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Duller, G. A. T.; Roberts, H. M.; Pinder, R. C.

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Duller G.A.T.*, Roberts H.M. and Pinder R.C.

Dept Geography and Earth Sciences, Aberystwyth University, Aberystwyth, SY23 3DB, UK

* Corresponding author: ggd@aber.ac.uk

Abstract

Thermal pretreatments are used prior to optically stimulated luminescence (OSL) measurements in methods of dose determination, but the reproducibility of such heating appears to be poor within laboratories, and between laboratories. Monitoring the position of the 110°C thermoluminescence (TL) peak in quartz can be used to routinely assess the reproducibility of heating within a repeated set of measurements on an aliquot (e.g. during the single aliquot regenerative dose protocol) providing feedback on whether changes in sample preparation can improve reproducibility. Poor reproducibility between measurements from a single aliquot are likely to arise from grains between the hotplate and sample holder. Variability between aliquots may also be affected by deformed sample holders. Differences between readers may also be caused by differences in the calibration of the hotplate. Reproducibility of heating can be improved within runs, and between instruments, by taking care to avoid loose grains, by removing deformed sample holders, and if necessary by calibrating hotplates. It is suggested that individual aliquots should only be accepted if the apparent 110°C TL peak temperature within a sequence of measurements varies by 5°C or less. Improving the reproducibility of thermal treatment is important in reducing scatter in equivalent dose determinations based on OSL from quartz, but also for signals such as TT-OSL from quartz, and the various feldspar luminescence signals. Temperature control is also critical for accurate kinetic analyses.

Keywords: Quartz; OSL; thermal lag; equivalent dose determination; precision

1. Introduction

A recent paper by Schmidt et al. (2018) highlighted the variability in the apparent temperature at which the 110°C TL peak in quartz was observed in different laboratories. In that study, sub-samples of the same quartz were distributed to eight laboratories, and a simple series of measurements were undertaken, including thermoluminescence (TL) to a temperature of 160°C. The temperature at which the maximum signal of the "110°C" TL peak was measured varied from 81 to 141°C when using a heating rate of $5°C.s^{-1}$, a range of 60°C. Such poor reproducibility in TL measurements is not new (e.g. Betts et al. 1993; Betts and Townsend 1993; Ege et al. 2007; Jain et al. 2007), and past studies have focussed on the role of thermal lag (the difference between the temperature of the hotplate and the temperature of the sample) as a persistent problem (e.g. Kitis et al. 2015). Schmidt et al. (2018) undertook their inter-laboratory comparison to assess the reproducibility with which kinetic parameters of the 110°C TL peak could be determined. In addition to being important for determination of kinetic parameters, control of the temperature of samples is also important for methods of dose determination in geological, archaeological and dose reconstruction applications.

The aim of this paper is to describe an approach that uses the 110°C TL peak as a means of routinely assessing the reproducibility of sample heating, thus allowing researchers to monitor their data and hopefully improve their reproducibility of the thermal pretreatment. The method is used to assess the reproducibility of heating at three different scales: (a) between replicate measurements on a single aliquot; (b) between different aliquots on the same instrument; and (c) between different aliquots on different instruments. The impact of inconsistent heating between measurements upon equivalent dose (D_e) determination using a single aliquot regenerative dose (SAR) method is explored.

2. Instrumentation

All measurements presented here were made using Risø TL/OSL instruments (Bøtter-Jensen et al. 2003a). Other instruments such as Lexsyg systems (Richter et al. 2013, 2015), which were part of the Schmidt et al. (2018) inter-laboratory comparison, could also use the same approach described here. Data are presented from 8 Risø TL/OSL readers, the oldest of which was installed in 1989, and the most recent in 2015. The older readers have been upgraded such that the control electronics date to 2009 or later. Samples of quartz were mounted on aluminium discs (~0.095 g in mass) obtained from the manufacturer, and all heating was undertaken with oxygen free nitrogen flowing through the measurement chamber at a rate of 1 litre per second. A heating rate of $5°C.s^{-1}$ was used throughout, and data were collected such that there was one data point per degree centigrade. Quartz from a variety of samples was measured, but in all cases these grains were ~200 µm in diameter, and were mounted on the aluminium discs using Silkospray™ silicone oil to produce a monolayer.

