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A new irradiated quartz for beta source calibration

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HIGHLIGHTS

- Calibration quartz (180–250 μm and 4–11 μm) for beta source calibration.
- Description of luminescence characteristics: multi-grain and single-grain.
- Dose recovery ratio is indistinguishable from unity with a standard deviation of <2%.
- >80% of individual grains give a useful signal.
- Unexplained dispersion of ~3% in multi-grain data.

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ABSTRACT

For luminescence dating to be an accurate absolute dating technique it is very important that we are able to deliver absolutely known radiation doses in the laboratory. This is normally done using a radiation source (alpha, beta, X-ray) calibrated against an absolutely known reference source. Many laboratories have used the various different batches of Risø calibration quartz for the calibration of beta and X-ray sources, but these have been largely undescribed. Here we describe in detail the preparation and luminescence characteristics of a new quartz standard, based on a North Sea beach sand collected from south-western Denmark (Rømø). Two grain sizes (4–11 μm and 180–250 μm) have been examined in detail. These were pre-treated (annealed, dosed and annealed again) to sensitise and stabilise the luminescence signals before being given an absolutely known gamma dose from a point ^{137}Cs source in scatter-free geometry. The luminescence characteristics are described; the very intense blue-light stimulated signal is dominated by the fast OSL component and the IR-stimulated signal is negligible. The material is shown to be suitable for measurement using SAR, and the dose recovery ratio is indistinguishable from unity with a standard deviation of <2% for multi-grain aliquots. The material is also shown to be suitable for single-grain calibration, with >80% of the grains giving a useful signal. Although there is an unexplained dispersion in our calibration data of ~3% (which we cannot attribute to instrument variability), we nevertheless conclude that this material is very suitable for transferring absolute known doses from a standardised gamma source to in-built irradiation sources.

1. Introduction

Luminescence dating depends on an accurate and precise absolute calibration of laboratory irradiations. Here we describe the preparation and luminescence characteristics of the most recent calibration quartz (CQ) used in our laboratory.

Recently, it has been pointed out that the measured dose in our CQ (batch 57) may depend on SAR parameters (Kadereit and Kreutzer, 2013). Nevertheless, using a standard SAR protocol (225 °C ph for 10 s; 200 °C cutheat; 125 °C stimulation with blue light) Bos et al. (2006) showed that the beta source calibration derived from Batch 8 prepared in 2004 was indistinguishable from that derived from an independently prepared and gamma irradiated quartz sample prepared in their laboratory, and calibrated using Fricke dosimetry.

We have tested the reproducibility of various batches of CQ using a single reader (and without dismounting the beta source) for

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>13 years. The results are summarised in Fig. 1, and the average standard deviation from an exponential decay (Fig. 1, solid line) is 4%. However, it appears that there may be some systematic variation in this decay which is not dependent on batch number. Unfortunately, it is not now possible to undertake a dose recovery on this material, because no undosed sample has been preserved after initial sensitisation. We have provided several thousand samples of our calibration quartz over many years, and many laboratories around the world depend on this material to calibrate their beta irradiations. Because of this uncertainty in performance and because of the questions raised by Kadereit and Kreutzer (2013) it was decided to manufacture a new batch of calibration quartz, and fully describe its characteristics before making it available to the wider community. This is done below.

2. Sampling, sample preparation and measurement facilities

A new bulk sample of quartz sand was collected from Rømø, off the west coast of Jutland (Denmark) in the Wadden Sea. The aeolian sand dunes have been studied extensively by Madsen et al. (2007) who describe the natural luminescence characteristics. Approximately 15 kg of bulk sand was collected in daylight from the face of an aeolian dune. In the laboratory ~2 kg of sand was treated using conventional sample preparation techniques, (10% HCl, 10% H_2O_2, 10% HF and 40% HF) followed by wet sieving. The <40 μm fraction was ground and settled in water to give a 4–11 μm fine-grain fraction.

The chemical purity of the bulk material after chemical treatment was confirmed by measuring 8 mm aliquots using a Risø XRF attachment; the resulting average feldspar to quartz ratio is $-1.3 \pm 0.9\%$ ($n = 3$). Portions of the various size fractions were then sensitised by heating to 700 °C for 1 h, giving a 2 kGy gamma dose using a ^{60}Co source (to stabilise luminescence sensitivity) and heating again to 450 °C for 1 h. A portion of this pre-treated material was kept aside for dose recovery measurements (see Section 3). The remaining fractions were packed in planar 10 × 10 cm glass cells (2 mm wall thickness, 1 mm cavity) wrapped in black plastic and irradiated at a distance of 2 m in a calibrated scatter-free geometry using a point-source of ^{137}Cs (662 keV). The ^{137}Cs source is calibrated using a standardised ionisation chamber measuring air Kerma, by the Danish State Institute for Radiation Hygiene; the dose rate is ~2.9 mGy ks^{-1}, and the resulting absorbed dose in water was 5.00 Gy with a total uncertainty of <3% (1 σ; Miller A., private communication). This gives an absorbed dose in quartz of 4.81 ± 0.14 Gy.

