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A luminescence dating intercomparison based on a Danish beach-ridge sand

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HIGHLIGHTS

- First major OSL dating intercomparison.
- Homogeneous material: RSD dose <3%; dose rate <7%.
- Interlaboratory RSD 10–25% for most parameters.
- Much larger RSD for ^{232}Th and saturated water content.
- RSD in ages ~18%, significantly larger than the 5–10% expected.

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ABSTRACT

There is a need for large scale intercomparisons to determine the degree of coherence of luminescence dating measurements made by different laboratories. Here we describe results from a laboratory intercomparison sample based on a quartz-rich aeolian and/or coastal marine sand ridge from the Skagen peninsula, northern Jutland (Denmark). About 200 kg of sand was sampled at night from a single beach ridge. The sand was homogenised using a cement mixer and packed in ~700 moisture and light-tight bags for distribution. The quartz luminescence characteristics are satisfactory (e.g. fast-component dominated and good dose recovery) and our own equivalent dose determinations and measurements of radionuclide concentrations for twenty of these bags demonstrate the degree of homogenisation. One natural sample and one sample of pre-processed quartz was made available on request; analysis of all the responses gives a mean dose (pre-processed quartz) of 4.58 Gy, $\sigma = 0.40$ ($n = 26$), to be compared to the mean dose (self-extracted quartz) of 4.52 Gy, $\sigma = 0.55$ ($n = 21$). The mean age is 3.99 ± 0.14 ka, $\sigma = 0.71$ ($n = 22$), i.e. a relative standard deviation of 18%. We present an analysis of all the important quantitative and qualitative responses we received between 2007 and 2012 and discuss the implications for our dating community and for users of luminescence ages.

1. Introduction

It has long been recognised by the luminescence dating community that more laboratory intercomparisons are needed to show users (and ourselves) that different laboratories are all able to measure the same equivalent dose, dose rate and age for a sample. Such intercomparisons are well established in other dating methods – radiocarbon dating has recently completed its 5th international intercomparison (Scott et al., 2010) and the cosmogenic dating community has started such laboratory intercomparisons (Blard et al., 2014; Vermeesch et al., 2012). Bassinet et al. (2014) report on an intercomparison of the doses recorded by electronic components, but in luminescence dating, interlaboratory comparisons have been limited in number and scope – the Risø calibration quartz (Hansen et al., these proceedings), used by many laboratories to calibrate their β-sources, provides material with reference behaviour (e.g. optical sensitivity of luminescence signal, reproducibility etc.), and Vandenberghe et al. (2004) presented D_e measurements from three different laboratories (Risø, Ghent, Heidelberg). However, no well-characterised

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natural material has ever been made widely available to the luminescence dating community.

Here we report on the preparation and characterisation of an optically stimulated luminescence (OSL) intercomparison sample based on laboratory-homogenised quartz-rich sand, believed to be well-bleached. The results reported by 30 different laboratories between 2006 and 2012 are then summarised, and the implications for the improvements in our routine measurements discussed.

2. Sampling, preparation and measurement

The bulk 200 kg sand sample was collected 150 cm below ground level from an ~5 ka old aeolian and/or marine beach ridge/swale sequence (57°31′, 10°24′) from the Skagen peninsula, ~2 km southeast of the town of Jerup. Nielsen et al. (2007) argue that it took about 15 years for each ridge to form. Although the natural dose in this material is thus relatively small (and so light levels are not particularly intense), the luminescence characteristics are well known (Nielsen et al., 2007), the site is easily accessible and a homogeneous layer is easily excavated. Topsoil and initial excavation was done during daylight, but final cleaning of light-exposed surfaces was at night. Eight sealed bags each of ~25 kg of damp sand were collected under moonlight, supplemented by low-level red LED light. To ensure that ambient light levels were sufficiently low, three aliquots of calibration quartz mounted on discs were exposed during sampling and the residual doses compared with those from 3 unexposed aliquots. The measured exposed to not-exposed ratio was 0.96 ± 0.02 (n = 3).

In the laboratory, the contents of the eight bags were distributed equally between six containers under subdued red/orange lighting. A cement mixer was then filled by taking ~17% of the sand from each container; this subsample was mixed for ~30 min, before emptying into a new container. This process was repeated, using a new container each time, until all the sand in the original 6 containers had been used up. The whole mixing cycle was then repeated eight times. Finally, 250 g of the homogenised sand was packed in each of ~700 heat-sealed moisture- and light-tight bags and labelled.

