very sensitive luminescence response to dose [7]. In practice, pellets are left buried in an aluminium tube of ~2-3 mm thickness (to prevent beta particles from reaching the pellets) for 6-12 months, after which the absorbed dose is sufficient to induce a measurable OSL signal [8], [9]. This directly measured dose rate is the sum of gamma and cosmic dose rates, as affected by escape of ^{222}Rn and the water content of the sediment over the period of measurement. The other common way of determining in situ gamma dose rates involves the use of a field gamma spectrometer, typically with a NaI(Tl) or LaBr probe. Calibration involves measurements in infinite media with known radionuclide concentrations [10]. Data analysis may consist of estimating effective K, U and Th concentrations using the ‘windows technique’ (see Appendix L in [4]), and then converting these concentrations into effective infinite matrix dose rates. The concentration of K is based on the emission rate of the 1461 keV peak, of U of the 1764 keV peak and of Th of the 2615 keV (e.g. [11]). Otherwise, one may use the proportionality between gamma dose rate and the count rate above a certain energy channel, in what is called the threshold technique [12], [13]. The advantage of the threshold technique lies in the inclusion of a greater portion of the recorded spectra to estimate the gamma dose rates, thereby reducing the uncertainties for a given counting time (typically several minutes), but this approach does not give any nuclide-specific information.

2.1.2. Practicalities

Water content measurements – Constraining the average water content of a deposit is very important to the accuracy of a luminescence age, because a 1% increase/decrease in life-time average

water content typically leads to a 1% increase/decrease in derived age¹. Well-sorted sands have only a small variation in porosity, from fully (~38%) to loosely (46%) compacted [14], and because the grain surfaces are in physical contact (in contrast to e.g. clay-rich deposits) it is very difficult to over-compress sand by increased overburden pressure. When fully saturated with water, 42% porosity corresponds to 21% by weight water content. Because the packing density is reproducible, a saturation water content can be measured for a sandy deposit by adding sediment and excess water to say, a pre-weighed 100 ml syringe and applying pressure to remove the excess water. The point at which the syringe plunger stops moving is well-defined, and increasing the pressure does not extrude more water. The syringe and wet sand are weighed to obtain the saturated weight, and then placed in an oven and dried to obtain the dry weight – the ratio of the weight loss of the saturated sample during drying to the dry weight is the saturation water content. If the sediment was likely to have been below the ground-water table throughout the burial period, this is the lifetime average water content. Otherwise, the likely fraction of saturation during the burial period must be estimated from a knowledge of the local hydrology (drainage, precipitation, etc.) and sediment characteristics [15]. The observed water content (*i.e.* the water content in a field sample, measured simply by drying a known weight of wet material) can often be taken as a lower limit to lifetime water content, since typical sampling sites (cleaned sections, gully walls, river or road cuts etc.) are usually significantly drier than corresponding material a few metres deeper into the section. In the worst case, where no further constraints can be placed on water content history, the mid-point between field and saturated water content can be chosen, and an estimate of uncertainty adopted, equal to 25% of the range. Then all possible values are covered by ± 2 times the uncertainty. For example a measured water content of, say, 9% (cf. saturation water content of 21%) would give a mean lifetime water content of $15 \pm 3\%$ by weight, implying an overall contribution to the uncertainty on the age of $\sim 3\%$ [16]. This is not large compared to the expected minimum uncertainty of $\sim 5\%$ (uncertainties added in quadrature, see Table S8), and so is not of major concern. Similar arguments can be used to constrain the effects of the infilling of porosity by carbonate.

The water content of a ceramic buried in a sediment is more complicated. The porosity of a ceramic will differ from that of the sediment matrix, but it is readily measured by simply saturating the ceramic in water, weighing, drying and weighing again. Then it is assumed that the fraction of saturation that applies to the matrix also applies to the ceramic [4]. The lifetime average water content estimated for the matrix is applied to the gamma dose rate, and that for the ceramic to the beta dose rate.

¹ The age calculation spreadsheet attached to this paper allows the reader to test the sensitivity of the ages to various parameters.

2.2. Testing the applicability of a SAR protocol

2.2.1. Dose estimation using SAR

(a) *Testing for quartz purity*

The chemical pretreatment intended to remove any feldspar contamination is not always completely successful [17, 18]. The reasons for this are unclear, but presumably involve occluded feldspar grains, or even feldspathic-like impurities within individual quartz grains. This is a problem because the luminescence from feldspar impurities is significantly unstable, and much more difficult to bleach. Unfortunately, feldspar is sensitive to the wavelengths used to stimulate quartz (usually 450 to 530 nm), and so can contaminate the quartz signal. If additional etching with HF does not remove residual signal contamination, then instrumental separation must be considered. For stimulation with blue or green wavelengths, the decay rate of the feldspar signal is considerably slower than that of the quartz fast component, and so the use of the early background subtraction approach helps to reduce the importance of any feldspar contamination. But, feldspar, in contrast to quartz [19], is sensitive to infrared (IR) stimulation. This characteristic can be used to test for, and to reduce, contamination of the quartz OSL by inserting an IR stimulation immediately prior to the measurement of L_i (but not T_i) in an additional recycling ratio test [20]. If this $(L_i/T_i)/(L_1/T_1)$ ratio is indistinguishable from that measured without IR (see section 2.2.2 in main text), then feldspar does not contribute significantly to the quartz signal. In samples where the feldspar contamination is of significance, it can be reduced by inserting the IR stimulation step in front of every L and T measurement [21], although this is expensive in measurement time. There are also other instrumental methods for minimising any feldspar contribution, but these are not in routine use [22, 23, 24].

(b) *Instrumentation for determining the D_e*

The instrumentation for determining the absorbed dose is relatively simple and comprises four main components: 1) luminescence detector, 2) stimulation light source, 3) source of ionising radiation and 4) heating system. **Error! Reference source not found.** summarises the relationship of these four components to the quartz grains to be measured.

The main challenge in designing such a system is the instrumental sensitivity. The ability to measure an absorbed dose depends both on the sensitivity of the sample (i.e. the number of photons emitted per unit mass [kg^{-1}] and per unit absorbed dose [$\text{kg}^{-1} \cdot \text{Gy}^{-1}$]), on the instrument sensitivity, i.e. the fraction of the emitted photons that are actually detected by the instrument (detection efficiency), and the relationship of this signal to the instrument background noise. Detection efficiency depends on the light path (controlling the fraction of the emitted signal reaching the detector, and dependent on

geometry, mirrors, lenses and filters), the choice of photon detector (how many photons arriving at the detector are required to give a detected event – the quantum efficiency) and the wavelength and intensity of the stimulation light source (the more rapidly the charge is released (detrapped), the stronger the initial luminescence signal compared to background. For a given detection wavelength, the electron detrapping rate depends on the wavelength-dependent photoionization cross-section and the power density of the stimulation light).

The luminescence detector consists of a photon detector and detection filter(s). Natural phosphors such as quartz are relatively inefficient in converting absorbed dose into luminescence and thus the emitted luminescence signal is usually too weak to be detected by the naked eye. To measure such signals precisely, a highly sensitive light detector with a low noise (background) level and good light collection are essential. The latter requires as large as possible solid angle subtended by the detector surface at the sample – this is usually achieved by as short as possible distance between sample and detector, or by focussing through lenses. In OSL the luminescence signal is emitted as a consequence of optical stimulation, which is $\sim 10^{15}$ times more intense than the OSL signal itself. OSL is an anti-Stokes emission, i.e. the energy of the optical stimulation is less than that of the emitted luminescence. Appropriate detection filters must be used both to separate the luminescence from the stimulation light and to define the spectral detection window.

Photon detector - Figure S8 shows the emission spectrum from quartz obtained by stimulation with 647 nm laser light. Quartz has a relatively broad OSL emission peaking at ~ 360 nm (full width half maximum, FWHM ~ 80 nm). Photomultiplier tubes (PMT) operating in photon-counting mode, sensitive in the blue/UV range are most commonly used for detecting quartz OSL, mainly because of their high sensitivity, large dynamic range and low dark count rate. A high signal-to-noise ratio is particularly important when measuring insensitive samples and/or samples having absorbed small radiation doses (i.e. young samples). The quantum efficiency (the effectiveness of the PMT at converting photons into electrons) is typically up to ~ 30 %, depending on the wavelength of the luminescence. The only major drawback of using PMTs is that they do not provide information on the spatial variation of the emitted luminescence. In applications where such spatial information is of importance (e.g. rock surface luminescence dating) electron-multiplier charge-coupled devices (EMCCD) can be used, although they are in general less sensitive than PMTs (e.g. [25], [26], [27]).

Detection filters - The use of detection filters is important both to define the wavelength window of interest and to separate the emitted luminescence from scattered stimulation light. In quartz OSL dating, the UV/blue emission is usually detected through a broadband Hoya U-340 glass filter, which has its peak transmission at 340 nm with a FWHM of 80 nm (see Figure S9). The main emission from quartz is centred at 365 nm and thus, in principle, a significant improvement in light detection is available by choosing a detection filter closer to the main emission of quartz. However, moving the

detection window closer to the stimulation wavelength (usually 470 nm, see below) is likely to lead to a significant increase in background signal (from breakthrough of the stimulation light) and hence the signal-to-noise ratio is not necessarily improved [28].

Stimulation light source – In quartz OSL, the electron detrapping rate depends both on the intensity and the wavelength of the optical stimulation light (see Figure S10; [29, 30]); the more intense and the shorter the wavelength the stronger the OSL signal per unit time. For most OSL applications, optical stimulation is at a constant light intensity (continuous wave mode), but modulating the intensity of the stimulation light (e.g. linear modulated OSL, LM-OSL; [31]) can be used to visually separate the traps involved in the OSL production. In some applications, pulsing the stimulation light and only detecting in between short (~ 50 μ s) bursts of stimulation light (Pulsed OSL, POSL) can help to separate quartz and feldspar OSL signals instrumentally [23]. POSL stimulation can also be used to gain insight into the physics of recombination processes (e.g. [32]).

Optical stimulation of quartz can be achieved using any visible light source such as filtered lamp systems [28], lasers [33, 34, 26], light emitting diodes (LEDs) [35] and laser diodes [36, 26]. In most quartz multi-grain OSL dating applications, blue (450-470 nm) or green (~ 525 nm) LEDs with power densities at the sample position ranging between ~ 40 and 100 mW/cm^2 are used, resulting in the decay of the fast OSL component in a few seconds. As LEDs have a broad emission (FWHM of ~ 30 nm) it is often necessary to stimulate through cut-off filters to ensure an effective separation of stimulation light and detection windows ([37], see Figure S9). Higher energy light sources (e.g. 405 nm laser diode [38]) can be used to access deeper traps insensitive to blue light. In single-grain applications, stimulation by a green (532 nm) solid state laser providing a power density of ~ 50 W/cm^2 is commonly used [39].

Source of ionising radiation - The measured natural OSL signal must be calibrated in terms of absorbed dose. As different aliquots can have very different luminescence sensitivities, it is necessary to calibrate aliquots individually. This calibration is performed by comparing the natural OSL signal to that arising from a known laboratory dose given to each aliquots (using a SAR protocol, Section 2.2 in main text). This known dose can be given by any source of ionising radiation, but for convenience this is usually a beta source containing the artificial radionuclide ^{90}Sr ($\tau_{1/2}$ 28.8 years) and its short lived progeny ^{90}Y . This combination of emitters has a maximum beta energy of 2.27 MeV and a mean energy of ~ 600 keV, which makes it relatively easy to shield sufficiently for it to be safe to use.

The laboratory dose rate to a quartz sample depends on the activity of the source, the source/sample distance, sample grain size, and on the substrate material on which the grains sit. Because of this the source must be calibrated for each irradiation geometry (particularly for different substrates and grain-size range). The calibration is usually done by using a portion of quartz grains that has been given a

known dose using a standardised gamma source. An appropriate SAR protocol then aims at determining the duration of beta irradiation that provides a sensitivity-normalised luminescence intensity equal to that induced by the reference gamma dose [40]. Some laboratories make available sensitive quartz which has absorbed a well-known dose, so that others without access to a calibrated gamma source can calibrate their own beta source as required.

Heating system – to obtain accurate measurements of D_e , it is important to perform reproducible thermal pre-treatments and to be able to keep the aliquot at a given temperature during optical stimulation. OSL dating systems will usually heat up to 700 °C using heating rates typically ranging between 0.01 and 10 °C /s, although temperatures above 350 °C are rarely used.

4. Reproducibility and data deposition

(a) **Data reporting:** It is common in the literature to read articles describing equivalent dose determination in detail, while dose rate data are often summarised in a way that renders recalculation or reproducibility studies difficult. Until recently most articles containing luminescence ages presented new data (with variable degree of detail). However, publications addressing larger scale questions by presenting meta-analyses of published datasets are becoming more frequent. To encourage this, detailed information behind the OSL ages is required, both to allow the assessment of the quality of the data, and to ensure that the same underlying assumptions are adopted, and so enable the derivation of a coherent data set. Bayesian modelling of OSL ages under stratigraphic constraints may require a combination of raw measurement files and details of calibration uncertainties, both for equivalent dose and dose rate measurements [41], [42]. Here we discuss data reporting in terms of good practice, in the sense that it provides sufficient information for later comparisons to be made between studies that focus on the same research question – and thus, on the same samples, stratigraphic records or dated events.

When publishing OSL results, it should be made clear to the reader whether the signal is fast component-dominated (perhaps with a figure showing a comparison with known fast-component dominated material such as calibration quartz [40]), and that the interpolation is not compromised by saturation effects (e.g. using an illustrative dose response curve, see Figure 4(c,f)). Although recuperation and recycling are important (and can be shown on the dose response curve), it is most important to document the dose or light recovery ratio for the material under consideration (Section 2.2.2(vi)). In general, it is not necessary to give a value for each sample from the same sedimentary suite, a single average with standard error and number of measurements (as in Figure 6(b)) is usually sufficient.

In presenting dose rates, the methods adopted should be presented first, starting with the type of instruments and experimental conditions (Table S1). Second, we recommend that the measured values are reported: for example, if high resolution gamma spectrometry has been used, then concentrations in K, U and Th should be given; if a beta counting system is used, then count-rates should be given. Reference to the calibration standards and procedures is of course desirable – laboratories commonly publish their protocols, e.g. [43], [44], [45].