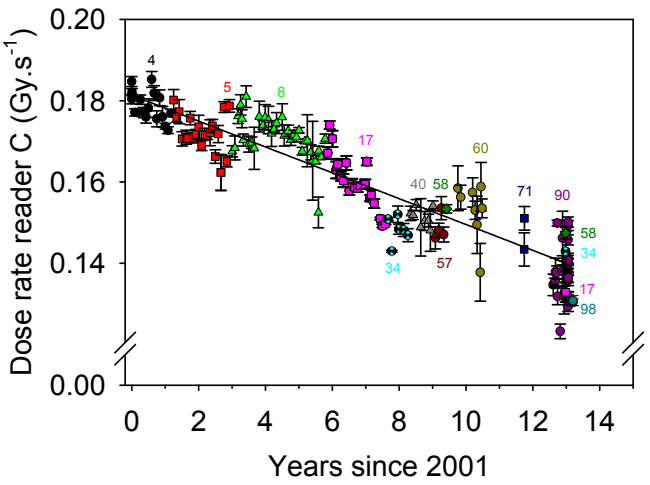

Fig. 1. Dose rate variation with time on reader C. Batch numbers given adjacent to data points.

All measurements described below use a Risø TL/OSL reader (designated as Reader C and upgraded to model DA-20 (Bøtter-Jensen et al., 2010) without disturbing the beta source); the dose rate of the beta source to coarse-grains mounted on stainless steel discs is ~0.14 Gy s^{-1} (see Fig. 1). OSL measurements used blue LEDs (870 ± 40 nm; ~80 mW cm^{-2}) for stimulation, and photon detection was through 7.5 mm of Hoya U-340 glass filter. All measurements used 8 mm diameter multi-grain aliquots; because of the light intensity, a pin-hole attenuator was added to the filter pack to reduce the initial count rate to below 10^6 s^{-1}. Single grain luminescence signals were measured using a 10 mW green laser focussed sequentially on individual grains mounted on an aluminium disc in square 10x10 (600 μm spacing) grid of 300 μm diameter holes. A SAR protocol (Murray and Wintle, 2000) was used for all measurements; unless otherwise specified, this employed a preheat of 260 °C for 10 s and a cut heat of 220 °C. Stimulation for 40 s was at 125 °C for multi-grain aliquots and 1 s for single grains, and a high temperature stimulation (40 s at 280 °C) was used at the end of each SAR cycle to minimise recuperation (Murray and Wintle, 2003).

3. Multi-grain luminescence characteristics

We first describe the luminescence characteristics of 8 mm diameter aliquots of 180–250 μm grains mounted on stainless steel discs using silicone oil, and fine grains deposited on aluminium discs. Fig. 2a shows that the IRSL signal measured at 125 °C is $-0.2 \pm 0.5\%$ ($n = 12$) of the corresponding blue-stimulated signal. More than 99% of the decay of the latter is derived from the fast component (Jain et al., 2003; Singarayer and Bailey, 2003). Despite this, the OSL IR depletion ratio (0.981 ± 0.001; $n = 72$) is distinguishable from unity (Fig. 2b); we attribute this to stimulation of the quartz fast component by IR rather than feldspar contamination. The corresponding pure blue recycling ratio is 1.009 ± 0.001 ($n = 72$).

The dose response of this 180–250 μm CQ allows it to be used for the measurement of dose up to >100 Gy (Fig. 3a, filled black circles). The 180–250 μm sensitivity changes slightly with repeated use (Fig. 3b), but nevertheless the SAR recycling ratio averaged over a preheat plateau from 160 to 300 °C is 1.013 ± 0.002 ($n = 48$) confirming that the test dose is correcting for this residual sensitivity change (data not shown, but collected as part of Fig. 4).

Fig. 4a shows the independence of the dose recovery ratio on preheat temperature, measured both with a fixed cut heat of 160 °C (filled circles), and a variable cut heat 40 °C below the preheat temperature for preheat temperatures above 180 °C (unfilled circles). These dose recovery experiments used sensitised quartz (Section 2) that had not received a 4.81 Gy gamma dose; aliquots of this material were given a beta dose of ~5 Gy and measured in the usual manner. The mean dose recovery ratio over the 160–260 °C temperature interval is 0.993 ± 0.002 ($n = 72$). Fig. 4b shows that the apparent beta source dose rate is also independent of preheat temperature, confirming that for this material the beta source calibration is not dependent on SAR measurement parameters.

The fine-grain luminescence characteristics are broadly similar to those of the coarse grains (from which they are derived) although the dose response curve grows to higher doses than the coarse grains (filled red triangles in Fig. 3a). Fig. 5 summarises dose recovery ratios for fine-grains mounted on aluminium discs; the mean dose recovery ratio using a fixed 260 °C preheat is 1.029 ± 0.005 ($n = 24$).

Table 1 summarises the calibration data for reader C; this table includes the dose rate for 180–250 μm grains mounted in stainless-steel cups, and the dose-rate ratios normalised to 180–250 μm