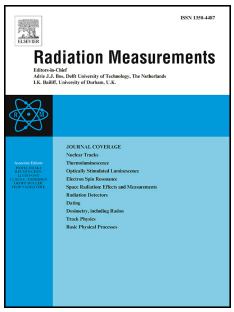
Accepted Manuscript



Strategies for equivalent dose determination without heating, suitable for portable luminescence readers

H.M. Roberts, G.A.T. Duller, M. Gunn, C.R. Cousins, R.E. Cross, D. Langstaff

PII: S1350-4487(17)30842-9

DOI: 10.1016/j.radmeas.2018.04.018

Reference: RM 5907

To appear in: *Radiation Measurements*

Received Date: 7 December 2017

Revised Date: 16 April 2018

Accepted Date: 26 April 2018

Please cite this article as: Roberts, H.M., Duller, G.A.T., Gunn, M., Cousins, C.R., Cross, R.E., Langstaff, D., Strategies for equivalent dose determination without heating, suitable for portable luminescence readers, *Radiation Measurements* (2018), doi: 10.1016/j.radmeas.2018.04.018.

Strategies for equivalent dose determination without heating, suitable for portable luminescence readers

H.M. Roberts[1], G.A.T. Duller[1], M. Gunn[2], C.R. Cousins[3], R.E. Cross[2], & D. Langstaff[2]

[1] Department of Geography and Earth Sciences, Aberystwyth University, Wales, SY23 3DB, UK
[2] Department of Physics, Aberystwyth University, Wales, SY23 3BZ, UK
[3] Department of Earth and Environmental Sciences, University of St Andrews, Scotland, KY16 9AL, UK

Abstract

In recent years a number of portable instruments have been built for measuring the optically stimulated luminescence (OSL) signal from naturally occurring minerals. Some of these instruments have incorporated ionising radiation sources, giving the possibility of determining an equivalent dose (D_e), but little use has been made of these. One challenge has been that heating samples in this type of equipment is a major engineering challenge, yet methods for D_e determination use thermal pretreatments to remove charge from unstable traps, making signals arising from irradiation in nature and the laboratory comparable. This paper explores three strategies for obtaining accurate estimates of the D_e of samples in situations where thermal treatments are not possible: (1) deriving a correction factor based on comparing D_e values obtained using protocols with and without heating; (2) removing the contribution from the 110°C TL peak and other unstable defects by component fitting the unheated OSL signal; and (3) adding a small beta dose to the sample prior to measurement of the natural luminescence signal so that the 110°C TL peak is filled, making this measurement comparable with regeneration measurements where this peak is also populated. All three methods are promising when applied to quartz that has been physically separated from samples using standard laboratory procedures. The next step in

this work will be to explore whether such methods can be applied to mixed mineral assemblages as would be encountered in the field.

Keywords

Quartz, preheating, dating, D_e determination, 110C TL peak

Highlights

- Portable luminescence readers increasingly used but heating samples is problematic
- 3 strategies tested in the laboratory to obtain D_e values for dating without heating
- Consistent relation between unheated and heated D_e values gives D_e correction factor
- OSL curve deconvolution gives a stable OSL signal from unheated quartz
- Adding small beta dose to fill 110 ˚C trap before measuring unheated L_n gives true D_e

1. Introduction

In recent years, there has been increasing interest in the development and use of portable luminescence systems to assess unprepared sediment samples, particularly for deployment at (or at least near) field sites (e.g. Poolton et al., 1994; Takeuchi et al., 2008; Sanderson and Murphy, 2010; Kook et al., 2011). The design of these systems has varied. Some systems simply measure optically stimulated luminescence (OSL) or pulsed OSL signals; other systems incorporate an X-ray source to allow calibration of luminescence signals and potentially obtain an estimate of equivalent dose. A critical consideration for any such portable luminescence system is whether or not they should incorporate a facility for heating sediment samples; some of the portable systems do, and others do not. Thermal pre-treatments and the use of heat during the stimulation of luminescence signals forms a

critical part of most measurement protocols in the laboratory, helping to remove thermally-unstable charge after irradiation, and enhancing initial OSL signal (Spooner, 1994). The ability to heat samples within a portable or field instrument could potentially be similarly advantageous in some situations, however the lack of ability to heat need not necessarily be critical. For example, Ankjægaard et al. (2006) demonstrated that D_e values of within 30% of the known value could be obtained for quartz without heating, by either i) delaying measurements of laboratory-given radiation doses by > 10 ks to allow unstable shallow traps to empty, or by ii) curve deconvolution to isolate a stable OSL signal. In practice, heating within portable systems presents complex challenges which must be overcome. These challenges include issues ranging from the physical presentation of samples to/within the instrument, and extend through to meeting the power demands of an instrument within a field-based setting. Challenges are also posed by heating of 'wet' untreated field sediments, and include the potential for condensation within the instrument itself which could impede measurements and cause damage within the instrument, aside from posing issues for the reproducibility of heating of the sediments.

This paper explores different approaches to obtaining chronologically-valuable information in situations where irradiation is possible but without the use of any heating during the measurement procedures employed. For simplicity, the study is conducted using coarse-grained quartz, as this is a mineral for which much is known about the basic luminescence characteristics, such as the source of the OSL signal, the trap-depth ('E') and frequency factor ('s') values for a number of defects giving rise to TL peaks, and the mechanism of luminescence production (Preusser et al. 2009). Measurements reported here are conducted on a laboratory-based instrument, but the findings are applicable to portable field instruments capable of irradiation.