The source of the conversion factors is important: in the two examples above, conversion is from (activity) concentrations to dose rates and from count rates to dose rates, for gamma spectrometry and beta counting, respectively (Table S2). The reader should also be able to find (i) which factors were used to account for grain size attenuation of beta dose rates (e.g. Table S4 below taken from [46]); (ii) if alpha dose rates were taken into account (and if so, which alpha dose rate efficiency factors were used); (iii) what water content value was used, how it was determined and which factors were used to account for the effect of water on dose rates (see Tables S5 and S6, and e.g. [47], [48], [16]); (iv) if an internal dose rate was considered - and if so, if it was measured or simply assumed [49]; (v) geographical coordinates, altitude and burial depth, together with the derived cosmic dose rates² (unless gamma and cosmic dose rates were measured directly using e.g. Al₂O₃:C pellets). In some cases, sediment may be rich in organic matter or carbonates; these may have changed through time, perhaps by oxidation (organic material) or by the formation of secondary carbonates in pore spaces. This leads to complexities resulting from sediment chemical composition; since quartz is the dosimeter, differential absorption of dose in the surrounding non-quartz material renders the infinite matrix assumption invalid, or at least approximate. If the amount of organic or carbonate material is quantified, differential dose rate absorption coefficients may then be used [47], and reported. Table S5 below gives attenuation factors to account for the effect of water and carbonates in infinite matrix conditions, while Table S6 details variations of the water correction factors in well-sorted sediment as a function of grain size.

To illustrate our suggestions, we provide Table S7(a,b); this represents our view of the minimum numerical reporting that allows the transparency required. The various underlying assumptions and the factors used should be given in the main text or as footnotes, together with any other relevant data (particularly the dose recovery summary).

For the reader to follow the details of an age calculation, and understand how the various effects and parameters are taken into account in practice, an age calculation spreadsheet is provided as Supplementary Material. This is not proposed as a standard data processing option (there are many more sophisticated software packages available) but it does provide a transparent and practical

² The age calculation spreadsheet provided here allows the calculation of cosmic dose rates as a function of latitude, altitude and burial depth following [3].

implementation of age calculation, parameterized functions to estimate cosmic dose rates and correction factors for the effect of water, grain size attenuation factors, etc.

(a) Uncertainty analysis of luminescence data follows guidelines such as those of the *Bureau International des Poids et Mesures* (the intergovernmental organisation through which Member States act together on matters related to measurement science and measurement standards). The Bureau's guide on the expression of uncertainty in measurement distinguishes between random and systematic uncertainties [50]. The measurement error is defined as the difference between the measured, or observed value, and the true value (measurand); it is unknown (since the true value is unknown) but it can be characterised by a probability density. Repetition of a measurement leads to random fluctuations of the observed values (and thus characterises random uncertainties), while the use of a given instrument, calibrated with a given standard, may lead to systematic bias in measurements. In other words, the scale of analysis dictates whether an uncertainty is random or systematic - for example, uncertainties that are systematic within each of several laboratories (e.g. calibration of a gamma spectrometer), will give rise to random fluctuations at the scale of an intercomparison study involving different laboratories (Table S8).

In both cases (systematic and random), provided everything has been done to reduce systematic uncertainties, both systematic and random errors may be assumed to follow a normal distribution. Table S8 lists the main parameters needed to calculate an OSL age (other than measurement uncertainties, which can, in principle, be reduced arbitrarily by simply making more measurements) and the typical standard deviation of the error, as well as comments on its nature (systematic or random).

Formally, one may write for all quantities at stake:

$$\tilde{x} = x + e$$

where \tilde{x} is the measurement or observed value, x the true value and e the measurement error. We then introduce σ , the standard deviation of e (and \tilde{x}). For example, if we measure the equivalent dose on n aliquots, we generally approximate σ by the standard deviation of the D_e values, divided by the square root of n . Many quantities must be determined and added, multiplied or divided to finally obtain an age; for uncertainty propagation, if $z = x + y$ (or $z = x - y$), it is standard practice to approximate σ_z by $\sqrt{\sigma_x^2 + \sigma_y^2}$. If $z = y/x$ (or $z = yx$), then $\frac{\sigma_z}{z}$ is approximated by $\sqrt{\left(\frac{\sigma_x}{x}\right)^2 + \left(\frac{\sigma_y}{y}\right)^2}$.

Although there is no formal convention, OSL ages are almost exclusively reported with an uncertainty of one standard deviation of the error, i.e. the interval $[A - \sigma_A; A + \sigma_A]$ is the 68% confidence interval.

Figure S1. Cosmic dose rate as a function of burial depth (left) and altitude (right) (based on [3]).

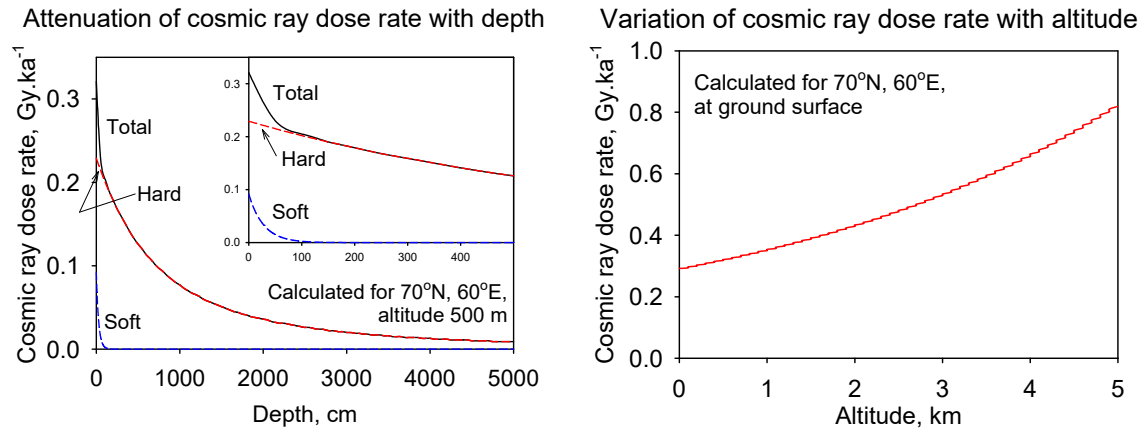


Figure S2. Schematic overview of the laboratory treatment to extract quartz grains from the sample. All these steps should be performed under subdued lighting conditions (using light of wavelength and intensity without a significant effect on the trapped charge population [51]). Q: quartz, H: heavy minerals, F: feldspar, NF: sodium-rich feldspar, KF: potassium-rich feldspar. The combined water drops [💧] and fan symbol [🌀] indicate thorough washing and drying.

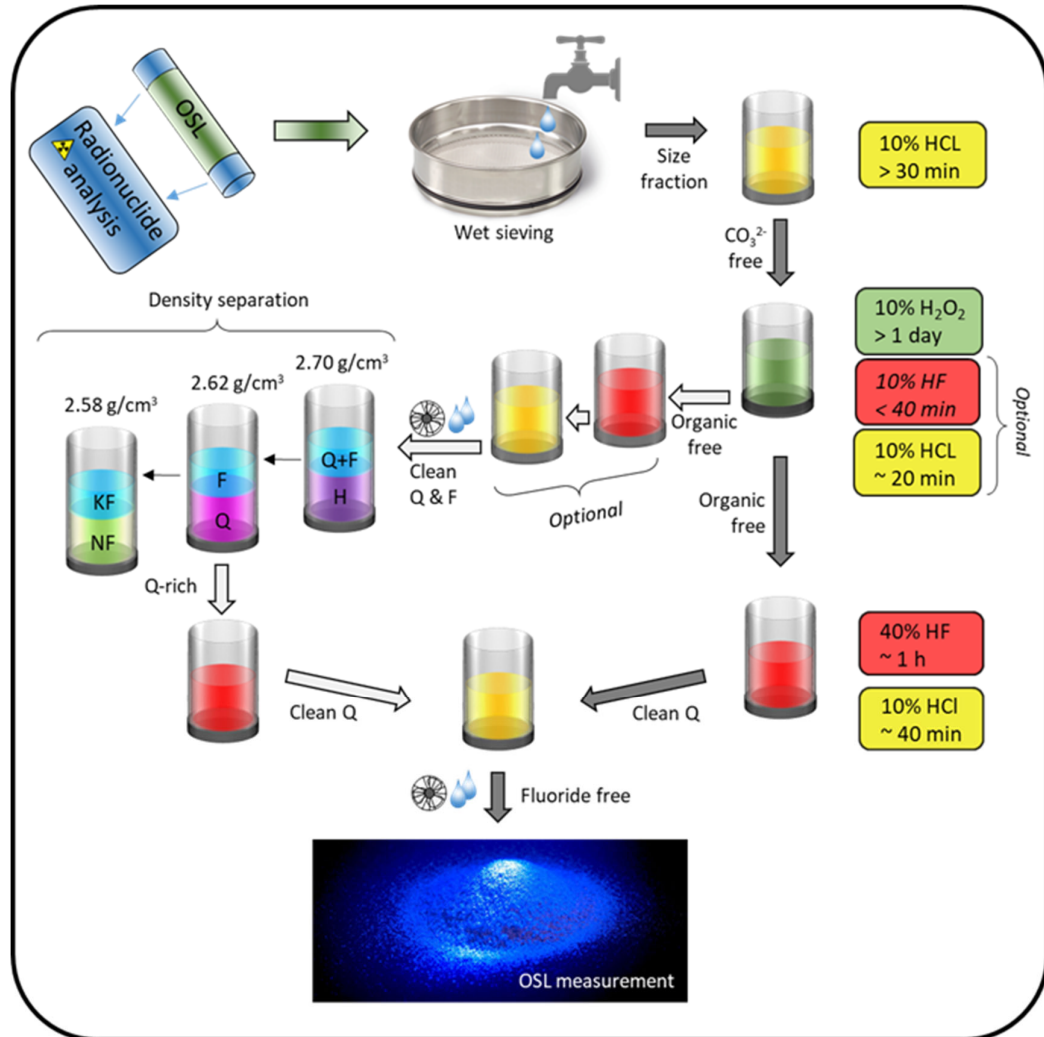


Figure S3. Radioactive decay chains of ^{238}U (left) and ^{232}Th (right). Long-lived radioisotopes of the same element are marked with rings of one colour. In secular equilibrium, the activity of each radionuclide is almost identical and follows the parent decay rate. While the ^{232}Th chain is unlikely to suffer from disequilibria (because of the short half lives of all of its progeny), the ^{238}U chain may be affected by ^{222}Rn escape (^{222}Rn is a gas and the half-life of several days makes it possible for it to move before decaying), or by loss or gain of ^{226}Ra and ^{238}U – both elements can be mobilised or deposited by changes in porewater geochemistry (e.g. salinity, dissolved carbonate, reduction/oxidation conditions).

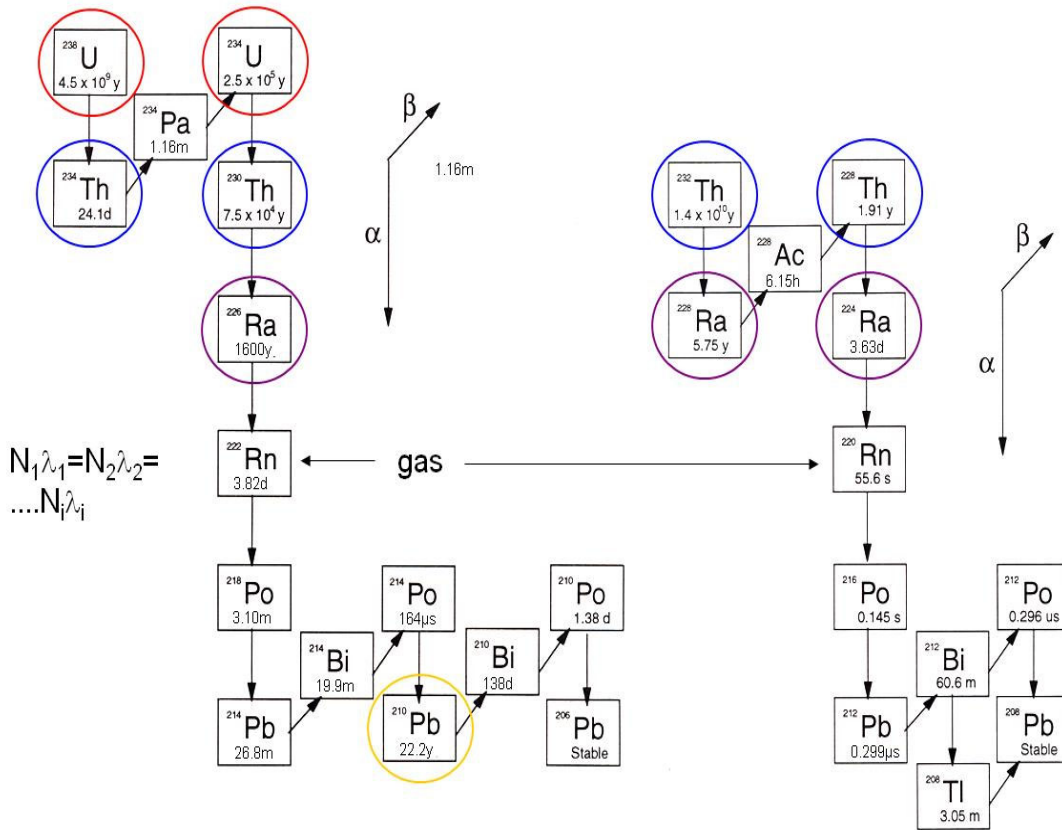


Figure S4. The radionuclides in the decay series are combined in groups reflecting the presumed potential for disequilibria based on half-lives and physico-chemical parameters (nuclides within each group can be deduced by comparison with Figure S3). The relative contribution of these groups to the total dose rate for a sand sample with the activities given in Table S2 is shown as %; these values can give some indication of the potential importance of radioactive disequilibria (bearing in mind that this is most likely to occur in the U series). Unless sediment is unusually rich in U (relative to K and Th), the contribution of the U series to the total dose rate is sufficiently small for most samples to make unlikely errors of more than 5% in the final age arising from disequilibrium.