The heating system of each of these readers was calibrated following the procedure described by Thomsen et al. (2007), which involves measuring the voltage across the thermocouple attached to the hotplate at a range of temperatures. The data are then used to correct for the non-linearity in the output of the K-type thermocouple. After calibration, the offset between the temperature specified by the controlling software and the temperature measured by the thermocouple is typically better than ~1.0°C. However, as noted by Thomsen et al. (2007), the data used for this calibration are collected with the hotplate at a static temperature. The accuracy of the temperature of the hotplate whilst it is ramped linearly through time (as is undertaken in preheating and TL procedures) is not checked by this calibration. Additionally, the calibration procedure described by Thomsen et al. (2007) does not allow any assessment to be made of thermal lag between the heater and the sample, or of whether such lag is variable from one measurement to another, either on different aliquots or on the same aliquot. To achieve this we need a method to directly measure the temperature of the

sample. This may be possible using a thermopile, but is not practicable for routine operation within the configuration of most commercially available instruments.

3. Use of the 110°C TL peak for monitoring heating

An alternative approach is to use a distinctive TL peak to directly assess the temperature of the sample. Fortunately the 110°C TL peak in quartz is ideal for this purpose as it is very common amongst different quartz samples after they have been irradiated, and although it may consist of emission from more than a single defect (Petrov and Bailiff 1995), it behaves as a first order peak, so that the peak position does not vary with charge concentration in the defect. The emission is most strongly seen at ~ 380 nm (Krbetschek et al. 1997) and is therefore visible when using filters such as the U-340 that are commonly employed when using blue or green stimulation for OSL. There have been reports of emissions from a peak at about 100 to 110°C at wavelengths of ~600 nm (see discussion in Scholefield et al. 1999), but much less is known about this signal and all the work reported here has focussed on the blue-UV emission detected through a U-340 filter.

To assess the suitability of the 110°C TL peak to provide a consistent assessment of heating of the sample, a single aliquot of 180-211 µm quartz (5 mm diameter of the disc was covered in grains) purified from a fluvial sediment from Kalambo Falls, Zambia (Duller et al. 2015) was irradiated with doses ranging from zero to 66 Gy (Fig. 1a). After each irradiation the sample was promptly heated to 450°C at a heating rate of $5°C.s^{-1}$. The position of the TL peak (T_m) was calculated by fitting the data with a Weibull function (Eq. 1) which has been shown to be well suited to analysis of the 110°C TL peak (Pagonis et al. 2001). This equation takes the form:

$$L(T) = 2.713 \, I_m \left(\frac{T-T_m}{b} + 0.996 \right)^{15} e^{-\left(\frac{T-T_m}{b} + 0.996 \right)^{16}} \qquad \text{Eq. 1}$$

where the luminescence (L) emitted at a temperature (T), can be fitted with three parameters, T_m, the peak temperature, I_m, the intensity of the luminescence signal at this peak, and b, a parameter describing the width of the peak.
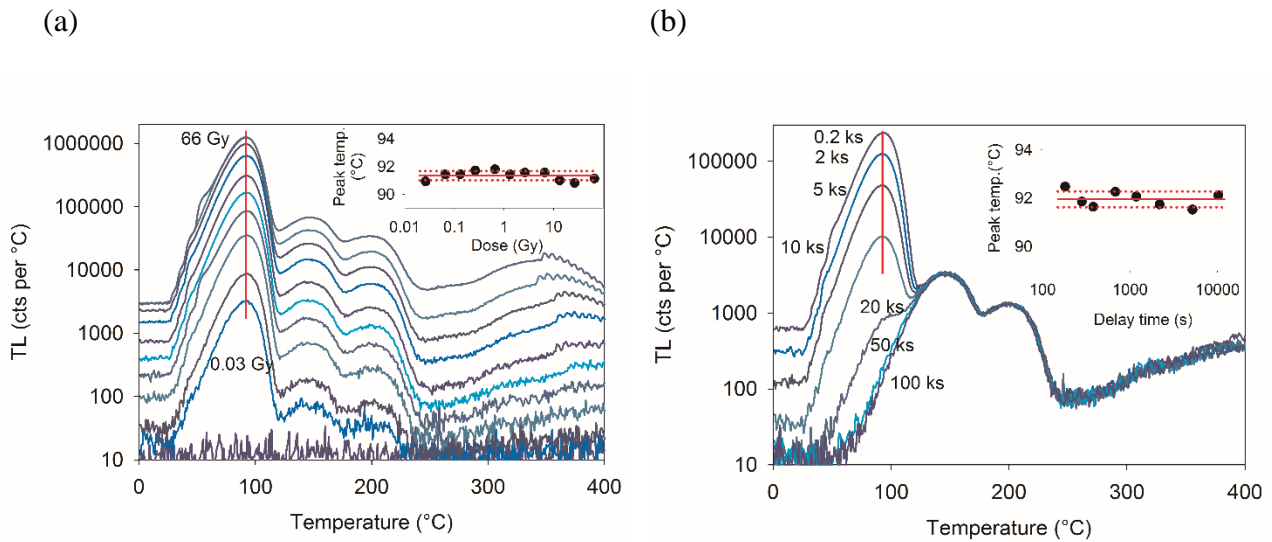
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(a) (b)