2. Instrumentation and Methods

All measurements were made using a Risø TL/OSL-DA-20 and a Risø TL/OSL-DA-12 laboratory instrument, equipped with Sr/Y beta irradiation sources, delivering between 0.097 and 0.014 Gy/s respectively. Optical stimulation was achieved using blue (470 Δ 20 nm) light emitting diodes (LED), and detection was through 7.5 mm thickness of Hoya U-340 glass filter. In this paper, the term 'heated aliquots' is used to describe aliquots preheated to 220˚C at a rate of 5˚C/s and held for 10s prior to the measurement of the Natural signal (L_n) or the signal from a regenerative dose (L_x), and a preheat of 160˚C/10s prior to measurement of the test dose (T_x), with all OSL stimulations conducted at a temperature of 125˚C for 40s. Where 'unheated aliquots' are described, aliquots have not received any thermal pretreatment, and measurement of the OSL signals is conducted for 40s at room temperature. All measurement sequences used in this study are 'run one at a time', meaning that the timing between different steps in any experiment will be the same for each aliquot. The materials used for measurement are coarse-grained quartz (spanning a narrow 20-30 µm range within a broad sand-sized fraction of 125-250 µm diameter grains), extracted from a range of samples. Samples were prepared using hydrochloric acid (10% v.v. HCl) to remove carbonates, followed by hydrogen peroxide (20 vols. H_2O_2) to remove organics, then density separated using sodium polytungstate to remove heavy minerals (>2.70 g/cm^3) and isolate a quartz-rich fraction (2.62-2.70 g/cm^3), which was then etched for 40 mins using 40% hydrofluoric acid (HF) followed by 40 mins of 37% HCl to remove insoluble fluorides, and finally re-sieved to remove any remaining small feldspar grains.

3. Results

3.1 Comparing heated and unheated quartz OSL signals and D_e values

The use of thermal pre-treatments and heat treatments during optical stimulation are standard practise within OSL measurements protocols (Wintle and Murray 2006). They are typically used to remove thermally-unstable charge following laboratory irradiation, and they can also enhance the initial OSL signal being measured (Spooner, 1994). The impact of thermal treatments is illustrated in Figure 1a for two aliquots of coarse-grained quartz isolated from Aber72/WD50-5, a dune sand ~6 ka (Bristow et al. 2007). Raw decay curves for Natural (L_n) and laboratory regenerated doses (L_x), measured using protocols with and without heating (as outlined in section 2.) are shown; each raw decay curve (no background subtraction) has been normalised to the first datapoint to facilitate comparison. The decay curves for the heated Natural (L_n) and heated regenerative dose signal (L_x) (measured at a temperature of 125˚C following preheating) are very similar in shape, as might be anticipated given that the role of the thermal treatments is to remove unstable charge from the laboratory irradiations and hence mimic in the laboratory the effect of the passage of time upon the signal in nature and create a compatible laboratory signal. In contrast, the unheated Natural signal (L_n, measured at room temperature, and with no thermal pre-treatment) gives a slower decay curve than the heated L_n and L_x decay curves. The unheated regenerative dose (L_x) signal shows a marked difference to all of the other heated and unheated decay curves shown in Fig. 1a, being much slower to decay and with a much larger, slowly decaying background. These observations are similar to those noted by Ankjægaard et al. (2006) as part of their examination of whether or not thermal pre-treatments are necessary for quartz.

The dose response curves derived from these measurement protocols with and without heat treatments, are similar at low doses but begin to diverge at doses greater than ~10 Gy (Fig. 1b; see caption for signal summation intervals), with the unheated L_x/T_x ratios being lower than the heated ratios in each case beyond ~10 Gy; the Natural L_n and T_n values also differ for heated and unheated quartz. These effects combine to give different mean equivalent dose (D_e) values for heated (10.8 ± 0.8 Gy; n=3) versus unheated (7.6 ± 1.4 Gy; n=3) quartz, with the unheated D_e value being 30% lower than the heated D_e value. This value for a sample that is ~ 6 ka (Bristow et al. 2007) is consistent with the 30-50% range for D_e underestimations that Ankjægaard et al. (2006) observed for three unheated samples, all of which were ~ 25-32 ka.

Ankjærgaard et al. (2006) used the D_e value obtained from conventional SAR measurements containing thermal pre-treatments to provide the "known value of D_e" against which to compare D_e values derived from approaches without any heating, to see if these were equivalent to the known (heated) D_e values. An alternative approach is to explore whether there is a consistent relationship between the D_e values obtained with and without thermal treatments, and to use this as a basis for correcting the D_e values obtained without heating. Heated and unheated D_e values were obtained for 32 samples of prepared sedimentary quartz from deposits that were believed to be well-bleached, with a range of (heated) D_e values between ~0 – 120 Gy, taken from ten localities around the world (Fig. 2). As already observed in Fig. 1b, the unheated quartz D_e values typically underestimate the D_e values from heated quartz, across the range of D_e values and samples. The relationship between unheated and heated D_e values across all samples and sites can be fitted with a second order polynomial ($y = 0.0063x^2 + 0.3832x + 0.6861$; $r^2 = 0.93$; Fig. 2). Hence, a scaling factor derived from the relationship between the unheated/heated D_e values of a suite of samples

could be used to correct unheated D_e values to give the approximate equivalent of heated D_e values.

3.2 The role of the 110˚C TL peak in unheated OSL signals

In conventional quartz OSL measurement protocols, thermal treatments are applied both prior to the measurement of quartz OSL signals to remove any unstable charge (termed 'preheating'), and also during optical stimulation to keep the unstable 110 ˚C TL trap empty. For unheated quartz, it is highly likely that the 110˚C TL trap plays a role in the OSL signal, and may account for at least some of the difference in decay curve shape between the Natural and regenerative dose decay curves for unheated quartz discussed previously for Figure 1a. The charge in the 110 ˚C trap is unstable and decays over a few hours, hence the 110 ˚C trap is empty in samples that have been naturally irradiated. However, the 110 ˚C trap will fill after the sample is irradiated in the laboratory, giving a visible 110 ˚C TL peak if the material were to be heated. Delaying measurement of the unheated OSL signal until >10 ks after irradiation was found by Ankjægaard et al. (2006) to yield a D_e value that agreed with that of heated quartz, although it was highlighted that delays of such duration were impracticable in the field. Although the focus of this paper is primarily upon what can be achieved with no heating, it is instructive to examine how moderate heating of the sample, to temperatures well below those normally used for preheating, could reduce the time needed to remove the 110°C TL peak. Isothermal storage experiments were undertaken on 180-210µm diameter quartz from 105/KB-15 (Kalambo Falls, see Duller et al. 2015). A pre-sensitised aliquot was irradiated with a 1.4 Gy beta dose, and then stored at room temperature (24°C), 40, 60 or 80°C. After storage for different periods of time, the aliquot