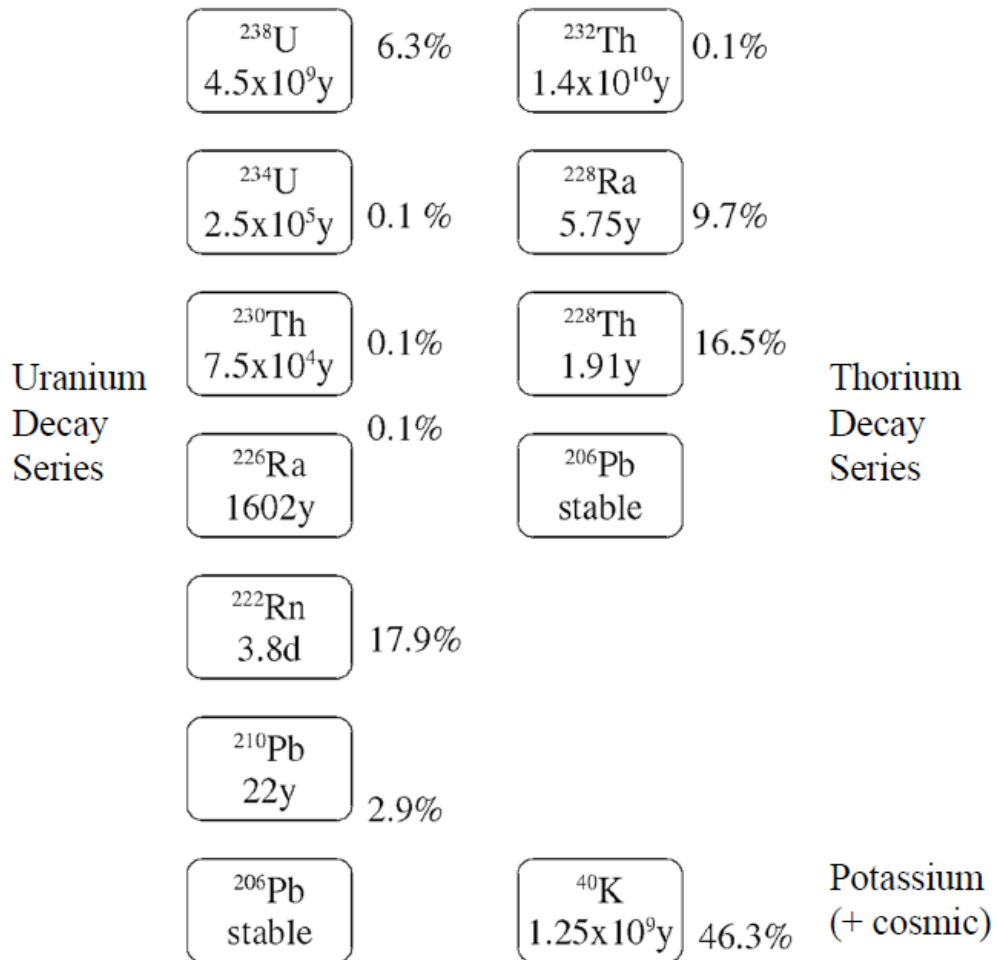
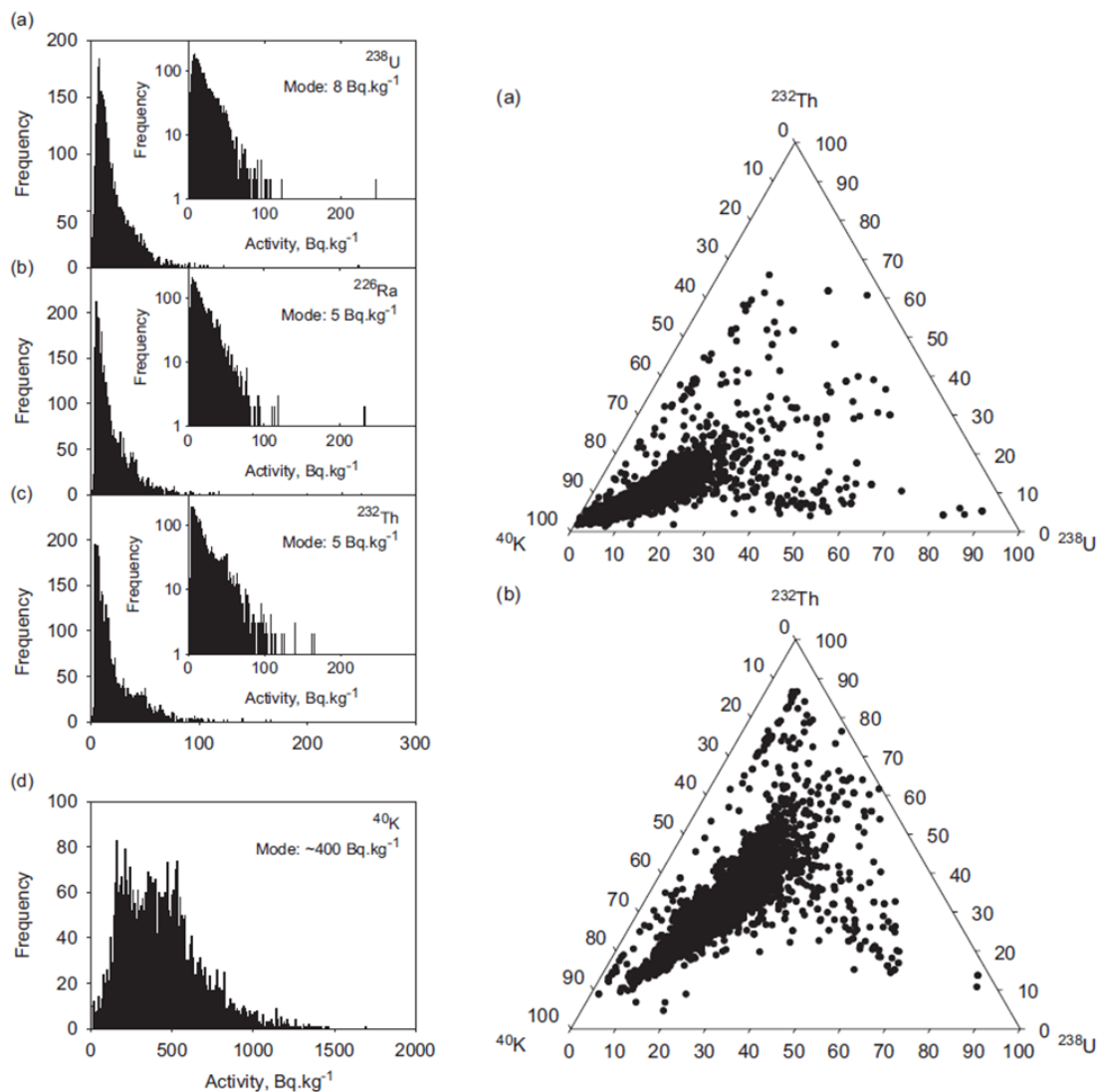


Figure S5 (reproduced from [52]). Variability in radionuclide concentrations in 3578 samples used for luminescence dating. Left – histograms of activity concentrations of ^{238}U (a), ^{226}Ra (b), ^{232}Th (c) and ^{40}K (d). Right – relative contributions to beta (a) and gamma (b) dose rates from ^{238}U , ^{232}Th and ^{40}K . The table summarises the concentration values.

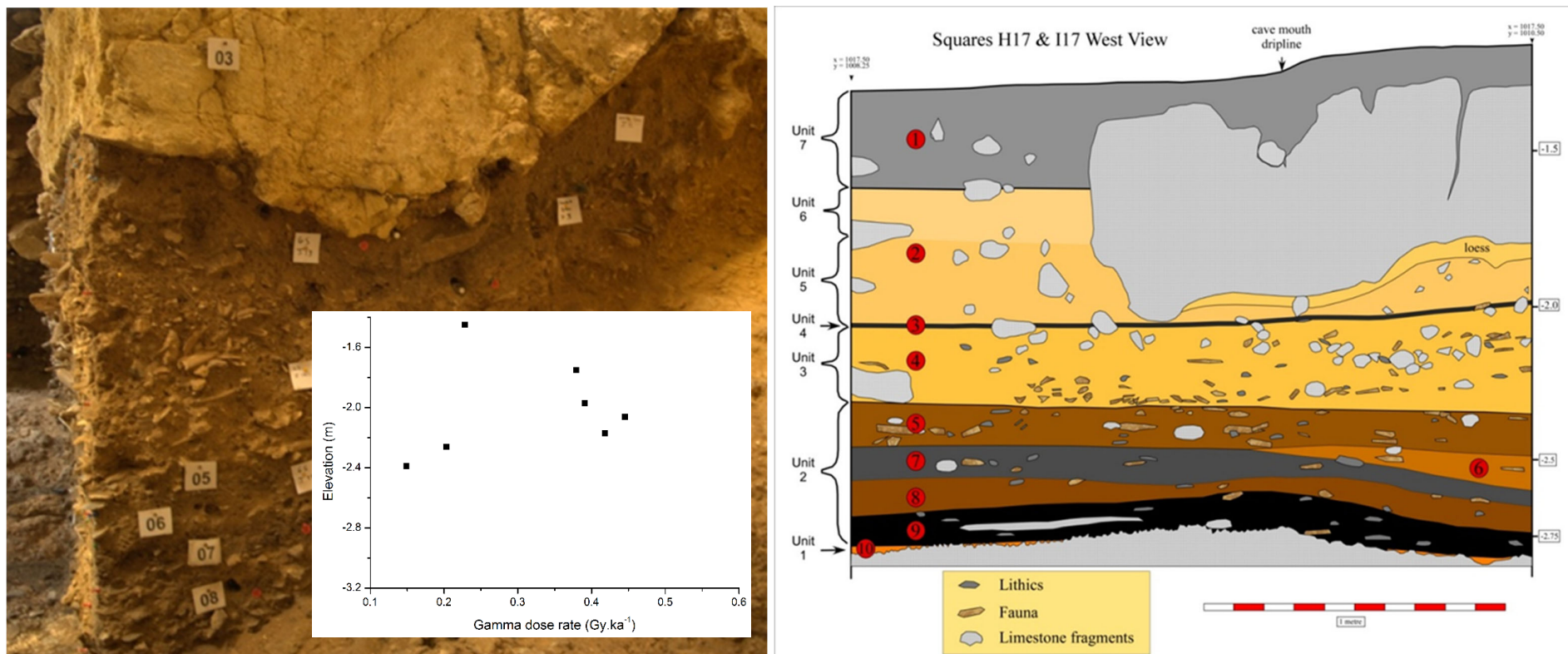


Mode, percentiles and median for the distributions of concentrations of ^{238}U , ^{226}Ra , ^{232}Th and ^{40}K in 3578 samples

	^{238}U	^{226}Ra	^{232}Th	^{40}K
Mode, Bq kg^{-1}	8	5	4	355
0.01-percentile, Bq kg^{-1}	0.20	2.72	2.84	40
Median, Bq kg^{-1}	15.30	14.35	15.34	406
0.99-percentile, Bq kg^{-1}	90.37	86.69	150.4	1182

Figure S6. Examples of heterogeneous dose rate distributions at the Palaeolithic site of Roc de Marsal [53, 54].

- a) Gamma dose rates. The stratigraphic section (left panel) comprises sediment between two calcareous rocky horizons: bedrock at the bottom and collapsed rocks from the cave roof on top; the right panel is a schematic view of the section. The graph (inset) illustrates that gamma dose rates measured in situ with $Al_2O_3:C$ dosimeters show large variations, because the limestone contains very low radionuclide concentrations compared to the sediment. In such cases, dating the sediment at or close to the interface requires careful assessment of the gamma dose rate.



b)

b) Beta dose rates. Beta autoradiography [55] (right panel) allows qualitative estimation of dose rate heterogeneities. In the example shown here, the limestone gravel and effects of dissolution ('Loose brown patch') shown in left panel induce large variations in beta dose rates (right panel). 'Hotspots' appear in the form of small dark circular patches on the right image, in this case presumably K-feldspar grains. Generally low dose rates, poor sorting and coarser grain size distributions tend to indicate greater variability in dose rates [56, 57, 58, 59].

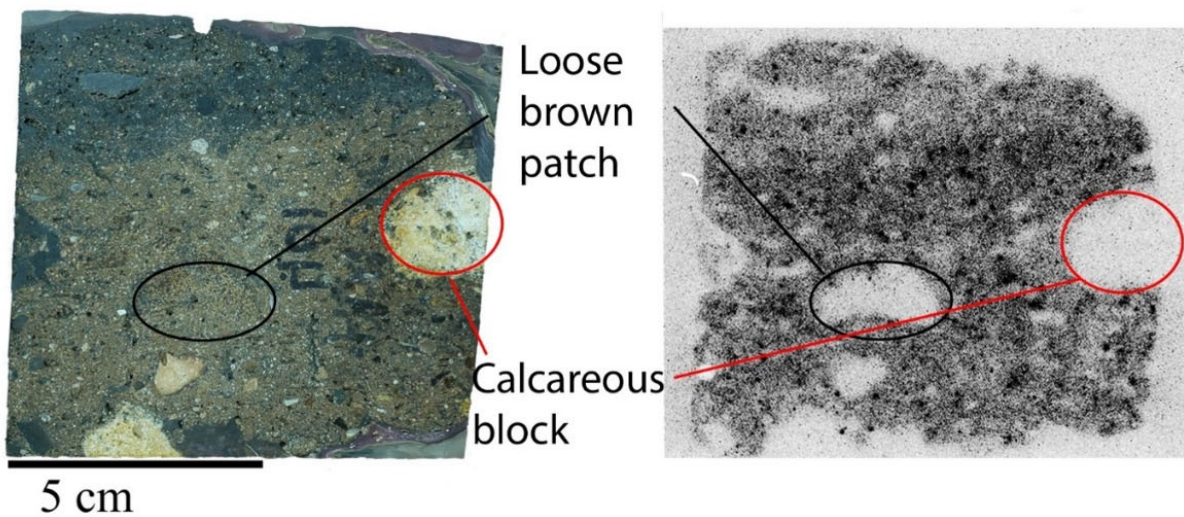


Figure S7. The main components in an OSL measurement system.

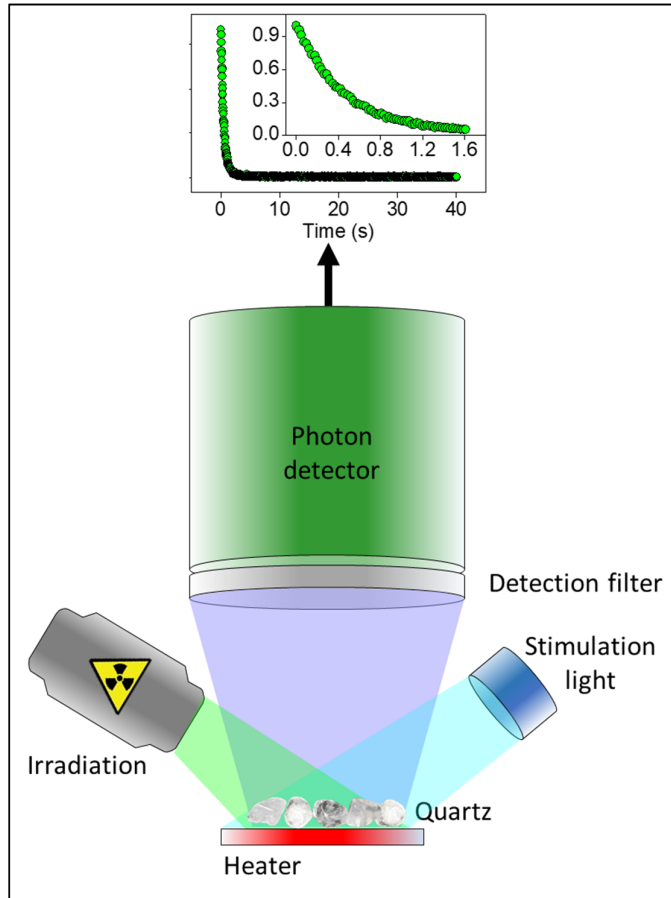


Figure S8. Emission spectra of several sedimentary quartz samples from South Australia obtained using stimulation at 647 nm (reproduced from [60]).