Figure 1: *Thermoluminescence measurements of one aliquot of quartz (180-211μm diameter grains) from sample 105/KB15 from Zambia. All measurements were made at a heating rate of 5°C/s in a nitrogen atmosphere. The sample is mounted on a ~0.095g aluminium disc. (a) Measurements following radiation doses of between 0 and 66 Gy. The position of the 110°C is invariant. (b) Measurement of the same aliquot after receiving a dose of 1.3 Gy, and then waiting for periods of time from 0.2 ks to 100 ks. The inset shows the apparent peak temperature of the TL signal obtained by fitting as a function of the delay time between irradiation and TL measurement. Note that for clarity TL data is only shown for some delay periods, but the inset shows data for all 8 of the different delay periods that were measured where a peak temperature could be calculated. Fitting uncertainties on the peak temperatures in the insets are within the symbols.*

With the exception of the measurement taken when no dose had been administered, an average peak temperature of 91.4 ± 0.3 °C (average ± standard deviation of the data, with a coverage factor k = 1) (n = 11) was calculated. As expected from previous studies (summarised in Bøtter-Jensen et al. 2003b), there is no evidence for any systematic changes in peak position with dose. A second, similar experiment, was undertaken where the aliquot was given a dose of 1.3 Gy, and then left within the reader (at room temperature) for varying periods of time so that the time between irradiation and measurement varied from 180 s to 100,000 s (1 day 4 hours) (Fig. 1b). The intensity of the 110°C TL signal decreases with storage time, as expected given the E and s values published for the peak (e.g. Spooner and Questiaux 2000). For periods exceeding 10 ks (2.8 hours) the peak becomes indistinct, becoming a shoulder of the more stable peak at 145°C, and at the longest delay

time of 100 ks no indication of the 110°C TL peak is seen. To allow use of the Weibull function even where the 145°C TL peak exists as a prominent shoulder, only the brightest part of the TL peak is used for fitting. The procedure followed is to find the maximum intensity of the peak in the range 50 to 140°C, and then to select data on either side of this peak for fitting if the intensity is greater than or equal to half that maximum intensity (see red line on Fig. 2 to show which part of the data set has been used for fitting). A Figure of Merit (FoM) (as used in Pagonis et al. 2001) is calculated and can be used to assess the goodness of fit. For the data in Fig 1b, the data for delays of 10 ks or shorter have FOM values of 1.1% or less, and give a consistent position for the peak of 92.0 ± 0.3°C (n = 8). Data collected after longer delay periods could not be fitted.
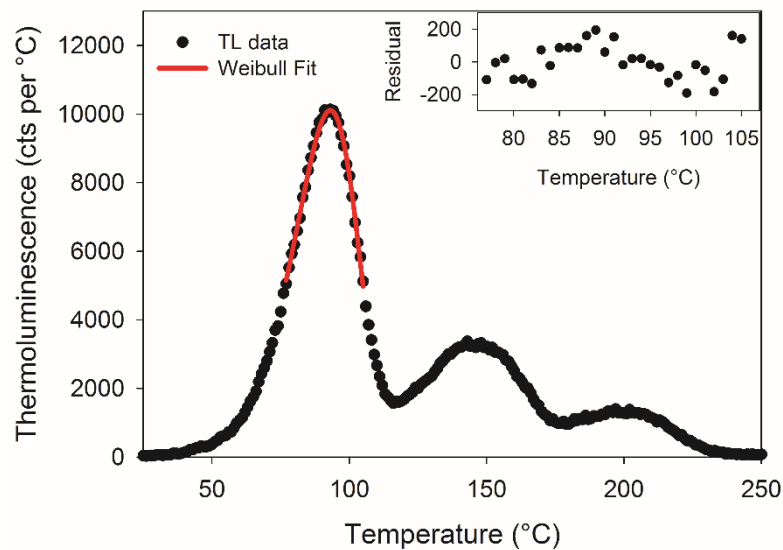