had its TL measured so that the 110°C TL peak could be assessed. The remaining signal is plotted in Fig 3. As expected the signal decays exponentially with storage time, and the rate is strongly dependent upon storage temperature. At room temperature (~24 °C), storage for 10 ks as recommended by Ankjærgaard et al. (2006) leaves ~3% of the original signal (Fig. 3). The same level of depletion can be achieved at 40°C after ~3100 s, at 60°C after less than 500 s, and less than 100 s at 80°C (Fig. 3). Thus if a portable system was capable of heating a sample to even moderate temperatures such as these then storage to remove the 110°C TL peak becomes feasible.

However, assuming that no heating is available, it is possible to look at the changing influence of the 110 ˚C trap over time by examining the change in the unheated OSL signal derived from laboratory regenerative doses measured after different periods of time delay following irradiation (Fig. 4). The same single aliquot of sample 105/KB-15 was used for this experiment as for the previous one, which included gentle heating. The aliquot had been pre-sensitised by heating and irradiating and reading out the OSL signal several times before use in this experiment, to ensure there was no sensitivity change between measurement cycles during the experiment itself. This pre-sensitised aliquot was irradiated with a 1.4 Gy beta dose in the reader, paused (ensuring the sample was not under the irradiator) for different durations from 150 s to up to 169,050 s before measuring the OSL signal at room temperature, then heated to 450 ˚C to remove any remaining charge, before giving the next irradiation dose and pausing for a different period of time before measuring the OSL signal at room temperature, etc. In Fig. 4, the OSL curves derived from the shortest delays are the uppermost curves, and steadily drop with increased time delay such that the longest delays (up to a maximum of 169 ks) are the curves shown towards the lower part of the plot.

The most obvious change to the OSL signal as the period between irradiation and OSL measurement decreases, is the increase in a slowly decaying component that is particularly obvious after ~5 s optical stimulation (Fig. 4). This is similar to previous observations made by others (e.g. Wintle and Murray 1997). What is less obvious from Fig. 4, however, is that the intensity of the initial OSL signal also decreases as the delay between irradiation and OSL measurement increases (Fig. 4). The change in the raw OSL signal (i.e. no background subtraction) with increasing delay between OSL measurement and irradiation time, is also shown in Fig. 5, normalising the data to the signal acquired following the shortest possible delay time between irradiation and OSL measurement (150 s). This shows a steady decline in the first channel of the OSL signal measured with increasing time delay between irradiation and OSL measurement (Fig. 5). Over the 169 ks of this experiment the signal drops by 38%, demonstrating that a large proportion of this unheated OSL signal is not stable over time, hence making it unsuitable for dating.

The OSL signals observed in Fig. 4 were measured without any preheat to remove charge from traps with low stability, and thus could arise from charge derived from a variety of defects, with different lifetimes. Several different background subtractions were therefore examined, to try to isolate a part of the unheated OSL signal that is stable over laboratory timescales, and hence might be suitable for dating. Employing a late background subtraction (i.e. the first channel minus the last 5 s of a 100s stimulation) gives a net signal that behaves similarly to the raw initial signal data from the first channel (Fig. 5). Using an early background subtraction (i.e. the first channel minus the signal at less than 2 s of the stimulation time) gives a signal that drops more slowly, but after 169 ks has dropped by 33%, not dissimilar to the raw OSL signal (38%). A slightly later-early background (i.e. the first channel minus the signal at 7.5s into the 100 s stimulation) gives a signal that has only

decreased by 22% (Fig. 5), but all of these signals decay over time, indicating that these net OSL signals for unheated quartz still contain contributions that are not stable on laboratory timescales. It seems, therefore, that a simple background subtraction is not sufficient to isolate a stable unheated OSL signal from quartz that is suitable for dating. Instead, perhaps a stable unheated quartz OSL signal can be isolated using curve deconvolution, as suggested by Ankjægaard et al. (2006)?

3.3 Curve deconvolution to isolate a stable unheated quartz OSL signal?

An attempt was made to identify a stable unheated OSL signal using deconvolution of the unheated quartz OSL decay curves (c.f. Ankjægaard et al., 2006) from sample 105KB-15 measured after different time delays since irradiation (shown in Fig. 4). These data were fitted in SigmaPlot 12™ using three exponentially-decaying components (Eq 1), giving values of the detrapping rate (b_x, s^{-1}) and the trapped charge population (n_x) for each component, for each of the curves shown in Fig. 4. To reduce scatter arising from small differences in the b values fitted for each OSL decay curve, a global fit was used. This involved having the same value for parameter b_1 and for b_2 for all the OSL decay curves, fitting all the data simultaneously. Fitted values were 2.661 s^{-1} (± 0.016, standard error) for b_1 and 0.630 s^{-1} (± 0.002, standard error) for b_2. Values of b_3 were allowed to vary between the different OSL decay curves, and gave a trend from 0.021 s^{-1} for the short pauses to 0.074 s^{-1} for the longer pauses. All the OSL decay curves were well fitted to this equation, and there was no sign of structure in the fitting residuals.

$$L(t) = a + (n_1 b_1 e^{-b_1 t}) + (n_2 b_2 e^{-b_2 t}) + (n_3 b_3 e^{-b_3 t}) \qquad \text{Eq. 1}$$

The change in the normalised trapped charge population (n_x) of each of the three components fitted, is shown in Fig. 6 plotted against increasing time delay between irradiation and measurement of the OSL signal. The trapped charge population of the first component (n_1, the fastest) decreases with increasing delay time between irradiation and measurement of the unheated OSL signal (Fig. 5), giving a pattern reminiscent of the data from the raw OSL signal using a simple background correction (Fig. 5), and suggesting that this first component is also not stable over time. However, the trapped charge population (n_2) of the next fastest component, component 2, remains consistent over the 169 ks of the experiment (Fig. 6), showing that this signal is stable over laboratory timescales. The trapped charge population of the third component (n_3) suggests that there is an unstable element to that component of the unheated quartz OSL signal.