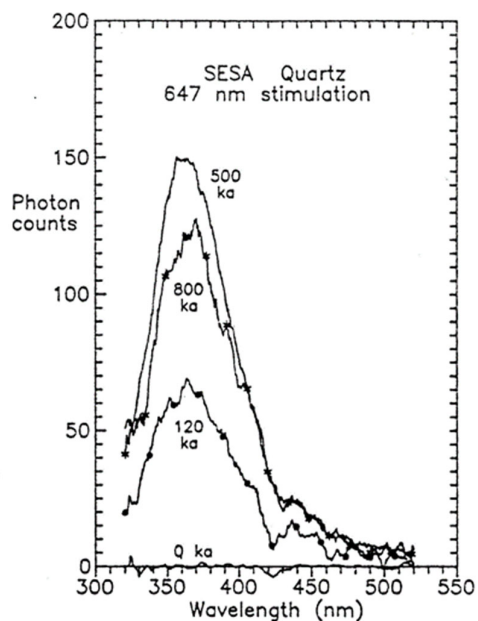


Figure S9. Detection: transmission of 7.5 mm of Hoya U-340 detection filter; Emission: Quartz OSL; Stimulation: emission spectra of typical blue and green LEDs. Cut-off filters: Typical cut-off filters used in front of blue and green stimulation LEDs to more effectively separate the stimulation light from the detection window.

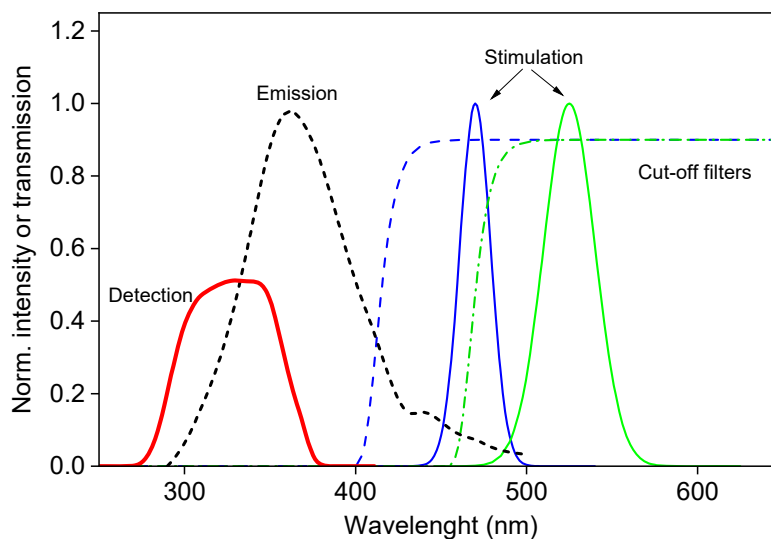


Figure S10. Optical stimulation (excitation) spectrum for a sedimentary quartz annealed at 850°C and detected through the Hoya U-340 glass filter. Reproduced from [61].

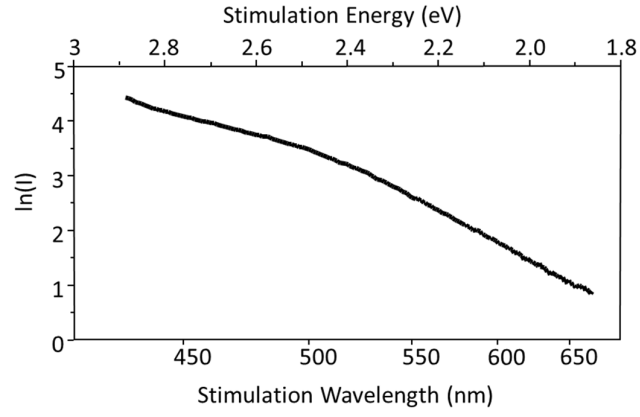


Table S1. Methods for the determination of dose rates. Where relevant (marked with a * below), it is usually desirable to ensure that ^{222}Rn is in equilibrium with its parent ^{226}Ra before measurement.

Method	Output	Comment	References
High-resolution gamma spectrometry*	[K], [U], [Th]	Allows some investigation of disequilibrium. Depending on detector, requires >10 g of sample.	[44], [62], [63]
Low-resolution gamma spectrometry*	[K], [U], [Th]	Slightly less sensitive than high resolution, but does not require cooling. Typically ~200 g sample	[64]
Beta counting*	Beta activity concentration	Conversion from count rate to dose rate is radionuclide dependent. Requires 10s of g sample.	[52], [65]
Alpha counting*	Alpha activity concentration	^{222}Rn difficult to control; lithium borate glassification can be used to ensure homogeneity	[4]; [66]
ICP-MS (Inductively Coupled Plasma-Mass Spectrometry), or OES (Optical Emission Spectrometry) for K	[K], [U], [Th]	Only a few hundred mg of material analysed, so homogenisation is of great importance.	[67] [68]
NAA (Neutron Activation Analysis)	[K], [U], [Th]	Only a few hundred mg of material analysed, so homogenisation is of great importance.	[69] [70]
In situ dosimeters	Gamma and cosmic dose rate at sample location	Requires long storage time on site (6-12 months). Useful in heterogeneous settings at the dm- or m-scale.	[8] [9] [71]
Field gamma spectrometer	effective [K], [U], [Th], or direct gamma dose rate at sample location	Useful when working in heterogeneous settings at the dm- or m-scale.	[12], [13]; [10]; [11]

Table S2. Energy release and dose rates in a) the ^{232}Th decay series, and b) the Uranium (^{238}U and ^{235}U) decay series, and c) Potassium (^{40}K) and Rubidium (^{87}Rb ; only beta dose rates). Adapted from [6]

a) ^{232}Th decay series.

Isotope	Half-life (s)	Alpha		Beta		Gamma	
		E	DR	E	DR	E	DR
^{232}Th	$4.43 \cdot 10^{17}$	4.003	0.0821	0.0113	0.0002	0.0011	0.0000
^{228}Ra	$1.81 \cdot 10^8$	-	-	0.0092	0.0002	0.0004	0.0000
^{228}Ac	$2.21 \cdot 10^4$	-	-	0.4171	0.0086	0.8602	0.0176
^{228}Th	$6.03 \cdot 10^7$	5.406	0.1109	0.0195	0.0004	0.0031	0.0001
^{224}Ra	$3.16 \cdot 10^5$	5.673	0.1164	0.0023	0.0000	0.0104	0.0002
^{220}Rn	$5.56 \cdot 10^1$	6.288	0.1290	-	-	0.0006	0.0000
^{216}Po	$1.45 \cdot 10^{-1}$	6.778	0.1390	-	-	0.0000	0.0000
^{212}Pb	$3.83 \cdot 10^4$	-	-	0.1721	0.0035	0.1437	0.0029
^{212}Bi	$3.63 \cdot 10^3$	2.175	0.0446	0.5034	0.0103	0.1039	0.0021
^{212}Po (0.641)	$2.99 \cdot 10^{-4}$	5.631	0.1155	-	-	-	-
^{208}Tl (0.359)	$1.83 \cdot 10^2$	-	-	0.2140	0.0044	1.2136	0.0249
total			0.7375		0.0277		0.0479
Pre-Rn total			0.3093		0.0094		0.0180

Notes.

1. Energies (“E”) are given in MeV and represent the energy emitted per disintegration.
2. Branching ratios are shown in parenthesis against the radioelements in the branches; associated values given for energy release are after adjustment for branching. Note that the branching also affects the energy release of the radioelement at which the branching occurs; thus the value given for the alpha release by ^{212}Bi is 35.9% of the full energy - because ^{208}Tl is formed by alpha emission from ^{212}Bi .
3. Beta components include Auger electrons and internal conversion; gamma components include X-rays and annihilation radiation; alpha recoil and neutrinos are not included due to their insignificant contribution to dose-rates (cf. [72]).
5. A dash indicates that no radiation of that type is mentioned by the National Nuclear Data Centre.
6. Dose rate values (“DR”) are given in $\text{Gy}\cdot\text{ka}^{-1}$ per ppm of parent (i.e. mg of parent per kg of sample), assuming equilibrium in the decay chains. The activity of the parent is 4.057 Bq per kg of sample.
7. The rows labelled ‘pre-Rn’ give the values for 100% escape of radon.
8. Relative differences are calculated between this paper and values from [72].
9. ^{216}At has been omitted since its contribution to the total energy is insignificant.

Table S2 continued.

b) Uranium (^{238}U and ^{235}U) decay series.

Isotope	Half-life (s)	Alpha			Beta			Gamma		
		E	DR	DR, nat. U	E	DR	DR, nat. U	E	DR	DR, nat. U
^{238}U	$1.41 \cdot 10^{17}$	4.193	0.264	0.262	0.007	0.0005	0.0004	0.001	0.0001	0.0001
^{234}Th	$2.08 \cdot 10^6$	-	-	-	0.059	0.0037	0.0037	0.008	0.0005	0.0005
$^{234}\text{Pa}_m$	$6.95 \cdot 10^1$	-	-	-	0.810	0.0509	0.0506	0.016	0.0010	0.0010
$^{234}\text{Pa}_{(0.0016)}$	$2.41 \cdot 10^4$	-	-	-	0.001	0.0001	0.0001	0.001	0.0001	0.0001
^{234}U	$7.75 \cdot 10^{12}$	4.759	0.299	0.297	0.012	0.0007	0.0007	0.001	0.0001	0.0001
^{230}Th	$2.38 \cdot 10^{12}$	4.664	0.293	0.291	0.013	0.0008	0.0008	0.001	0.0001	0.0001
^{226}Ra	$5.05 \cdot 10^{10}$	4.775	0.300	0.298	0.004	0.0002	0.0002	0.007	0.0005	0.0005
^{222}Rn	$3.30 \cdot 10^5$	5.489	0.345	0.343	-	-	-	0.000	0.0000	0.0000
^{218}Po	$1.86 \cdot 10^2$	6.001	0.377	0.375	-	-	-	-	-	-
^{214}Pb	$1.61 \cdot 10^3$	-	-	-	0.291	0.0183	0.0182	0.239	0.0150	0.0149
^{214}Bi	$1.19 \cdot 10^3$	0.001	0.000	0.000	0.654	0.0411	0.0408	1.475	0.0928	0.0921
^{214}Po	$1.64 \cdot 10^{-4}$	7.687	0.483	0.480	-	-	-	0.000	0.0000	0.0000
^{210}Pb	$7.01 \cdot 10^8$	-	-	-	0.033	0.0021	0.0021	0.005	0.0003	0.0003
^{210}Bi	$4.33 \cdot 10^5$	-	-	-	0.389	0.0245	0.0243	-	-	-
^{210}Po	$1.20 \cdot 10^7$	5.304	0.333	0.331	0.000	0.0000	0.0000	0.000	0.0000	0.0000
^{238}U total			2.695	2.676		0.1429	0.1419		0.1104	0.1096
^{238}U Pre-Rn total			1.156	1.148		0.0570	0.0566		0.0022	0.0022
^{235}U	$2.22 \cdot 10^{16}$	4.114	1.663	0.012	0.029	0.0117	0.0001	0.164	0.0665	0.0005
^{231}Th	$2.20 \cdot 10^6$	-	-	-	0.146	0.0591	0.0004	0.023	0.0094	0.0001
^{231}Pa	$1.03 \cdot 10^{12}$	4.924	1.990	0.014	0.032	0.0130	0.0001	0.040	0.0160	0.0001
^{227}Ac	$6.87 \cdot 10^8$	0.070	0.028	0.000	0.012	0.0049	0.0000	0.001	0.0002	0.0000
$^{227}\text{Th}_{(0.986)}$	$1.61 \cdot 10^6$	5.808	2.347	0.017	0.050	0.0202	0.0001	0.154	0.0621	0.0004
$^{223}\text{Fr}_{(0.014)}$	$1.32 \cdot 10^3$	0.005	0.002	0.000	0.000	0.0002	0.0000	0.001	0.0003	0.0000
^{223}Ra	$9.88 \cdot 10^5$	5.664	2.289	0.016	0.068	0.0275	0.0002	0.135	0.0546	0.0004
^{219}Rn	$3.96 \cdot 10^0$	6.753	2.729	0.019	0.007	0.0027	0.0000	0.058	0.0235	0.0002
^{215}Po	$1.78 \cdot 10^{-3}$	7.392	2.987	0.021	-	-	-	-	-	-
^{211}Pb	$2.17 \cdot 10^3$	-	-	-	0.450	0.1817	0.0013	0.064	0.0258	0.0002
^{211}Bi	$1.28 \cdot 10^2$	6.549	2.647	0.019	0.013	0.0053	0.0000	0.047	0.0191	0.0001
^{211}Po	$5.16 \cdot 10^{-1}$	0.021	0.008	0.000	-	-	-	-	-	-
^{207}Tl	$2.86 \cdot 10^2$	-	-	-	0.495	0.2002	0.0014	0.002	0.0009	0.0000
^{235}U total			16.690	0.1185		0.5265	0.0037		0.2807	0.0020
total				2.795			0.1457			0.1116
Pre-Rn total				1.267			0.0603			0.0042

Notes.

1. See notes 1-8 of Table S2(a).
2. The mass abundances used in the natural Uranium calculations for ^{238}U and ^{235}U (respectively 99.29% and 0.71%) correspond to the natural atomic abundances of 99.28% and 0.72% respectively.
3. The activity of the parent (per ppm of parent) is 12.44 Bq per kg of sample for ^{238}U , 79.94 for ^{235}U and 12.92 for natural Uranium.
4. The rows labelled 'pre-Rn' give the values for 100% escape of radon in the case of ^{238}U series, but because of the short half-life of ^{219}Rn the values given for natural Uranium include contributions of that gas and its daughters.
5. ^{218}At , ^{218}Rn , ^{210}Tl , ^{206}Tl and ^{215}At have been omitted since their contribution to the total is insignificant.

Table S2 continued.

c) Dose-rate data for Potassium and Rubidium.

		⁴⁰ K	⁸⁷ Rb
Natural abundance (mg.g ⁻¹)		0.119	283
Half-life (Ga)		1.248	48.1
Average energy per disintegration (MeV)	Beta	0.499	0.0817
	Gamma	0.1557	
Specific activity (Bq.kg ⁻¹) for concentration of 1% nat. K and 50 ppm of nat. Rb	Total	316.4	44.8
	Beta	282.5	44.8
	Gamma	33.73	
Dose-rate (Gy.ka ⁻¹) for concentrations as above	Beta	0.7982	0.0185
	Gamma	0.2491	

Notes.

1. The energy given for Potassium is that released per disintegration, i.e. after allowance for branching between beta and gamma (89.28% and 10.72% respectively).
2. The contents given in row 1 correspond to natural atomic abundances of 116.7 ppm and 27.8%.

Table S3. Typical concentrations of radionuclides in a well-sorted sand, and the derived infinite matrix dose rates (without grain size attenuation taken into account). In this case, ^{40}K alone contributes about 55% to the total, and the gamma dose rate about 35%.