Figure 2: *Example of fitting the Weibull function (Eq. 1). The red line is the fitted Weibull function, and shows which part of the data is used for fitting (i.e. only where the signal is at least half the maximum intensity of the 110°C TL peak). This avoids the complicating impact of adjacent peaks such as that apparent at 145°C.*

The characteristics of the 110°C TL peak make it well suited as a monitor for the temperature of the sample (as distinct from the temperature of the hotplate). Furthermore the peak is well suited for providing detailed monitoring within the different cycles of a SAR protocol because quartz is routinely preheated to temperatures varying from 160 to 300°C in SAR (Wintle and Murray 2006), and so the 110°C TL peak can be observed during the preheats, prior to every OSL measurement of both the regenerative dose (L_x) and test dose (T_x) responses. The method suggested here is that the temperature of the TL peak observed as part of the preheats should be obtained by fitting Eq 1 and used to monitor the reproducibility of the peak temperature as part of quality control.

4. Application of the monitoring method

4.1 Reproducibility of heating

To assess the reproducibility of the apparent peak temperature within a single instrument, and between different instruments, SAR data collected for a series of beta source calibration measurements were analysed. The same batch of calibration quartz (Batch number 118, which had received a gamma dose of 4.81 Gy) supplied by DTU Nutech was used for all measurements (Hansen et al. 2015), and data from 8 different instruments were collected. For each reader, 6 aliquots (5 mm diameter coverage of the aluminium disc) were measured. The same sequence of commands was used for all instruments, and the test doses and regenerative doses administered were within 10% of each other between readers (dose rates on different instruments varied from ~0.01 to 0.09 Gy.s^{-1}).