For conventional heated OSL data collected at a stimulation temperature of 125 ˚C, the first component isolated in curve deconvolution is typically the 'Fast' component (Bailey et al. 1997), i.e. the signal associated with the 325 ˚C TL peak. However, for the unheated quartz OSL signals discussed here, the calculated detrapping rates (b values) imply that the b value for the second (i.e. the stable) unheated OSL component is consistent with those values for the heated fast-component from quartz measured at 125 ˚C. In contrast, the first component in the unheated OSL signal has a much higher detrapping rate.

For unheated quartz, it is the second component (trapped charge population, n_2) that gives a stable signal over laboratory timescales, and which therefore seems the most likely of all the unheated OSL signals examined thus far to give D_e values for unheated quartz that will match the heated D_e values. The validity of this approach was tested using a different sample (72WD50-5), to see if the heated quartz D_e value of 10.8 Gy for that sample could be

matched using the second component derived from unheated quartz OSL signals to generate component-fitted SAR dose-response curves and D_e values. Using a standard integrated (i.e. non-component resolved) unheated OSL signal gave a D_e value of 7.6 Gy, and a ratio for unheated to heated quartz D_e values of 0.7, as previously seen from the earlier comparisons in Fig. 2, discussed in section 3.1. Calculating D_e based on component 1 of the curve deconvolution using three-components, i.e. an unstable component over time but the fastest component to be fitted, gives an unheated D_e value of 5.5 Gy, which is approximately half of the heated D_e value, and even lower than using the previous, simpler approach of integrating the whole OSL signal. However, if component 2 of the three-component fit is used to derive a D_e value from unheated quartz, the D_e of 12.2 Gy is 13% larger than the heated D_e value for this sample, 72WD50-5. Using curve deconvolution to isolate a stable component from unheated quartz OSL decay curves therefore looks promising (albeit computationally rather cumbersome on a regular basis, unless semi-automated) as a means of isolating a potentially stable signal which gives data commensurate with that derived from heated OSL signals from quartz.

3.4 Compensating for an empty 110 ˚C trap in the Natural signal

The methods considered thus far have focused upon attempting to make the distribution of charge when making measurements of L_x and T_x, match those when making measurements of the Natural signal (L_n), i.e. by removing the influence of the 110 ˚C trap in the laboratory-generated signals such that they mimic the Natural signal, where the 110 ˚C trap has emptied naturally over time. This has been achieved by, 1) emptying the 110 ˚C trap by preheating and keeping it empty by stimulating at a temperature greater than 110 ˚C, which

could be difficult to achieve in a field-portable instrument for the reasons outlined in the introduction, and 2) by component fitting to extract a signal equivalent to the stable 'Fast' OSL component, which is rather complicated.

An alternative, and potentially simpler approach, may be to try to make the charge distribution of the Natural OSL signal (L_n) mimic that of the signal from a laboratory dose (L_x). This may potentially be achieved by adding a known small radiation dose prior to measuring the Natural signal to try to populate the 110 ˚C trap. To that end, fresh quartz aliquots of sample 72WD50-5 were prepared and given a beta dose that varied from 0-20 Gy; the value of D_e plus the added dose (Gy) was determined using an unheated or heated SAR protocol, as appropriate, and the D_e could then be calculated by subtracting the value of the added dose in Gy (Fig. 7). This experiment suggests that a dose of 10 Gy added to the sample prior to any measurement is sufficient to yield a D_e value from unheated quartz using a basic integrated OSL signal with late-background subtraction that is equivalent to the D_e calculated from heated quartz (Fig. 5). This approach offers a relatively straightforward and simple approach to the potential D_e underestimate that would otherwise be achieved using the unheated OSL signal from quartz.

4. Summary and conclusions

The key question explored in this paper is whether it is possible to obtain chronologically-valuable information in situations where irradiation is possible, but without the use of heating (e.g. for use with portable luminescence systems). Three strategies were evaluated using the OSL signal derived from separated quartz. Firstly, a relationship was observed

between unheated and heated D_e values derived from a variety of quartz samples measured in the laboratory. The second order polynomial fitted to this data could be used to correct unheated D_e values assessed in the field or laboratory, and derive D_e values that can be used for dating. A wide geographical range of samples were analysed in order to give some sense of how consistent this relationship may be across different samples and different sources for the quartz. Secondly, curve deconvolution to isolate the Fast (i.e. the signal associated with the 325 ˚C TL peak, not necessarily the fastest) component (c.f. Ankjægaard et al., 2006) showed that for the sample tested in the present study the second component (n_2) for unheated quartz OSL was stable, and gave D_e values using the unheated data commensurate with the target D_e values from heated quartz OSL. Automated curve deconvolution would need to be implemented if this solution were used in a field instrument, possibly utilising genetic algorithms that have proved robust in solving these complex problems (Adamiec et al. 2006). Finally, adding a small beta dose (~10 Gy) to the Natural signal before measuring the D_e using a SAR protocol without heating, proved to be a relatively straightforward approach which gave unheated quartz D_e values that were comparable to those from heated quartz, for the sample tested. While this would be expected to work well for samples with low De values, the approach would be expected to become less effective for older samples where problems of saturation will become increasingly significant.