Radio-nuclide	Activity Bq.kg ⁻¹	Concentration	Beta dose rate, Gy.ka ⁻¹	Gamma dose rate, Gy.ka ⁻¹
^{40}K	300	~1%	0.76	0.24
^{238}U	20	~1.5 ppm	0.23	0.17
^{232}Th	20	~5 ppm	0.14	0.24
Total:			1.12	0.65

Table S4. Beta self-dose values (ϕ) used to derive attenuation factors ($1 - \phi$) following the superposition theorem [73]. Values were calculated with Geant4 for the ^{232}Th decay series, the Uranium (^{238}U and ^{235}U) decay series (all in secular equilibrium), and ^{40}K , for two different minerals: quartz and potassium feldspar.

GEANT4						
Grain size (ϕ , μm)	Potassium		Uranium		Thorium	
	Quartz ($d=2.65$ g.cm^{-3})	KAlSi ₃ O ₈ ($d=2.60$ g.cm^{-3})	Quartz ($d=2.65$ g.cm^{-3})	KAlSi ₃ O ₈ ($d=2.60$ g.cm^{-3})	Quartz ($d=2.65$ g.cm^{-3})	KAlSi ₃ O ₈ ($d=2.60$ g.cm^{-3})
20	0.007	0.007	0.038	0.037	0.048	0.047
40	0.014	0.014	0.057	0.056	0.077	0.075
60	0.022	0.021	0.072	0.07	0.098	0.097
80	0.029	0.028	0.084	0.082	0.117	0.115
100	0.037	0.035	0.095	0.093	0.134	0.131
120	0.044	0.042	0.105	0.103	0.149	0.146
140	0.052	0.05	0.115	0.113	0.163	0.16
160	0.059	0.057	0.125	0.122	0.176	0.173
180	0.067	0.064	0.134	0.131	0.188	0.185
200	0.075	0.072	0.143	0.14	0.2	0.196
250	0.095	0.091	0.165	0.161	0.225	0.221
300	0.114	0.11	0.185	0.181	0.247	0.243
400	0.154	0.148	0.223	0.217	0.286	0.281
600	0.231	0.223	0.289	0.282	0.35	0.343
800	0.304	0.294	0.346	0.338	0.403	0.395
1000	0.372	0.36	0.395	0.387	0.449	0.441

Table S5. Water and carbonate correction factors for a) beta radiation and b) gamma radiation.
 Reproduced from [47].

a) The water and carbonate correction factors for beta radiation.

Water content (m_w/m_s)	Carbonate content (m_c/m_s)							
	0		0.04		0.20		1.00	
0	–		K	0.99	K	0.99	K	0.99
			Th	0.99	Th	0.99	Th	0.99
			U	0.99	U	0.99	U	0.99
0.04	K	1.19	K	1.09	K	1.03	K	1.00
	Th	1.20	Th	1.09	Th	1.03	Th	1.00
	U	1.19	U	1.09	U	1.02	U	1.00
0.20	K	1.20	K	1.16	K	1.09	K	1.02
	Th	1.20	Th	1.17	Th	1.09	Th	1.02
	U	1.20	U	1.16	U	1.09	U	1.02
1.00	K	1.19	K	1.18	K	1.15	K	1.09
	Th	1.19	Th	1.18	Th	1.16	Th	1.09
	U	1.19	U	1.18	U	1.15	U	1.08

The water and carbonate content of the sediment is given as mass (m_w and m_c , respectively) per mass sediment (m_s). The factors are tabulated for independent determination of water and carbonate. The use of these tables requires the assessment of the mass of the *dry* sediment (the fraction having no water or carbonate). It is suggested that intermediate values are calculated using log interpolation.

b) The water and carbonate correction factors for gamma radiation.

Water content (m_w/m_s)	Carbonate content (m_c/m_s)							
	0		0.04		0.20		1.00	
0	–		K	1.02	K	1.02	K	1.01
			Th	1.04	Th	1.04	Th	1.03
			U	1.04	U	1.04	U	1.04
0.04	K	1.06	K	1.04	K	1.03	K	1.02
	Th	1.02	Th	1.03	Th	1.04	Th	1.03
	U	1.02	U	1.03	U	1.04	U	1.04
0.20	K	1.06	K	1.05	K	1.04	K	1.02
	Th	1.02	Th	1.02	Th	1.03	Th	1.03
	U	1.01	U	1.02	U	1.03	U	1.04
1.00	K	1.04	K	1.04	K	1.04	K	1.04
	Th	0.99	Th	0.99	Th	1.00	Th	1.03
	U	0.98	U	0.98	U	1.01	U	1.03

The water and carbonate content of the sediment is given as mass (m_w and m_c , respectively) per mass sediment (m_s).

Table S6 (reproduced from [48]). Water correction factors for gamma radiation obtained using Monte Carlo simulations for the three radioelements found in sediments, as a function of the packing system and grain size.

Grain radius (μm)	Cubic simple			Body-centred cubic			Face-centred cubic		
	K	U	Th	K	U	Th	K	U	Th
20	1.181 <i>0.002</i>	1.157 <i>0.017</i>	1.164 <i>0.019</i>	1.194 <i>0.008</i>	1.176 <i>0.030</i>	1.185 <i>0.034</i>	1.205 <i>0.002</i>	1.193 <i>0.034</i>	1.188 <i>0.032</i>
40	1.165 <i>0.002</i>	1.132 <i>0.016</i>	1.137 <i>0.019</i>	1.184 <i>0.010</i>	1.160 <i>0.027</i>	1.163 <i>0.027</i>	1.198 <i>0.002</i>	1.176 <i>0.027</i>	1.173 <i>0.033</i>
60	1.154 <i>0.003</i>	1.112 <i>0.015</i>	1.118 <i>0.021</i>	1.177 <i>0.011</i>	1.152 <i>0.024</i>	1.167 <i>0.022</i>	1.189 <i>0.003</i>	1.165 <i>0.032</i>	1.169 <i>0.038</i>
80	1.146 <i>0.003</i>	1.102 <i>0.014</i>	1.105 <i>0.017</i>	1.170 <i>0.013</i>	1.134 <i>0.024</i>	1.137 <i>0.017</i>	1.177 <i>0.003</i>	1.138 <i>0.030</i>	1.153 <i>0.031</i>
100	1.140 <i>0.003</i>	1.086 <i>0.014</i>	1.104 <i>0.019</i>	1.164 <i>0.014</i>	1.128 <i>0.025</i>	1.139 <i>0.032</i>	1.173 <i>0.003</i>	1.139 <i>0.029</i>	1.154 <i>0.045</i>
150	1.131 <i>0.003</i>	1.071 <i>0.017</i>	1.085 <i>0.016</i>	1.154 <i>0.016</i>	1.113 <i>0.027</i>	1.127 <i>0.036</i>	1.162 <i>0.004</i>	1.116 <i>0.032</i>	1.137 <i>0.030</i>
200	1.124 <i>0.004</i>	1.061 <i>0.015</i>	1.074 <i>0.016</i>	1.144 <i>0.018</i>	1.094 <i>0.022</i>	1.106 <i>0.028</i>	1.156 <i>0.004</i>	1.095 <i>0.031</i>	1.111 <i>0.038</i>
300	1.115 <i>0.005</i>	1.058 <i>0.015</i>	1.073 <i>0.017</i>	1.137 <i>0.02</i>	1.086 <i>0.025</i>	1.095 <i>0.027</i>	1.147 <i>0.005</i>	1.101 <i>0.032</i>	1.107 <i>0.033</i>
400	1.109 <i>0.005</i>	1.044 <i>0.014</i>	1.054 <i>0.017</i>	1.129 <i>0.023</i>	1.082 <i>0.023</i>	1.087 <i>0.035</i>	1.148 <i>0.005</i>	1.106 <i>0.029</i>	1.112 <i>0.038</i>
500	1.106 <i>0.006</i>	1.042 <i>0.017</i>	1.053 <i>0.016</i>	1.127 <i>0.025</i>	1.062 <i>0.022</i>	1.082 <i>0.031</i>	1.143 <i>0.006</i>	1.088 <i>0.029</i>	1.084 <i>0.031</i>

Table S7. Example tables containing suggested minimum information that should be presented in a publication containing quartz OSL luminescence ages.

a)

Lab.code	^{238}U , Bq.kg $^{-1}$	^{226}Ra , Bq.kg $^{-1}$	^{232}Th , Bq.kg $^{-1}$	^{40}K , Bq.kg $^{-1}$	*beta dose rate, Gy.ka $^{-1}$	*gamma dose rate, Gy.ka $^{-1}$
1	51 ± 12	46.0 ± 1.2	61.0 ± 1.2	562 ± 20	2.29 ± 0.06	1.48 ± 0.04
2	72 ± 12	48.2 ± 1.1	60.6 ± 1.2	566 ± 16	2.32 ± 0.05	1.50 ± 0.04
3	58 ± 9	45.2 ± 0.9	60.2 ± 1.0	581 ± 13	2.33 ± 0.05	1.48 ± 0.04
4	46 ± 8	43.8 ± 0.8	32.2 ± 0.8	158 ± 8	1.05 ± 0.04	0.81 ± 0.04

Note: * dry infinite matrix dose rates

b)

Lab.code	Site	Depth, cm	w.c. %	OSL D_e , Gy	(n_r)	(n_a)	Total dose rate, Gy/ka	OSL age, ka
1	Test site-A	65	16	95 ± 4	1	23	3.16 ± 0.16	30.0 ± 2.0
2	Test site-B	65	16	73 ± 4	0	12	3.19 ± 0.16	22.9 ± 1.7
3	Test site-C	65	16	107 ± 6	0	24	3.20 ± 0.16	33.5 ± 2.5
4	Test site-C	30	11	43 ± 2	1	35	1.79 ± 0.09	23.9 ± 1.7

Note: (a) Mean grain size 215µm. (b) Site altitude 0 km. (c) w.c.: water content. (d) n_r , n_a : number of rejected and accepted aliquots, respectively.

Table S8. Typical dose rate uncertainty budget for a 50 ka sample with dose rate of 2 Gy.ka⁻¹ (neglecting sample measurement uncertainties). Dose rate derived from gamma spectrometry measurements of K,U,Th.

	□Est. Relative Uncertainty %	Absolute uncertainty, ka	Nature of uncertainty when ages compared with:		
			#OSL ages from same section	#OSL ages from other sites	Non-OSL ages
Saturation water content	2	1	Usually <i>Random</i> , (but likely <i>Systematic</i> if same grain-size distribution in all samples)	<i>Random</i>	<i>Random</i>
Fraction of saturation	4	2	<i>Random</i> (assumed values for each sample chosen independently of each other) and/or <i>Systematic</i> (e.g. if site was wetter than assumed).	<i>Random</i>	<i>Random</i>
Gamma spectrometer calibration	2	1	# <i>Systematic</i> for a single spectrometer (but each spectrometer has its own uncertainty on the counting of the calibration standards)	<i>Systematic</i> for a single gamma spectrometer (but see cell to left)	<i>Systematic</i> for a single gamma spectrometer (but see cell to left)
Conversion - activity to dose rate	2	1	<i>Systematic</i>	<i>Systematic</i>	<i>Systematic</i>
Beta source calibration	2	1	# <i>Systematic</i> for a single source (but each source has its own uncertainty on measurement of the known-dose standard)	<i>Systematic</i> for a single source (but see cell to left)	<i>Systematic</i> for a single source (but see cell to left)
Cosmic ray uncertainty	0.02 Gy.ka ⁻¹ *	0.5	<i>Systematic</i> for given elevation, relative depth and latitude.	<i>Random</i> (unknown depth variation between sites)	<i>Random</i>
Beta attenuation factor	2	1	<i>Systematic</i>	<i>Systematic</i>	<i>Systematic</i>
Internal activity of quartz (assumed 0.02 Gy ka ⁻¹)	0.01 Gy.ka ⁻¹ *	0.03	<i>Systematic</i>	<i>Systematic</i>	<i>Systematic</i>
	Total:	3 (i.e. 6%)			

□ Estimates only

* these two contributions are fixed, rather than relative to the dose rate.

for all OSL ages from the same laboratory (i.e. assuming a single set of calibration materials).

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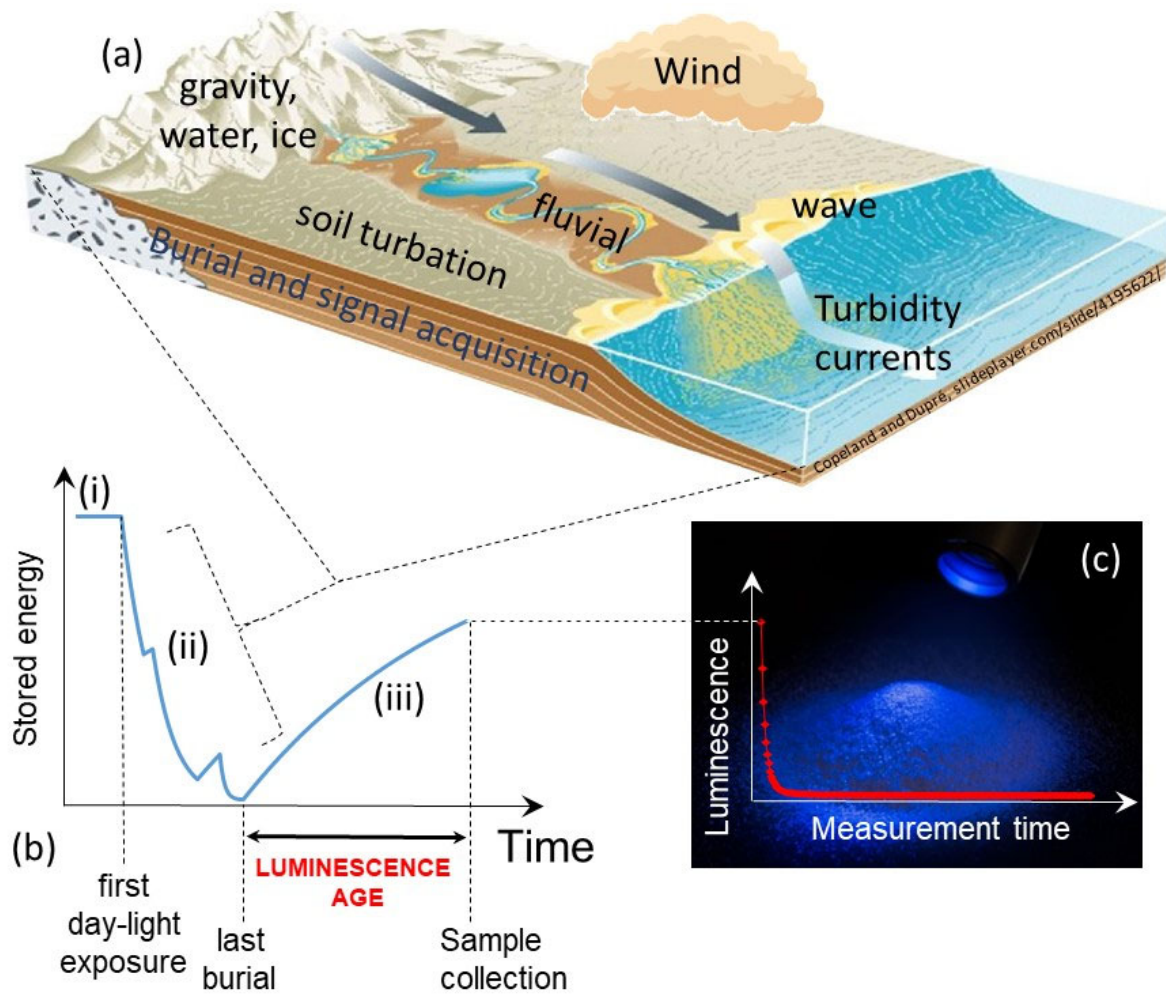


Figure 1: The erosion, transport and sedimentation cycle, and its relationship to the acquisition of a luminescence dating signal. (a) illustrates the various transport agencies that can lead to daylight exposure of mineral grains before final burial*. (b) any existing energy (i) stored in the quartz crystal structure is released by repeated daylight exposure (ii) during erosion and transport by wind, water, ice, gravity or turbation, interspersed with periods of burial. Sufficient net daylight exposure completely removes the previously stored energy. This energy builds up again (iii) during the most recent burial, until the quartz sand grains are sampled and taken to the laboratory. (c) Stimulating the sand grains with blue light releases the stored energy, some of which gives the luminescence signal. This signal decays rapidly with measurement time, as all the energy is released.