Thermoluminescence data were fitted with a Weibull function as described in section 3, to obtain the best estimate of the peak temperature (T_m) for the '110°C' TL peak for each preheat. For each aliquot this resulted in at least 13 values of T_m through the SAR cycle, making it possible to assess the variability in the temperature for a single aliquot. Of the 48 aliquots measured, 42 of them had a range in apparent peak temperature of less than 3°C in the replicate analyses (Fig. 3a). As might be
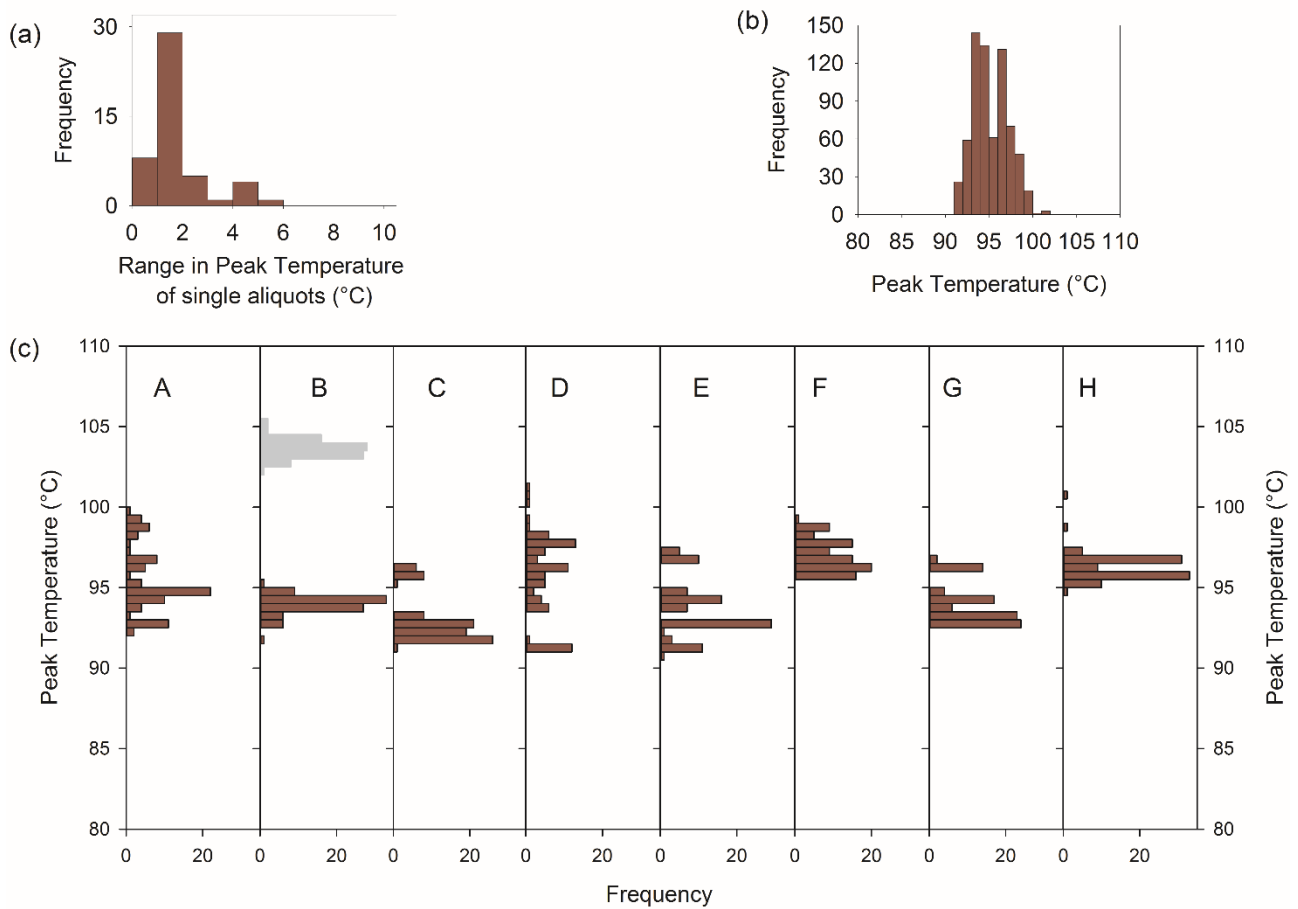
Figure 3: (a) Histogram of the range of peak temperatures observed for individual aliquots throughout the SAR sequence. Data are shown for 48 aliquots of calibration quartz (batch 118). (b) The individual apparent peak temperatures of the 584 measurements of the 48 aliquots across 8 instruments used for measurement. (c) Histograms of the apparent peak temperatures for each instrument (indicated by the letters A to H). Six aliquots were measured for each instrument, and a minimum of 11 preheats were analysed for each aliquot, giving a minimum of 78 analyses for each instrument. For instrument B the histogram in grey shows a set of data collected when the hotplate had not been calibrated.

expected, the variation in apparent peak temperature between aliquots on a single reader was greater than that within an aliquot, and ranged from 3.3 (Instrument B, Fig. 3c) to 9.7 °C (Instrument D).

Across the whole data set for the 8 instruments, the range in apparent peak temperatures is 10.0 °C (Fig 3b and c), and the average peak temperature is 94.8 ± 2.0°C. Analysis of the Schmidt et al. (2018) data collected at a heating rate of 5°C.s^{-1} gives a range of 60.2°C (81.1 to 141.3 °C), but that compilation included analyses with different sample holders (e.g. aluminium and steel of different thicknesses, and both cups and discs) and with instruments that Schmidt et al. (2018) suggest may

not have had their hotplates correctly calibrated. The impact of calibrating the hotplate is illustrated in Fig. 3c for Instrument B which was first installed in the early 1990s, prior to the routine calibration that occurs now during manufacture. The data shown in grey were collected when the hotplate had not been calibrated, while those in red were collected after calibration. The average peak temperature prior to calibration is $103.4 \pm 0.5°C$, while that after calibration is 93.7 ± 0.6 °C for Instrument B.