To test the homogeneity of the mixed sand, 40 samples were then picked randomly from the ~700 bags, and prepared for analysis at the Nordic Laboratory for Luminescence Dating. The material in 20 of these bags was processed using conventional sample preparation techniques (wet sieving, 1 h 10% HCl, 1 h conc. H_2O_2, heavy liquids [2.62 g cm^{-3} and 2.58 g cm^{-3}], 1 h 40% HF and sieving again) to extract 180–250 μm quartz. The purity of the quartz extracts was examined using an IR-test (infrared stimulated/blue stimulated luminescence ratios <1%), and between 5 and 20 g of clean quartz was recovered from each bag. About 0.5 g of quartz from each sample was put aside to be used for determination of the OSL characteristics. The remaining quartz was combined, mixed again and split into ~700 feldspar-free 400 mg aliquots of 180–250 μm quartz ('Provided Quartz'). The other twenty unprocessed samples were individually dried, ground to <200 μm, ignited at 450 °C for 24 h, and cast in wax to retain radon. They were then stored for >3 weeks before analysis using high-resolution gamma spectrometry (Murray et al., 1987).

In our laboratory, OSL measurements were made using Risø TL/OSL readers (model DA-15) (Bøtter-Jensen et al., 2010) equipped with an array of infra-red (IR, 870 ± 40 nm) and blue (470 ± 30 nm) stimulation LEDs giving stimulation powers of ~80 mW and ~40 mW/cm^2, respectively. Preheat plateau and thermal transfer test results are given by Nielsen et al. (2007); based on these data we employed a SAR protocol (Murray and Wintle, 2000) with a preheat of 200 °C for 10 s, a cut-heat to 180 °C and a test dose of ~4 Gy for equivalent dose estimation on large (8 mm) quartz aliquots. At the end of every SAR cycle the aliquots were optically bleached for 40 s at 280 °C (Murray and Wintle, 2003).

3. OSL characteristics

The net natural test dose signals (first 0.8 s minus background averaged over last 4 s) were ~1600 s^{-1} Gy^{-1} (relative standard deviation, RSD ~60%, n = 48). This result was obtained on the least sensitive of our luminescence readers, and so it was concluded that the sensitivity of this quartz is more than sufficient for an intercomparison standard. The luminescence is dominated by the fast OSL component (Jain et al., 2003; Singarayer and Bailey, 2004) indistinguishable from the fast decay from calibration quartz (inset to Fig. 1a). A representative growth curve illustrating the excellent recycling and low recuperation is given in Fig. 1a. The average recycling ratio is 1.014 ± 0.003 (RSD = 4%; n = 284) and the average recuperation is equivalent to 55 ± 4 mGy (σ = 63 mGy; n = 284), <2% of the equivalent dose (D_e) reported below; histograms of these data are given in Fig. S1. The average dose recovery ratio is 0.976 ± 0.006 (RSD = 6%; n = 96; see Fig. 1b).

4. Homogeneity

In this section, all results (n = 20) have been normalised to their average for discussion of homogeneity, and systematic contributions have been omitted from the calculation of uncertainties associated with each point. For D_e measurements, between 11 and 18 aliquots were measured for each sample; uncertainties on individual estimates of the D_e of each sample vary between ~2 and ~9% and the overall RSD of the twenty sample D_e estimates is 6%. There is no evidence for over-dispersion in the distributions of sample D_e values (Fig. 2a). The distribution of radionuclide concentrations are summarised in Fig. 2b–d, and there is no evidence for over-dispersion in the ^{226}Ra or ^{232}Th concentrations. A small over-dispersion of 5.3 ± 1.1% is present in the ^{40}K distribution. (^{238}U is measured by our gamma spectrometry facility, but the uncertainties are too large to be useful in this discussion). Our measurements of saturated water contents have a relative standard deviation of 9% (n = 20); these saturated values (rather than field values) were used in our age calculations because the saturation water content is less likely to be affected by storage, and therefore more suitable for an intercomparison exercise. In calculating the total annual dose rate to each sample, an internal dose rate to quartz of 0.06 ± 0.03 Gy ka^{-1} was assumed (conservatively based on Mejdahl (1987)), and a cosmic ray dose rate calculated appropriate to a burial depth of 1 m, and the sea level of the site (Prescott and Hutton, 1994). There is no significant over-dispersion in the ages (2.6 ± 1.6%).

In our experience this sample represents a typical mid-Holocene beach sand, and from these measurements we conclude that the quartz OSL characteristics make it a satisfactory material for use in an intercomparison. The results of analysis of the over-dispersion of 20 estimates of D_e and radionuclide concentrations from randomly selected sub-samples indicate that the material is homogeneous; this is also important in an intercomparison sample.

5. Results from participating laboratories

On request, a laboratory received one 250 g pack of untreated sand, one 400 mg pack of 'Provided Quartz' (see above) and a form detailing possible measurements/responses (Table S1). In total, 30 laboratories responded before June 2012 (listed in Table S2). Anonymised detailed responses are given in Table S3 in Supplementary information, and only the most frequent or most relevant measurements are summarised here. Two laboratories (apart from NLL)