*gravity acts as an independent transport agency only on high gradients.

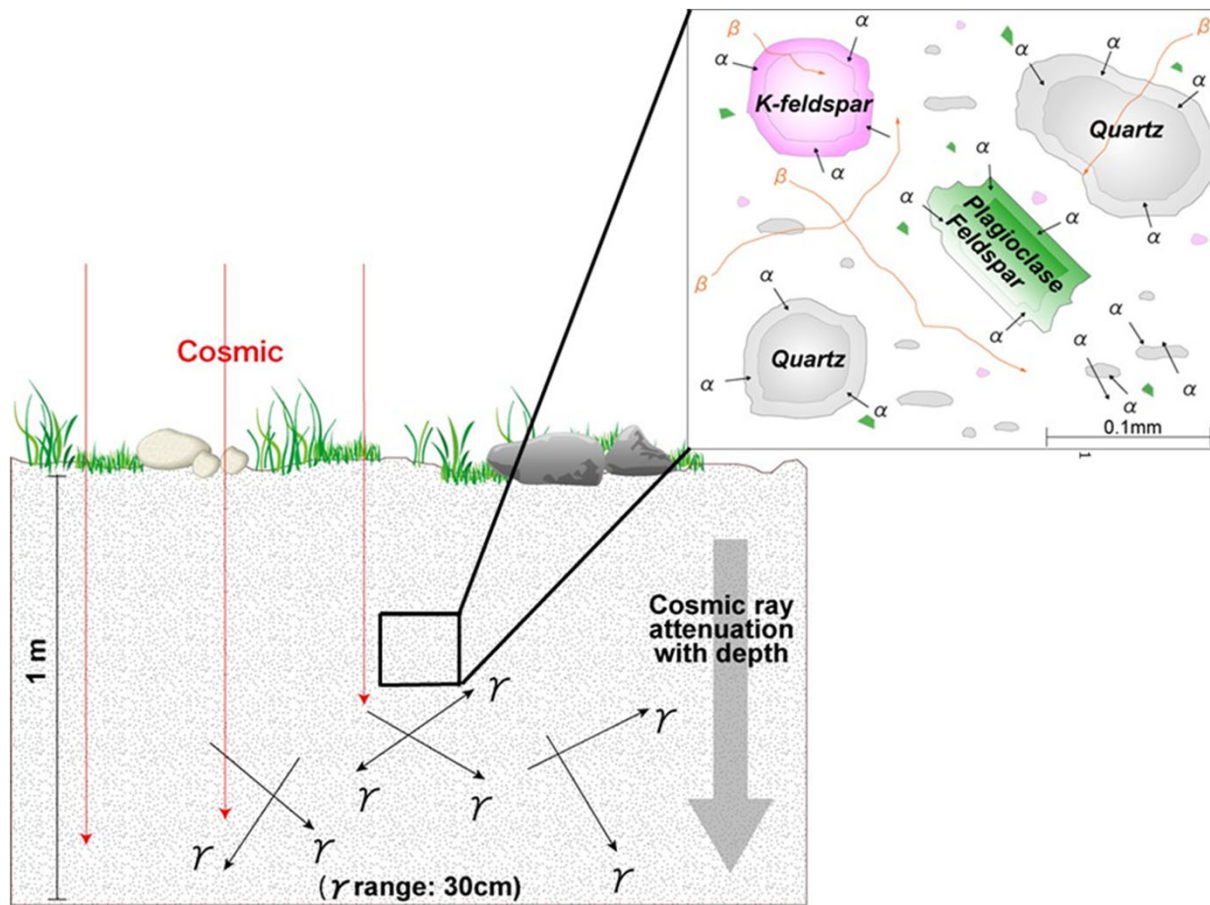


Figure 2. Dose rate components in luminescence dating. Decay of naturally occurring radionuclides (^{238}U , ^{232}Th and ^{40}K) produces ionising radiation (a-particles, b-particles, g-photons), each with a specific range in sediment. A small portion of the dose rate comes from cosmic rays hitting the Earth's surface. Figure redrawn from [206].

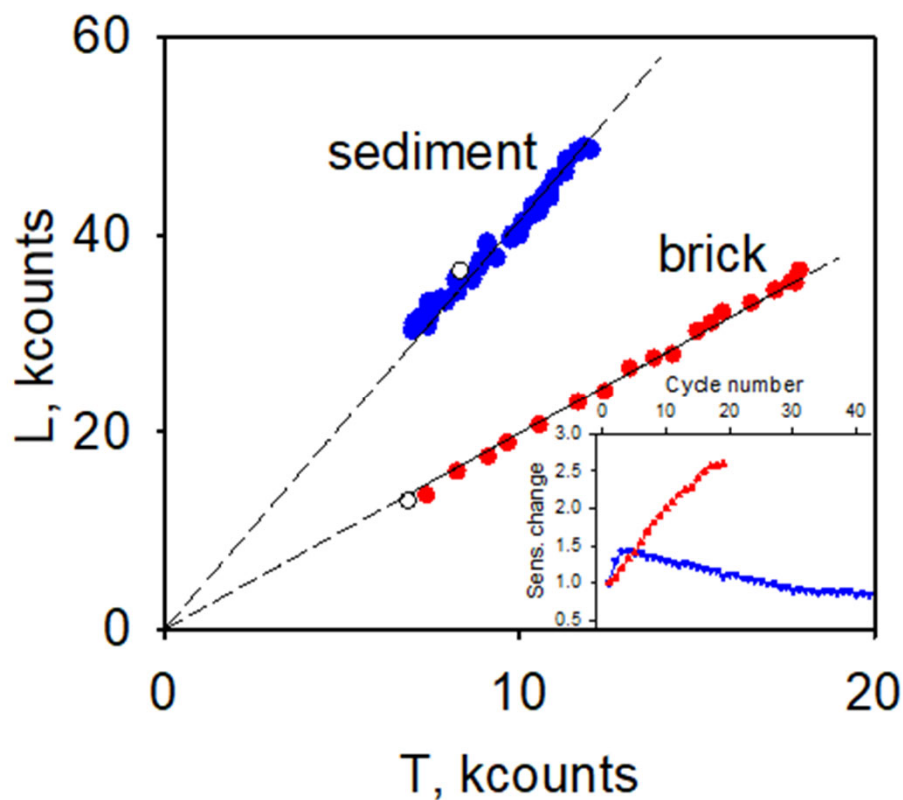


Figure 3. The proportional relationship between L_i and T_i for a large aliquot (containing hundreds of grains) of 180-250 μm quartz extracted from a fired brick (filled red circles, 19 cycles) and a quartz aliquot from a desert sediment (filled blue circles, 42 cycles). The sediment sample comes from the Kalahari Desert in Namibia (lab code: 175405) and the brick from a monastery floor in Sweden (lab code: 173701). The natural signals (L_0) are shown as open circles. Both datasets are consistent with straight lines (dashed) going through the origin, indicating that the ratio L_i/T_i is constant for each sample. The inset shows the sensitivity change as measured by T_i (normalized to first test dose measurement) as a function of measurement cycle. Note that heated quartz typically displays a greater sensitivity change during SAR measurements compared to quartz extracted from sediment. Sediment (175405): $D_i=44.4$ Gy, $D_T=5.9$ Gy. Fired brick (173701): $D_i=3.6$ Gy, $D_T=1.0$ Gy.

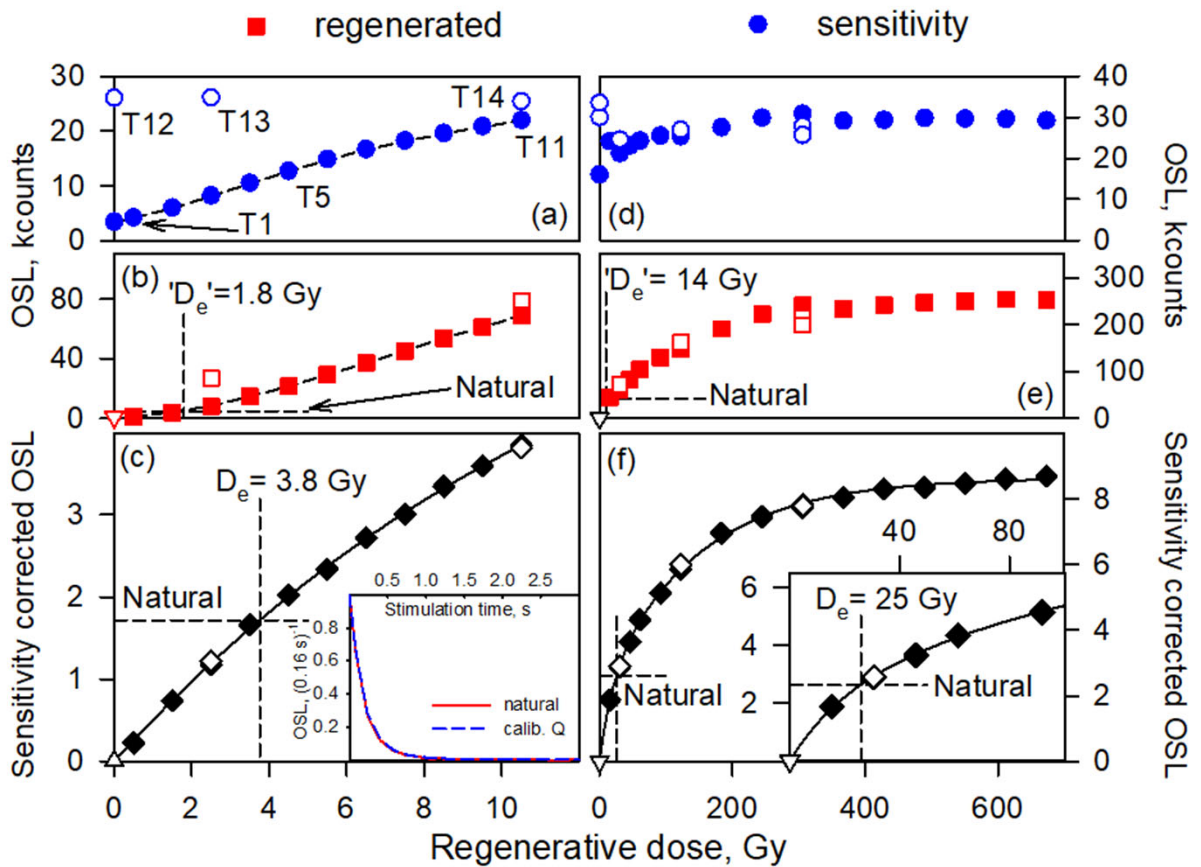


Figure 4. (a) T_i and (b) L_i plotted against dose for an aliquot of heated brick quartz; some T_i are labelled to indicate measurement order. (c) Sensitivity corrected (L_i/T_i) growth curve obtained by dividing data from (b) by data from (a); inset, stimulation curves for natural signal and calibration quartz [36]. (d) - (f) same as (a)-(c) but for quartz extracted from sediment. Open triangles show the response to $D=0$ Gy. Other open symbols indicate repeated dose measurements. Note that without sensitivity correction (b, e) the repeated values of L are inconsistent with earlier measurements at the same dose; correcting for sensitivity change by dividing by T_i corrects this (c, f). The interpolated doses ($'D_e'$ in b,e and D_e in c,f) and growth curve shapes are also significantly different for the uncorrected and sensitivity corrected data. Samples are the same as used in Figure 3.