4.2 Application to measurement of a natural dose

To illustrate the application of this method for monitoring thermal contact when dating a geological sample, two sets of SAR measurements undertaken to determine D_e for sample Aber179/6916 (an aeolian dune in the United Arab Emirates (Farrant et al., 2015)) are discussed: quartz of 180-211 µm diameter was used for all measurements. The first set of measurements were made for that publication, while a second set were made more recently by a different operator. In both cases a preheat of 260°C for 10 seconds was used for the regenerative doses and a preheat of 220°C for 10 seconds was used for the test doses, both using a heating rate of $5°C.s^{-1}$. Figure 4(a, b) shows dose response curves for two aliquots, one from each set of measurements. The data in Fig. 4a is for an aliquot collected for Farrant et al. (2015), produces a set of L_x/T_x ratios that can be well fitted with a single saturating exponential function, and there is little scatter in the data about this line. In contrast, the data for the other aliquot collected more recently, is more scattered (Fig. 4b). The 110°C TL peak positions observed during the preheats prior to each OSL measurement (except the natural and zero regeneration dose points where no 110°C TL peak occurs) are plotted in Fig. 4c and show that while the peak temperature was very stable for the aliquot with low scatter in L_x/T_x about the dose response curve (range in peak position was 0.3°C), the TL peak position varied by 14°C for the aliquot with large scatter in the L_x/T_x values. Subsequent inspection of this second aliquot after completion of the measurements showed many grains of quartz adhering to the edges of the disc (Fig. 4e), rather than being confined to the centre of the disc as intended (Fig. 4d). It is
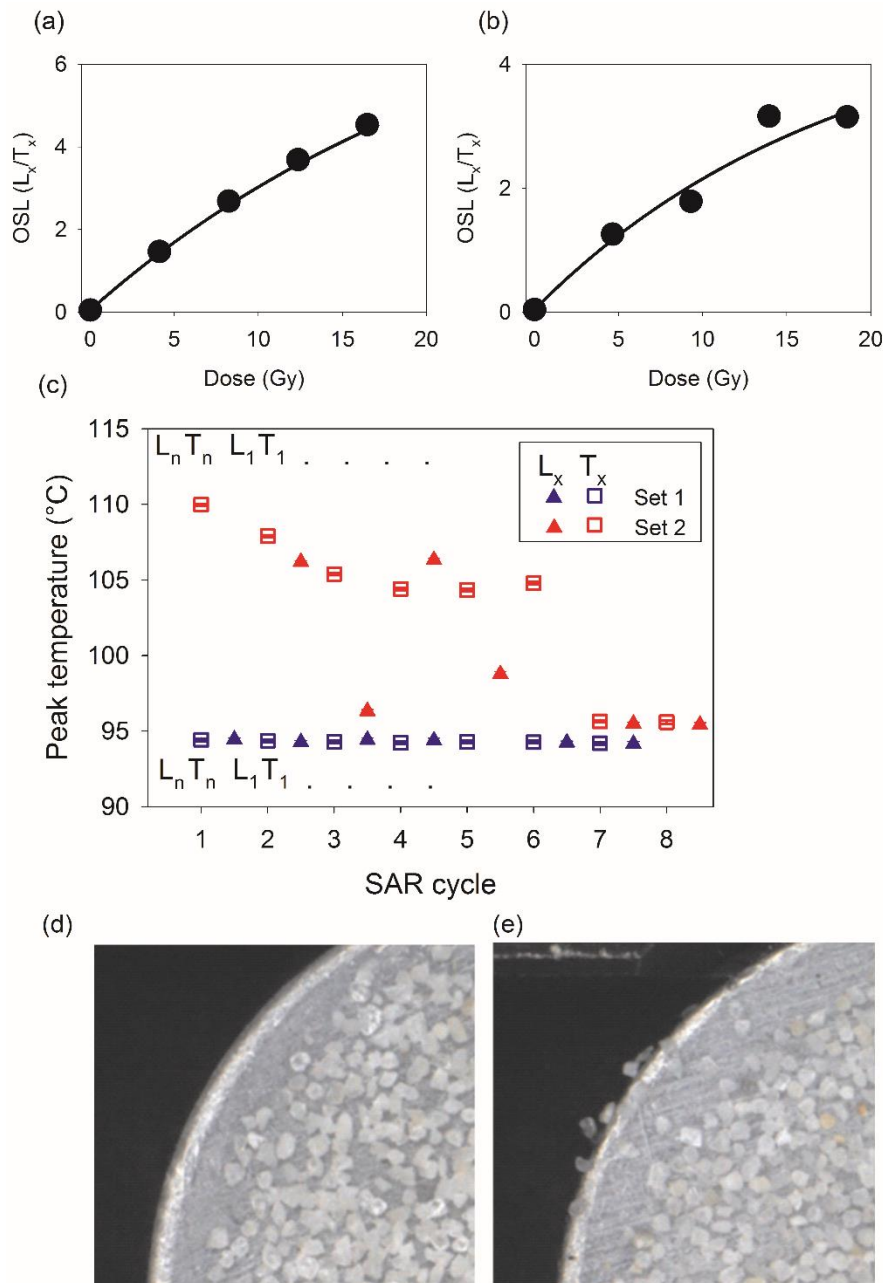
Figure 4: *Data for an aliquot of quartz from sample Aber179/6916 from the United Arab Emirates. The dose response curve fits the data well (a), and the peak temperature for the 110°C TL peak is reproducible (c, blue triangles and squares). The disc has no grains nears its margin (d). In contrast, dataset 2 has an irregular pattern of L_x/T_x (b), very variable peak temperature for the 110°C TL peak (c, red triangles and squares), and the photograph of the aliquot shows a larger number of grains around its margin (e). Note that L_x data are not shown in (c) when no 110°C TL peak is observed (i.e. for the natural (L_n) or when no regenerative dose is given (e,g. cycle 6 for Set 1)).*