Each of these three approaches gave D_e values comparable to those of heated quartz without actually heating, which could be extremely helpful for use in field instruments, and is promising enough to warrant further study across larger datasets in future. The next obvious step, if these methods are to be developed for a field-portable reader, is to combine these approaches with pulsed OSL (Thomsen et al., 2008) or time-resolved

luminescence methods (Ankjaergaard et al. 2010) for isolating an OSL signal from quartz that is mixed with other minerals, as would be the case in the natural environment, and to evaluate whether these methods are still effective in this more complex situation.

Acknowledgements

This work was supported by the UK Space Agency CREST3 program under grant ST/P001998/1. Research in Next Generation Luminescence methods in Aberystwyth is supported by NERC grant CC003, and by HEFCW infrastructure funding for SPARCL. Hollie Wynne is thanked for sample preparation and data collection. We thank the reviewers for their helpful comments on this work.

References

Adamiec, G., Bluszcz, A., Bailey, R., Garcia-Talavera, M., 2006. Finding model parameters: Genetic algorithms and the numerical modelling of quartz luminescence. Radiat. Meas. 41, 897-902.

Ankjærgaard, C., Jain, M., Thomsen, K.J., Murray, A.S., 2010. Optimising the separation of quartz and feldspar optically stimulated luminescence using pulsed excitation. Radiat. Meas. 45, 778-785.

Ankjærgaard, C., Murray, A.S., Denby, P.M., 2006. Thermal pre-treatment in the OSL dating of quartz: is it necessary? Radiat. Prot. Dosim. 119, 470-473.

Bailey, R.M., Smith, B.W., Rhodes, E.J., 1997. Partial bleaching and the decay form characteristics of quartz OSL. Radiat. Meas. 27, 123-136.

Bristow, C.S., Duller, G.A.T., Lancaster, N., 2007. Age and dynamics of linear dunes in the Namib Desert. Geology 35, 555-558.

Duller, G.A.T., Tooth, S., Barham, L., Tsukamoto, S., 2015. New archaeological investigations at Kalambo Falls, Zambia: Luminescence chronology and site formation. J. Hum. Evol. 85, 111-125.

Kook, M.H., Murray, A.S., Lapp, T., Denby, P.H., Ankærgaard, C., Thomsen, K., Jain, M., Choi, J.H., Kim, G.H., 2011. A portable luminescence dating instrument. Nucl. Instrum. Methods B 269, 1370-1378.

Poolton, N.R.J., Bøtter-Jensen, L., Wintle, A.G., Jakobsen, J., Jørgensen, F., Knudsen, K.L., 1994. A portable system for the measurement of sediment OSL in the field. Radiat. Meas. 23, 529-532.

Preusser, F., Chithambo, M.L., Gotte, T., Martini, M., Ramseyer, K., Sendezera, E.J., Susino, G.J., Wintle, A.G., 2009. Quartz as a natural luminescence dosimeter. Earth Sci. Rev. 97, 184-214.

Sanderson, D.C.W., Murphy, S., 2010. Using simple portable OSL measurements and laboratory characterization to help understand complex and heterogeneous sediment sequences for luminescence dating. Quat. Geochron. 5, 299-305.

Spooner, N.A., 1994. On the optical dating signal from quartz. Radiat. Meas. 23, 593-600.

Takeuchi, T., Shibutani, T., Hashimoto, T., 2008. Construction of a portable mini luminescence-measurement system equipped with a miniature X-ray generator. Geochronometria 30, 17-22.

Thomsen, K.J., Jain, M., Murray, A.S., Denby, P.M., Roy, N., BZtter-Jensen, L. 2008. Minimizing feldspar OSL contamination in quartz UV-OSL using pulsed blue stimulation. Radiat. Meas. 43, 752-757.

Wintle, A.G., Murray, A.S., 1997. The relationship between quartz thermoluminescence, photo-transferred thermoluminescence and optically stimulated luminescence. Radiat. Meas. 27, 611-624.

Wintle, A.G., Murray, A.S., 2006. A review of quartz optically stimulated luminescence characteristics and their relevance in single-aliquot regeneration dating protocols. Radiat. Meas. 41, 369-391.

Figure Captions

Figure 1: a) Normalised optically stimulated luminescence decay curves for Natural (L_n) and regenerative dose (L_x) signals (initial 10 s of 40 s stimulation only shown, for clarity), and b) dose response curves acquired using measurement protocols with (red symbols) and without heating (blue symbols), applied to coarse grained quartz prepared from sample 72WD50-5, calculated using a signal from the first 0.2 s (2 channels) minus a background from the final 5s (50 channels) of the 40s stimulation; L_n/T_n values are denoted by triangle symbols, and L_x/T_x regenerative dose points by circle symbols.

Figure 2: Unheated D_e values plotted against heated D_e values (Gy), for 32 samples taken from units believed to be well-bleached, across ten sites worldwide; an inset figure is shown for clarity at low D_e values. Each data point is the mean of three D_e determinations. The data were fitted with a second order polynomial (shown by the solid black line), and the 1:1 line is also indicated (grey dashed line). The sites are Dungeness, UK (73BH), South Africa (FL19), Namibia (72- and 96-), Gwithian, UK (184- and 161), USA (59CY), New Zealand (TNE), Ghana (220RMU).

Figure 3: The signal observed in the 110°C TL after storage at different temperatures, and for different periods of time. The signal is expressed as a percentage of the signal that is observed when a prompt measurement (150 s after irradiation) is made.

Figure 4: Raw OSL signal decay as a function of OSL stimulation time, normalised to the first datapoint for each curve showing different time delays between laboratory irradiation and OSL measurement. Inset shows just the first 5 seconds of raw OSL signal for clarity.

Figure 5: OSL signal remaining as a function of the time between irradiation and measurement of the OSL signal from sample 105KB-15, plotted using different signal integration limits normalised to the first datapoint.

Figure 6: Results of component fitting of the OSL decay curves shown in Fig. 4. The concentration of charge in each of the three components fitted to the data are shown as a function of the storage time between irradiation and OSL measurement.