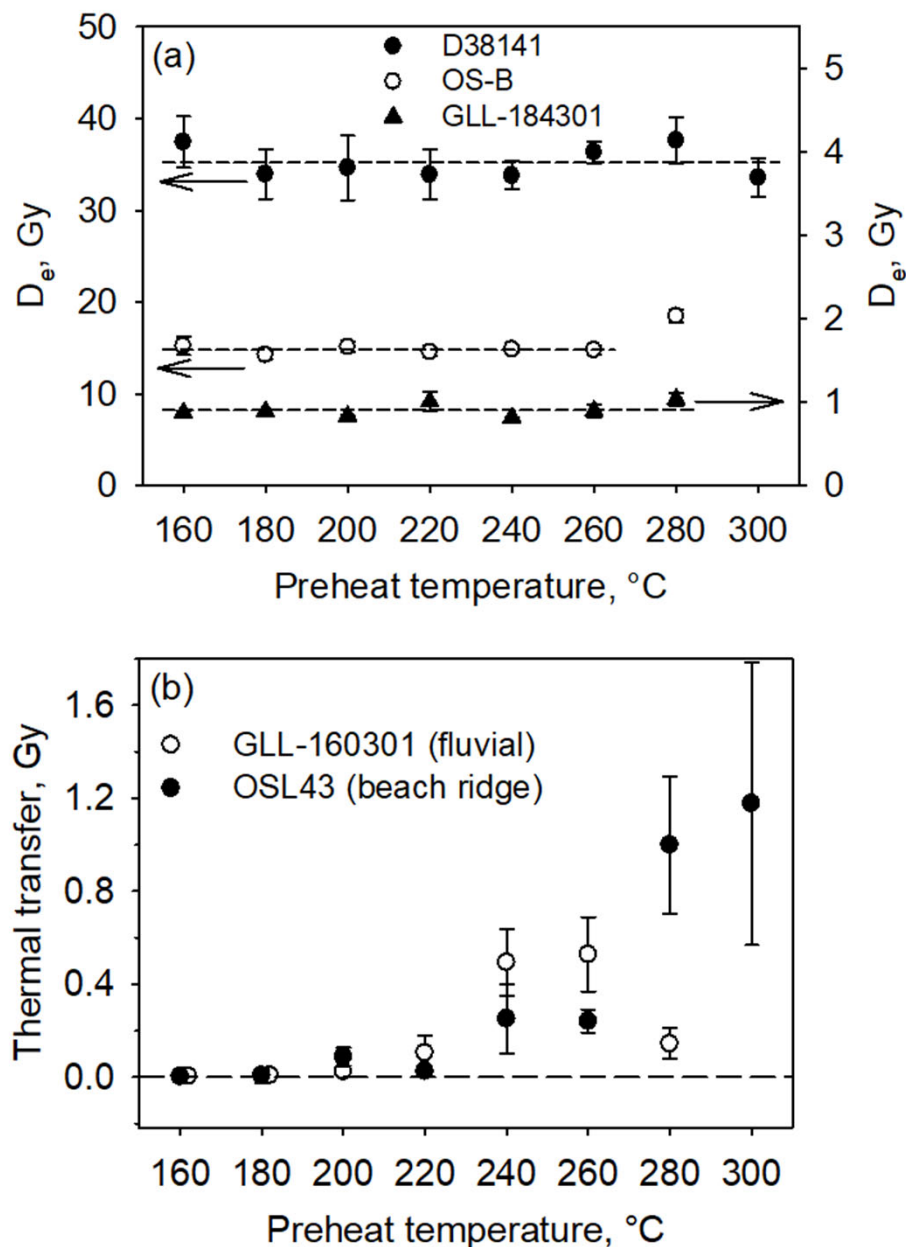


Figure 5. (a) Preheat plateaus for sand-sized quartz extracted from loess (D38141 [60], coversand (OS-B; [61]) and heated sediment from a charcoal kiln (GLL-184301; [259]). For all three samples the D_e is independent of preheat temperature over a wide temperature interval (dashed line). (b) Thermal transfer plateaus for sand-sized quartz extracted from a fluvial sand (GLL-160301; [260]) and a beach ridge (OSL43; [261]). Quartz aliquots were bleached at room temperature twice using blue light stimulation to remove the natural OSL signal before measurement of the thermally transferred dose. The effect of thermal transfer becomes visible for preheats $\geq 200^{\circ}\text{C}$.

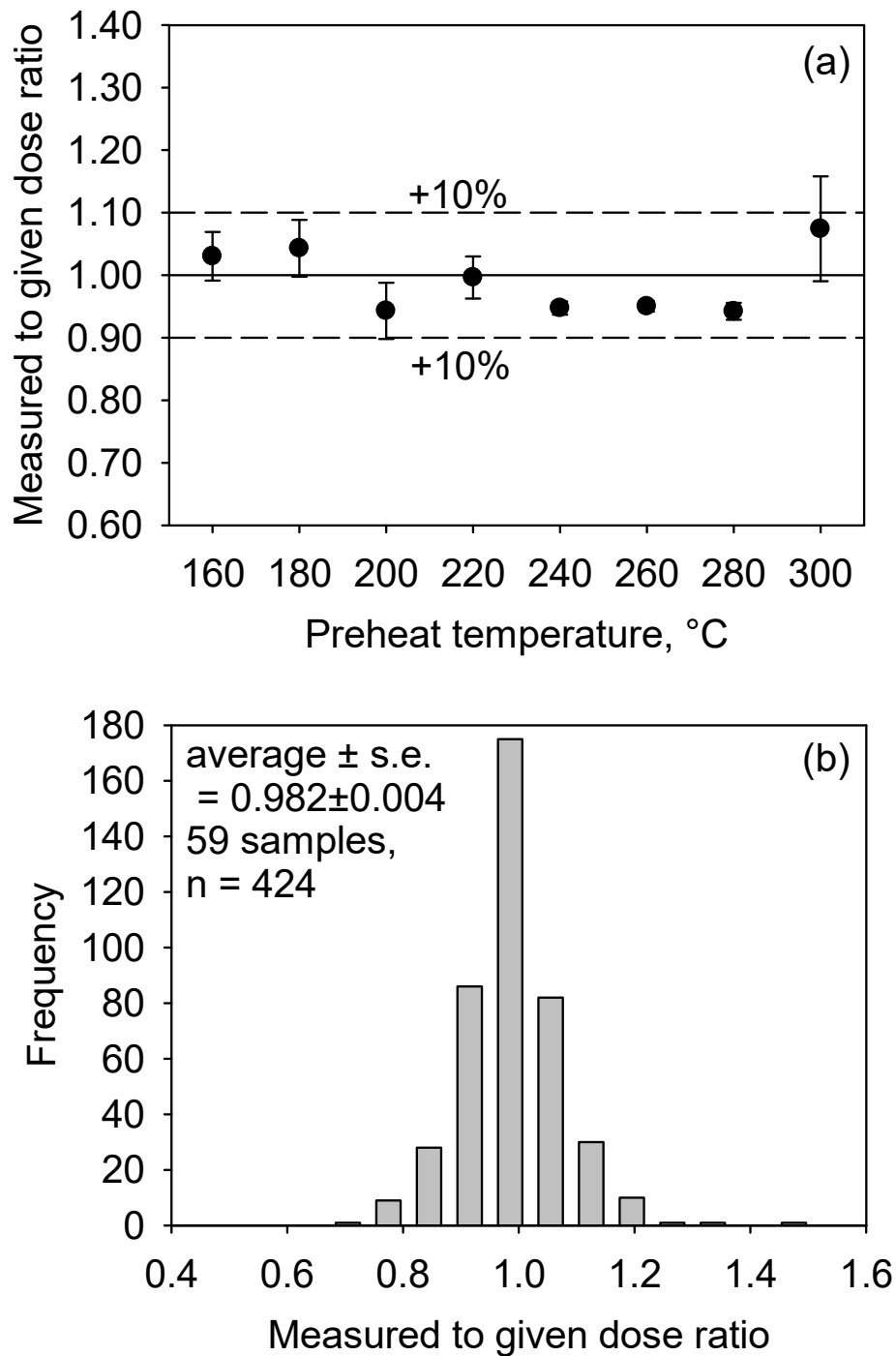


Figure 6. (a) Dose recovery plateau for sand-sized quartz extracted from Chinese loess. The measured to given dose ratio is insensitive to preheat temperature over a wide temperature range. (b) Histogram of dose recovery results measured with a preheat temperature of 260°C. Adapted from [64].

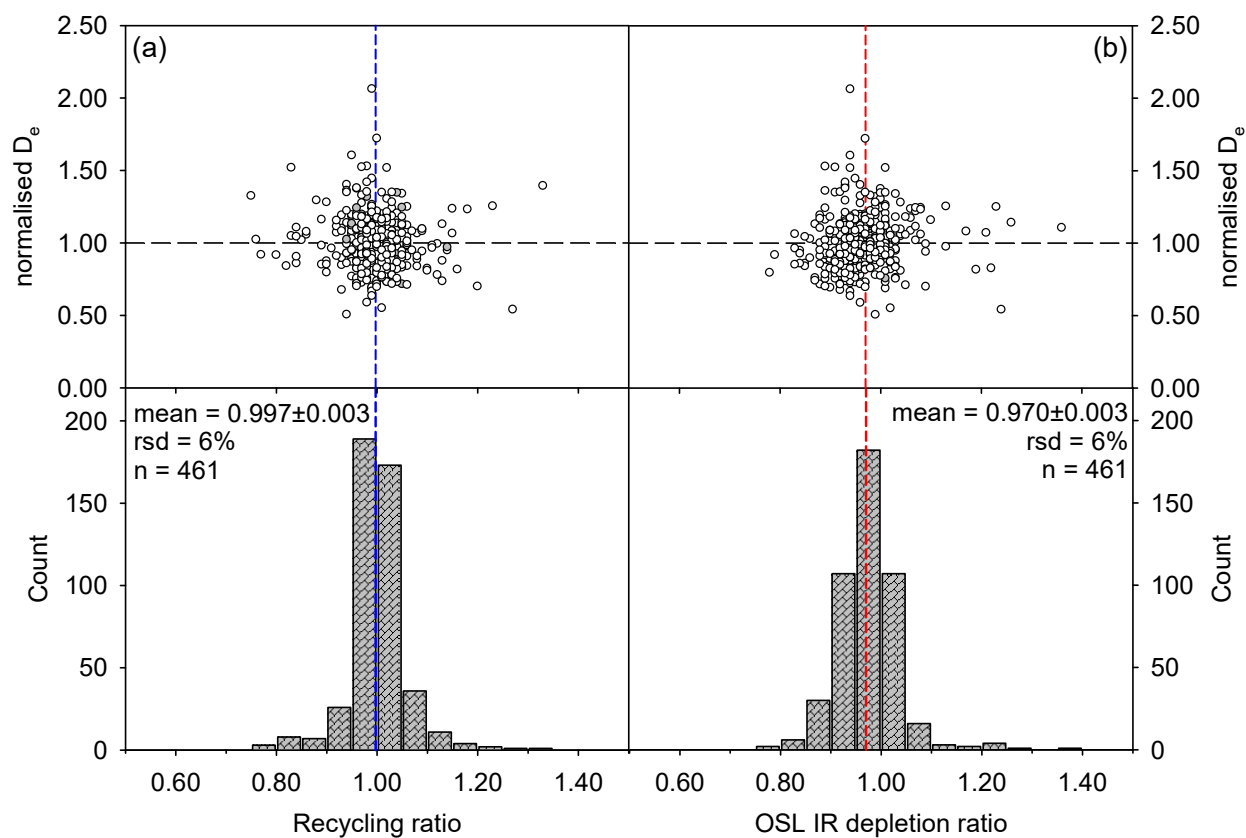


Figure 7. Relationship between large aliquot (containing many hundreds of grains) D_e and recycling ratio (a) or OSL IR depletion ratio (b) for 90-180 μm quartz extracted from Chinese loess. Upper figures show the individual aliquot D_e values (normalized to the average D_e of each sample) as a function of recycling ratio and OSL IR depletion ratio. Lower figures show the recycling and OSL IR depletion ratios as histograms with mean ratios plotted as blue and red dashed lines, respectively. From the upper figures it can be seen that there is no relationship between the measured D_e and the recycling or OSL IR depletion ratio; rejection of aliquots based on recycling or OSL IR depletion ratios does not improve these sample D_e . Figure adapted from [60].

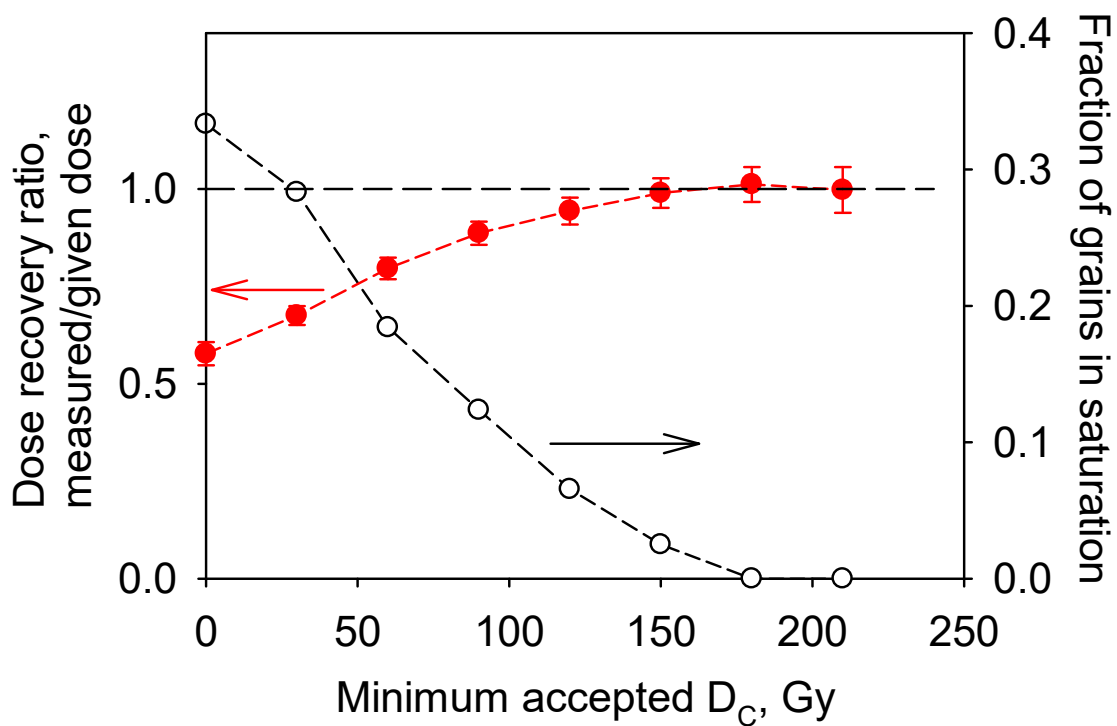


Figure 8. Effect of D_C filtering on grains with different D_C [237]. The dose recovery data (red data, given dose 170 Gy) shows the effect on the dose recovery ratio of only accepting results from aliquots with increasingly larger D_C values. These data suggest that only those grains with $D_C > 125$ Gy give accurate results (red data). Even with this minimum D_C , ~3% of the accepted grains still give doses in measured doses in saturation (black data).