assumed that grains could also have found their way underneath the aluminium sample holder, thereby reducing contact between the disc and the hotplate and causing the TL peak position to vary through the sequence of measurements. The impact of the poor thermal contact was not only to reduce the precision of the L_x/T_x ratios for an individual aliquot (e.g. Fig. 4b), but also to increase the scatter in equivalent dose between aliquots. Fig 5 plots D_e values obtained for single aliquots of the sample in the two sets of measurements, and shows that the overdispersion increased from 6±0% for the data set where peak temperatures were reproducible (e.g. blue symbols in Fig 4c) to 16±2% for the data set with much greater variability in peak temperature (red symbols in Fig 4c). The two sets of data yielded Central Age Model values of 11.9±0.2 and 12.8±0.9 Gy respectively, showing the larger uncertainty in the second data set.

The impact of thermal treatments upon the fast component of the quartz OSL signal has been studied extensively by Wintle and Murray (1998, their Fig. 3 and 4) who showed that changing the preheat from ~150°C up to ~260°C had little impact upon the size of the OSL signal. Above this temperature, the OSL signal drops rapidly as preheat temperature is increased, presumably due to thermal eviction of charge from the 325°C TL peak which is the trap commonly associated with the fast component of the quartz OSL signal. This pattern is very commonly seen in data collected during a preheat test, as shown by Roberts and Duller (2004, their Fig. 3b). Small variations in the temperature to which a quartz aliquot is heated is likely to have little impact in the range below 260°C, but a much greater impact above this temperature.