Figure 7: Equivalent dose for sample 72 WD50-5 obtained using protocols with heating, and without. Aliquots were given various added doses ranging from zero to ~20 Gy prior to equivalent dose determination. The mean of the D_e values for the measurements using heating is shown as a guide for the target value.

Figure 1:

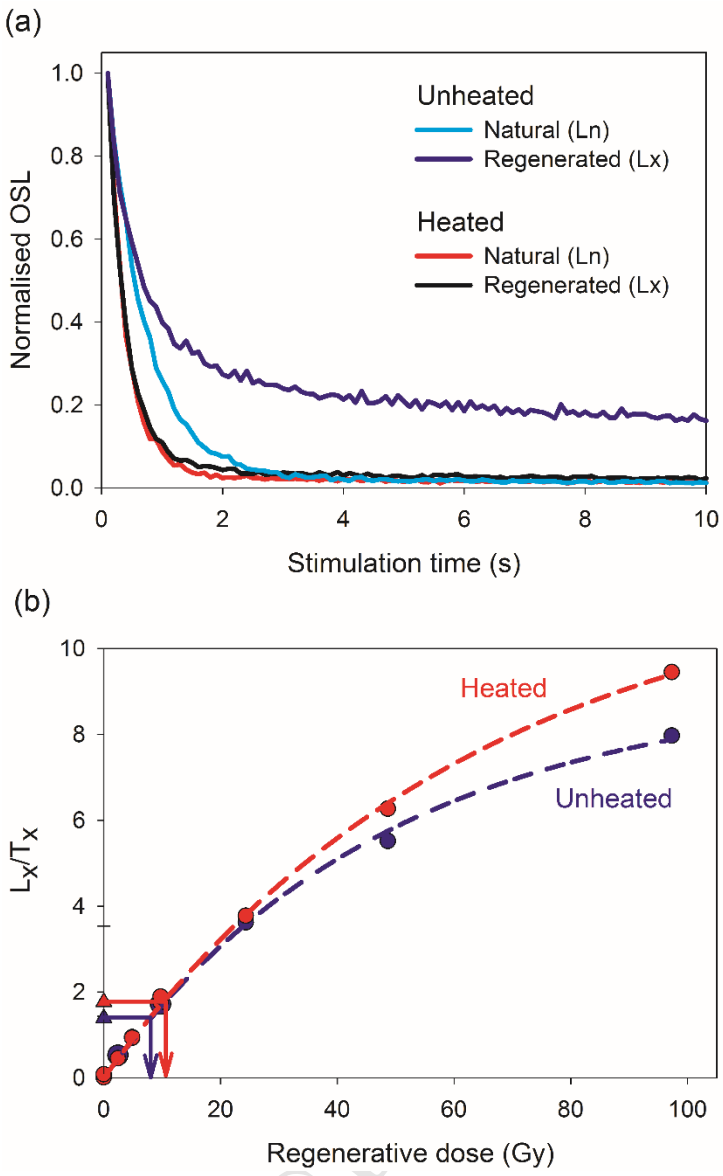

(a)
Normalised OSL
Stimulation time (s)
Unheated
Natural (Ln)
Regenerated (Lx)
Heated
Natural (Ln)
Regenerated (Lx)
(b)
L_x/T_x
Regenerative dose (Gy)
Heated
Unheated

Figure 2

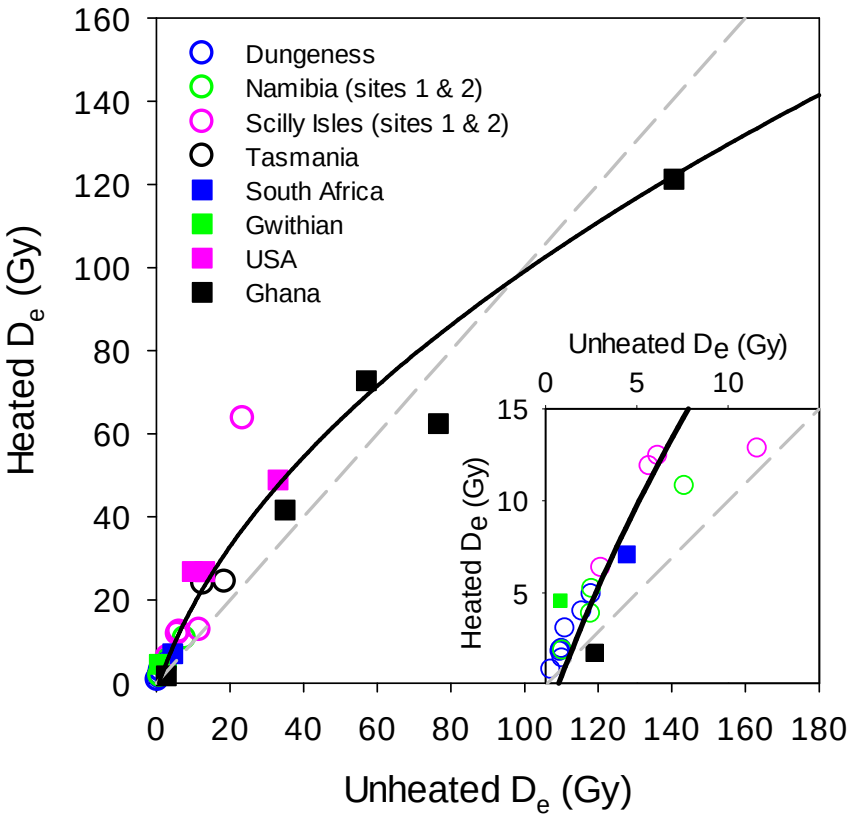
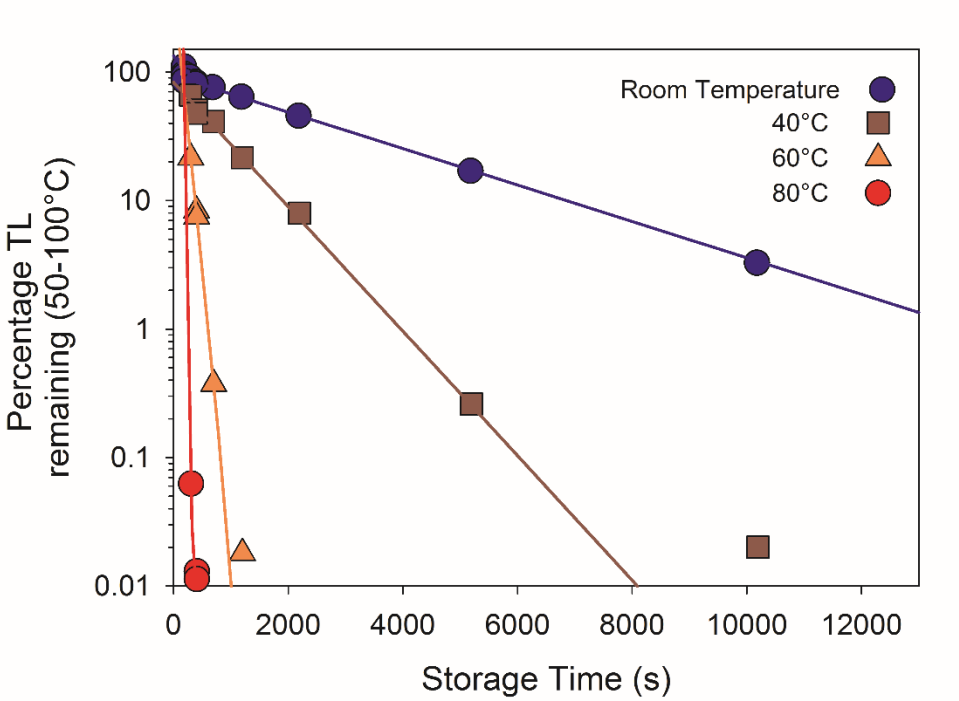