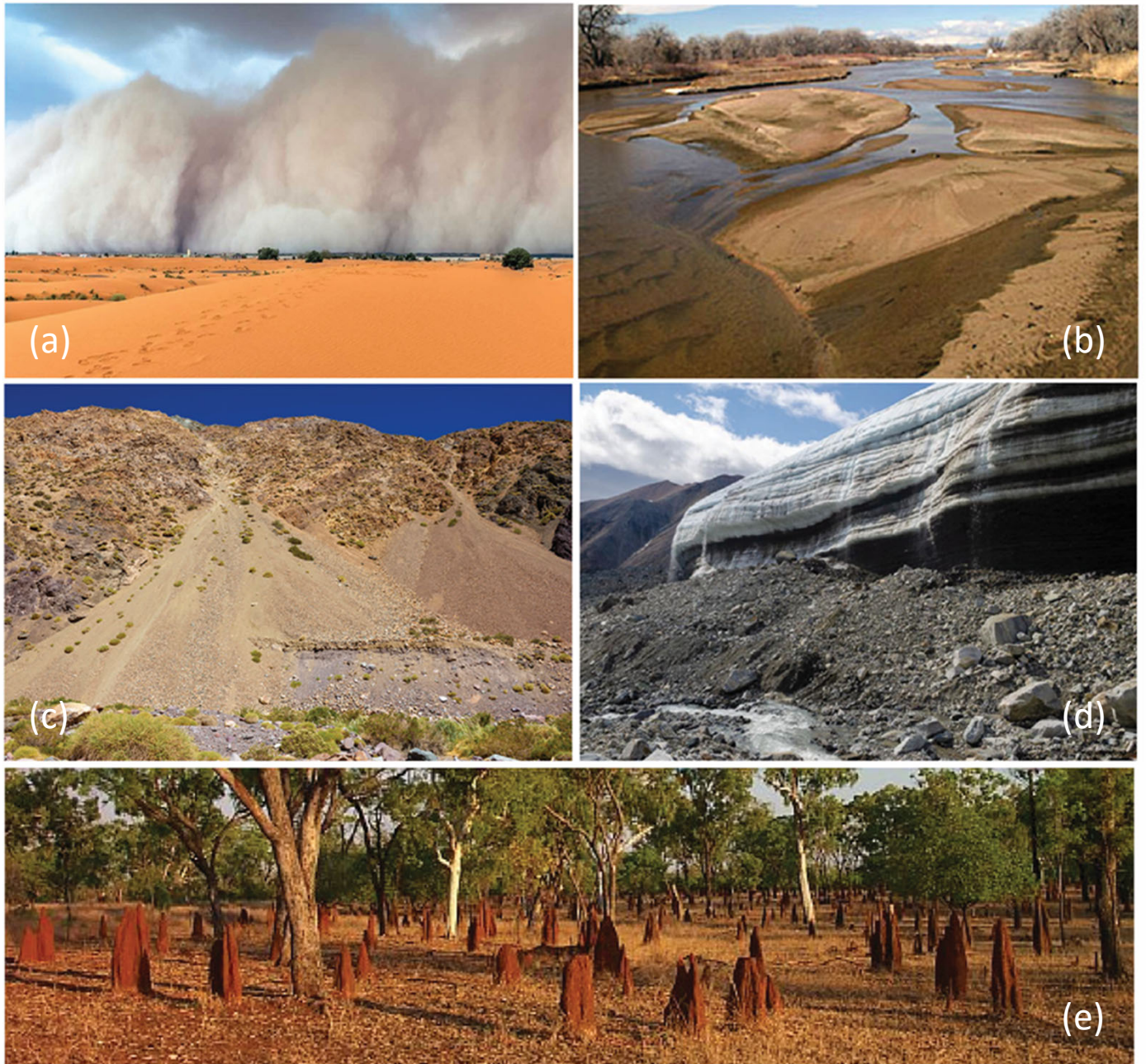


Figure 9. Opportunities for bleaching of the OSL signal in different environments. (a) aeolian transport during dust storms (Pavliha-istockphoto.com). (b) fluvial transport (Marek Uliasz - race.fit2paddle.com). (c) gravity-driven debris fans (unknown) (d) sediment transported by ice (Michael Hambrey - Glaciers online)(e) bioturbation - termite mounds (ourterritory.com).

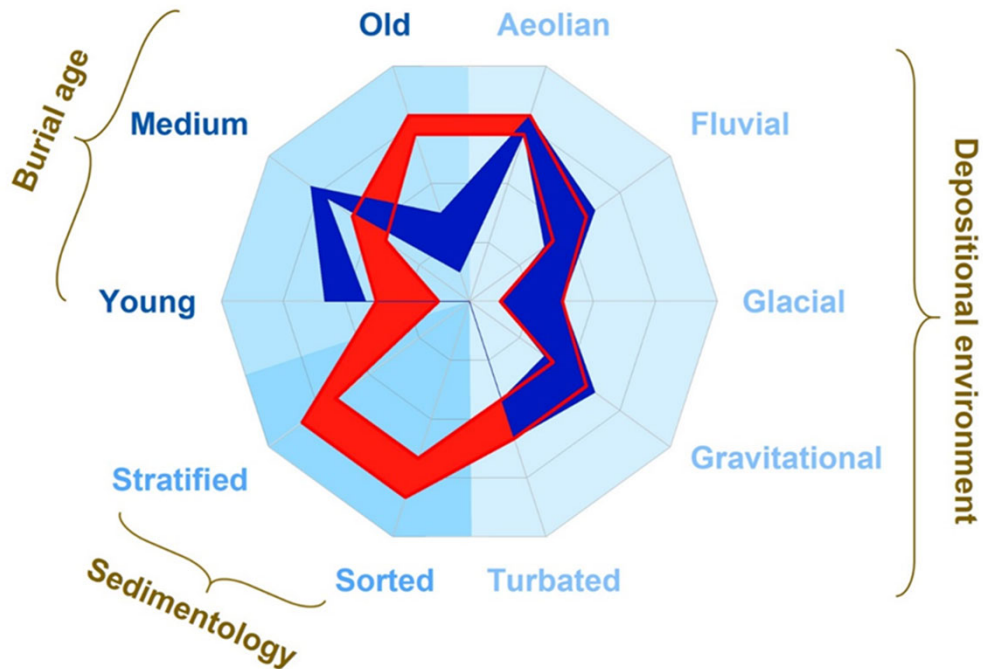


Figure 10a. The likely complexities in D_e determination arising from luminescence characteristics (dark blue band) and bleaching (red band), expressed qualitatively in terms of field observables and expected age (see Section 3 for details). These observables and estimates are shown on the 10 radial axes, grouped according to Depositional environment and implied transport mechanism (very light blue), Sedimentology (light blue) and the expected Burial age of the samples (median blue). “Young” refers to quartz grains with expected D_e values of less than ~ 10 Gy, “Old” to quartz grains with D_e values of larger than ~ 130 Gy. Each axis is scaled from most complex (centre of the figure) to least complex (outer edge of figure). The likely variability of each feature is shown by the band width on a particular axis. There is no reason to expect a correlation between luminescence characteristics and sorting/stratification, and so there is no dark blue band in that portion of the figure.

For example, the figure shows that aeolian sediments are likely to show low complexity, because they tend to be well bleached and have good luminescence characteristics, both with low variability. In contrast, glacial sediments are likely to show high complexity, because they tend to be poorly bleached and have poor luminescence characteristics although the variability in this conclusion is considerable. Bleaching becomes progressively less important (and less variable) as the Burial age increases, and so the complexity arising from this parameter decreases with age, whereas complexity from luminescence characteristics is at a maximum for oldest sediments (mainly because of saturation effects), and a minimum for medium age material, where signals are strong, and the saturation effects are small.

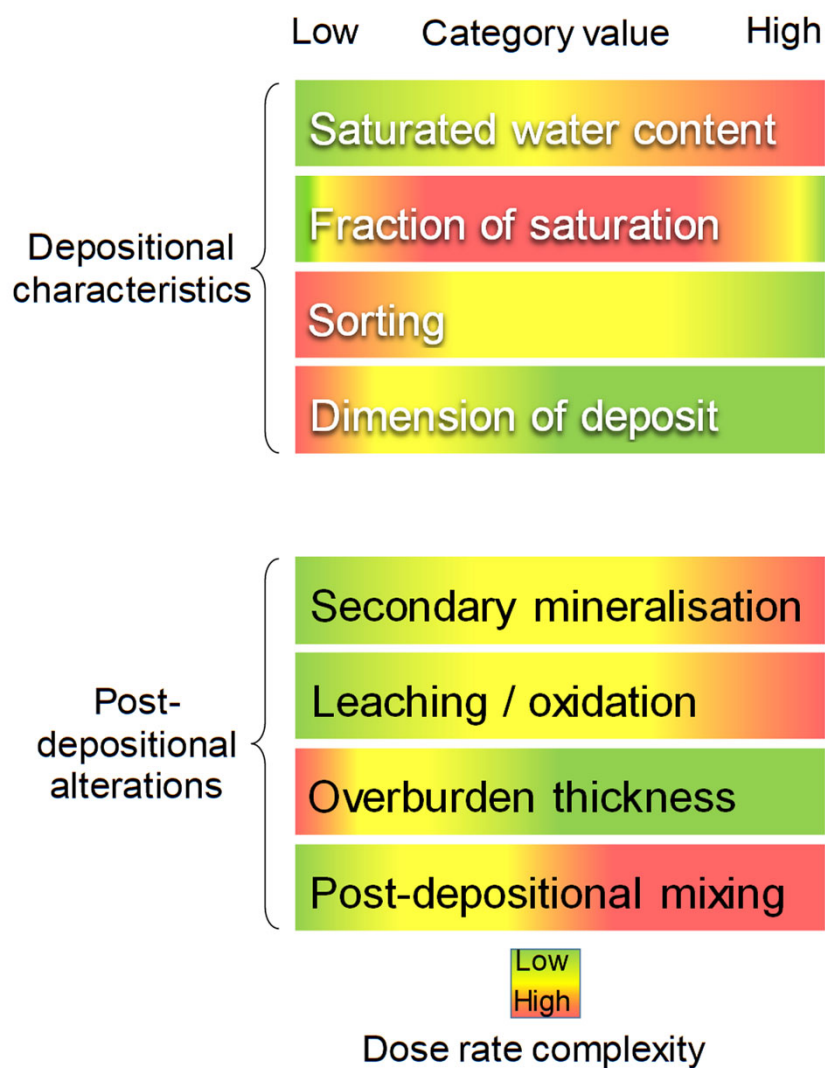


Figure 10b. Bar chart showing the relative influences of different sedimentary characteristics on dose rate complexity. Each bar depicts an observable sedimentary characteristic (y-axis) that can be evaluated at the time of OSL sample collection or during sample preparation (white labels – present at the time of deposition; black labels – post-depositional alteration). Each bar is gradationally coloured according to the degree of beta, gamma and cosmic dose rate complexity (in both space and time) expected for a given category value (x-axis).

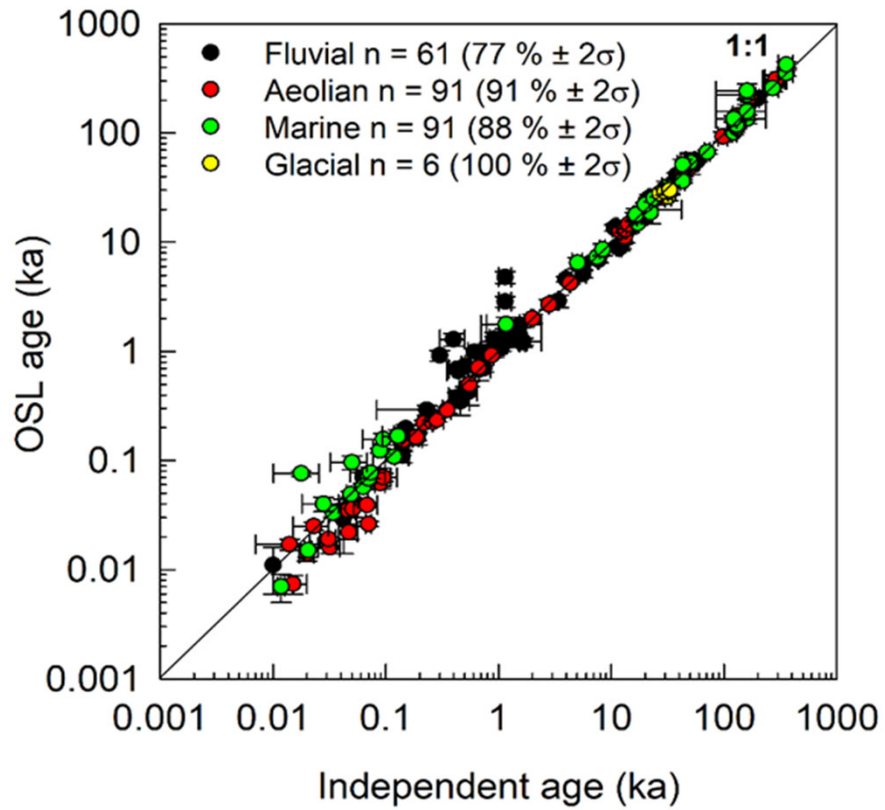


Figure 11. [77], [262], [80]: Comparison of quartz OSL ages with independent age control (n = number of samples, followed by the % of samples within $\pm 2\sigma$ of the line of unit slope).

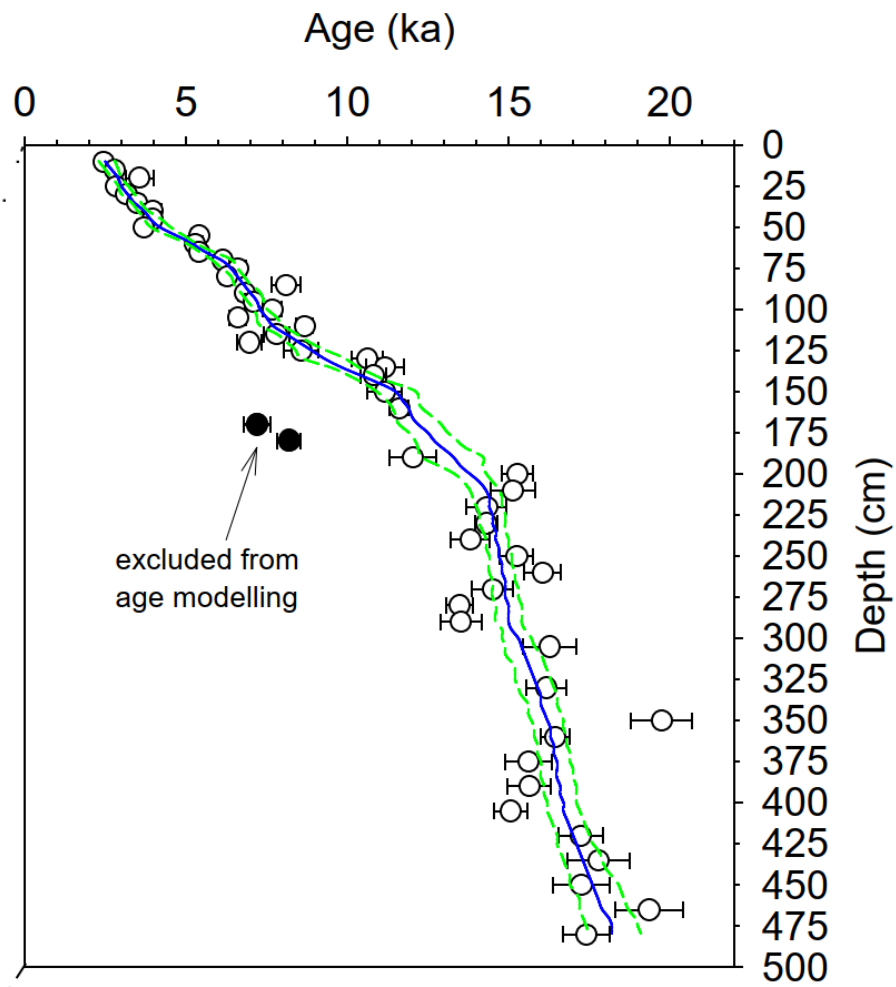


Figure 12. Large aliquot 63-90 μm quartz OSL ages plotted as a function of depth for a section of Chinese loess encompassing last glacial loess and Holocene soil. Uncertainties are random only. A Bayesian model (with 1σ error band) is also shown illustrating how higher precision can be achieved. Ages excluded from modelling are from samples deliberately sampled in crotona. Figure modified from SI in [60].