Figure 3

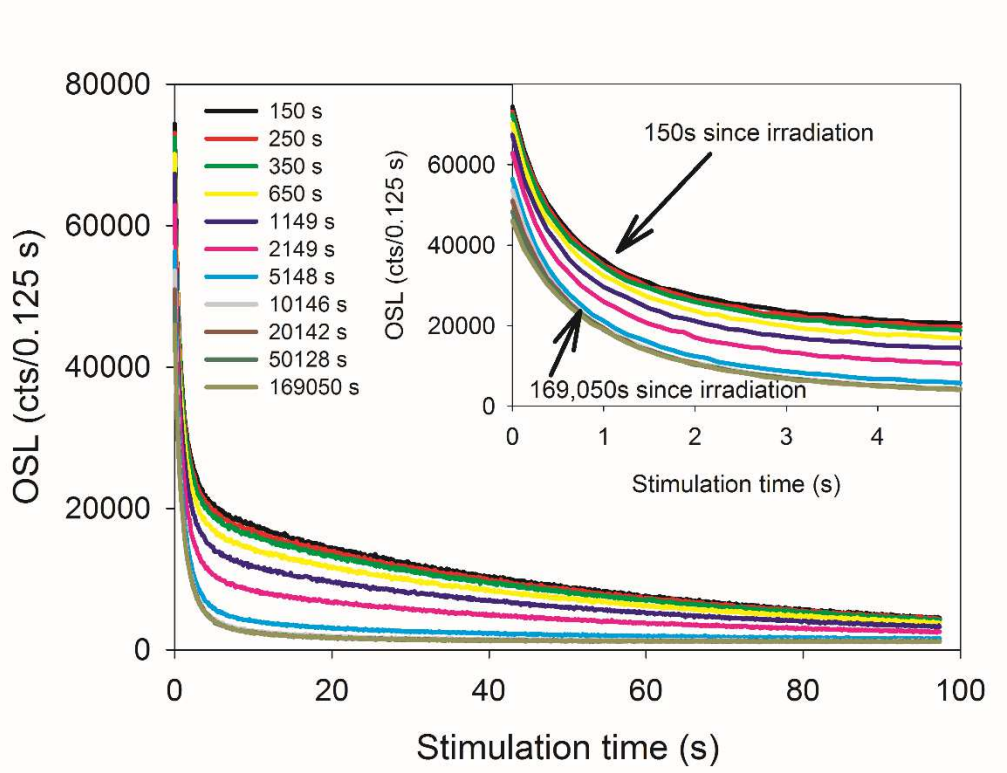


1

2

3

1 Figure 4:

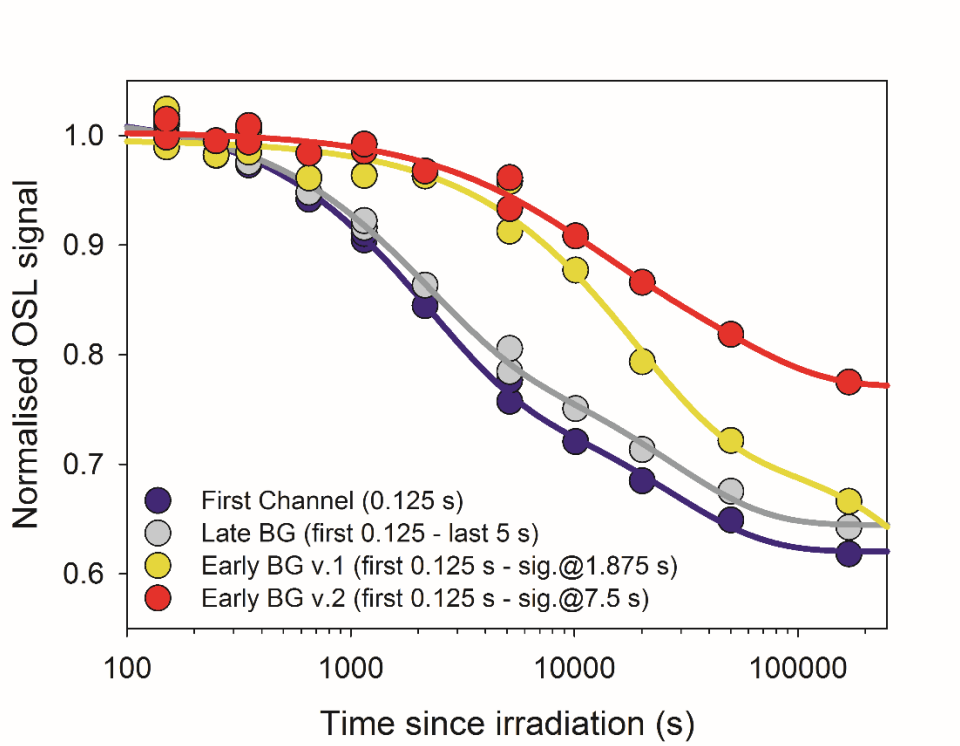


2

3

4

1 Figure 5:



2

3

4

Figure 6:

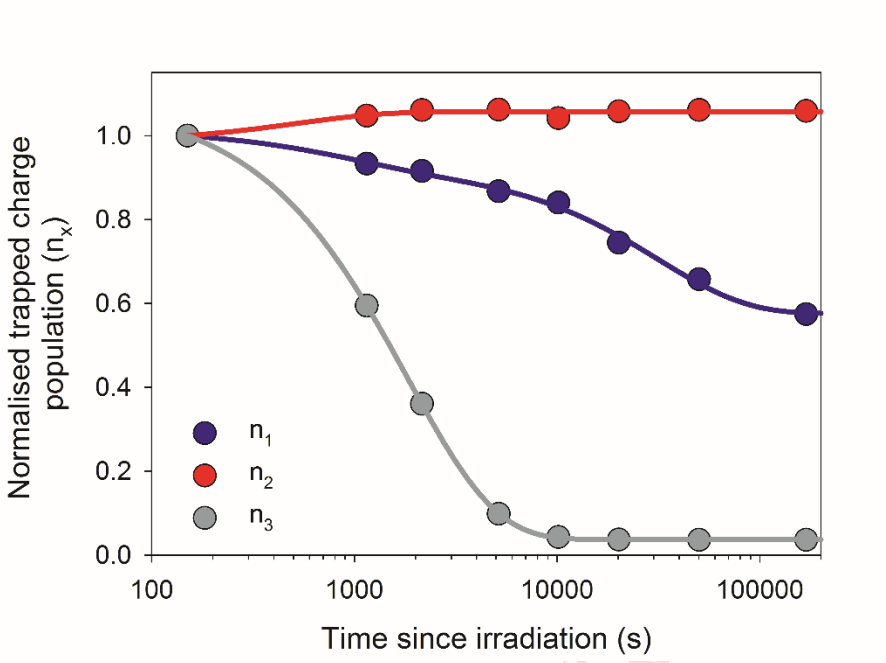
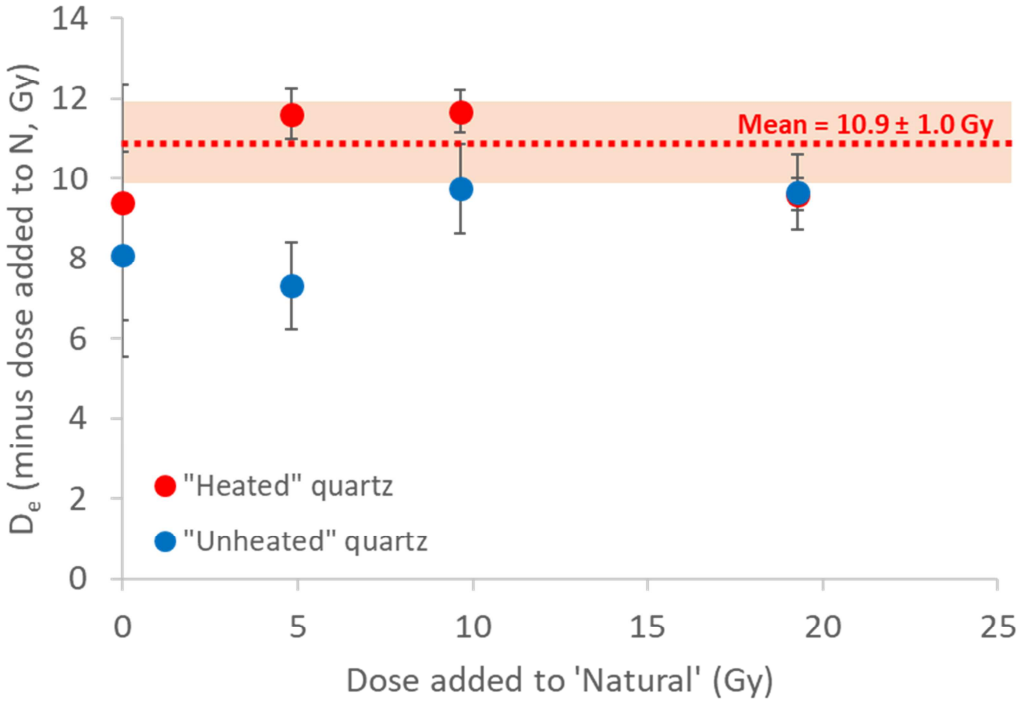


Figure 7



Science Highlights: Roberts et al., "Strategies for equivalent dose determination without heating, suitable for portable luminescence readers"

Paper by: H.M. Roberts, G.A.T. Duller, M. Gunn, C.R. Cousins, R.E. Cross, & D. Langstaff,

- Portable luminescence readers increasingly used but heating samples is problematic
- 3 strategies tested in the laboratory to obtain D_e values for dating without heating
- Relationship between unheated and heated D_e values gives D_e correction factor
- OSL curve deconvolution gives a stable OSL signal from unheated quartz
- Adding small beta dose to fill 110 ˚C trap before measuring unheated Ln gives true D_e