Fig. 6 shows data from a preheat dose recovery test for small aliquots of quartz from sample 219/TSW03 (Larkin et al. 2017). Eighteen aliquots were bleached with blue LEDs (470 nm) for 100 seconds at room temperature, paused for 10,000 seconds to reduce the 110°C TL peak, bleached again for 100 seconds using the blue LEDs, and then given a dose of 3.1 Gy. The aliquots were subdivided into 6 groups, each with 3 aliquots. Each group was measured using the same SAR protocol except that the preheat temperature prior to measurement of L_x varied from 160 to 260°C
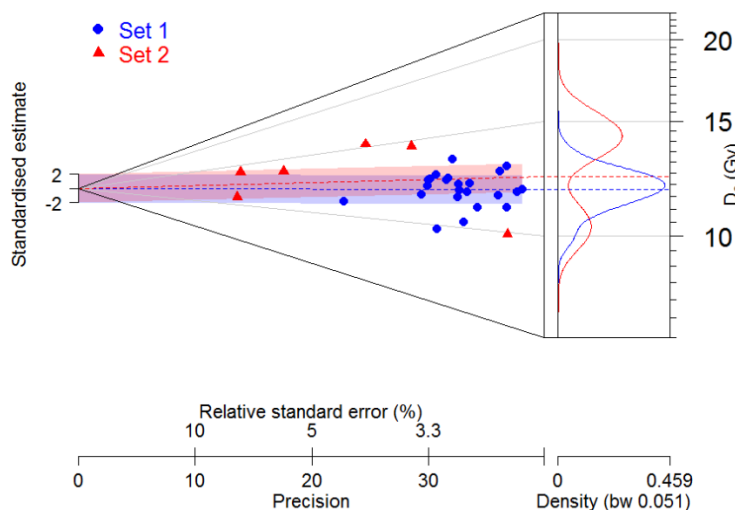
Figure 5: *Blue circles are those with good thermal reproducibility (Set 1 in Figure 4) and the resulting D_e values have an overdispersion of 6.3%. Red triangles are those with poor reproducibility (Set 2 in Figure 4) and have an overdispersion of 15.5%.*
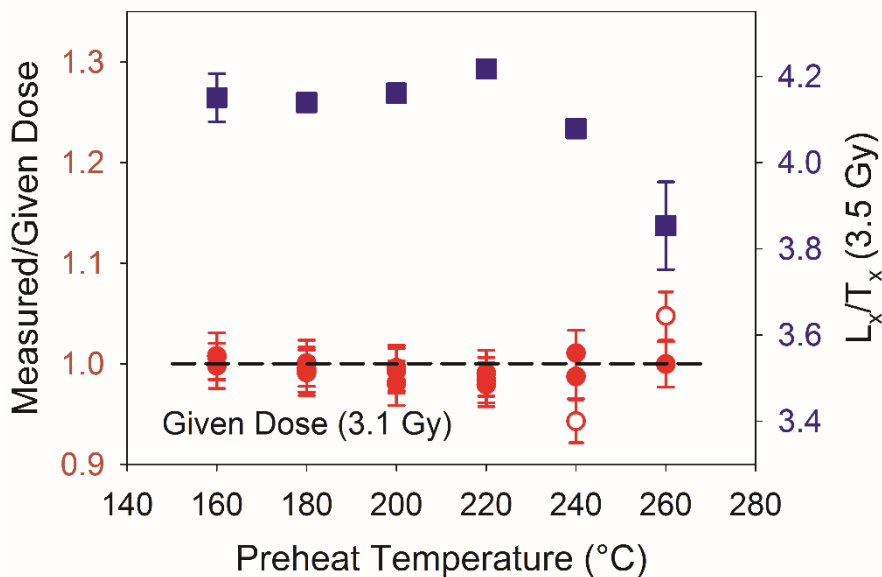
Figure 6: *Preheat Dose Recovery data for 219/TSW03. The red points give the ratio of the measured dose to the given dose (3.1 Gy) for each aliquot. Greater scatter is apparent at preheats of 240 and 260°C, and the two points shown as open circles have much higher scatter in the position of the 110°C TL peak. The blue squares plot the L_x/T_x ratio for the 3.5 Gy regenerative dose point at each preheat temperature and illustrate the loss of OSL at temperatures of 240°C and above.*

(held for 10 s). For all aliquots the same preheat (heating at 5°C.s^{-1} to 160°C followed by immediate cooling) was used prior to measurement of the signal T$_x$ arising from the test dose. The blue squares show the average ratio of L$_x$/T$_x$ for measurements of a regeneration dose of 3.5 Gy (with a test dose of 0.8 Gy). As observed previously (Roberts and Duller, 2004), changing the preheat temperature had little impact upon the normalised OSL signal (L$_x$/T$_x$) for preheats of 220°C or lower, but at preheats of 240°C and higher the signal drops. The dose recovery ratios (red data points on Fig 6) are very close to unity at all preheat temperatures, but are more scattered when using preheats of 240 or 260°C. In our experience, this increased scatter at higher preheat temperatures is a common observation. The range in apparent peak temperature for individual aliquots averages 4.0°C for the whole suite of 18 aliquots, but for the aliquot at a preheat of 240 and the one at 260°C that yield discrepant dose recovery values (shown as open red circles on Figure 6), the range is much higher, at 9.5 and 6.5°C respectively. The impact of this difference in heating upon the resulting D$_e$ depends upon where the differences in heating occur within the SAR sequence, but illustrates the potential impact of poor reproducibility upon D$_e$ determination.

5. Can we look at heating prior to measurement of the natural OSL signal?

The procedure described above can be applied to a SAR protocol with no modification to the treatment of the sample. However, one problem with this approach is that it is not possible to measure the 110°C TL peak from quartz when no laboratory regeneration dose is given. This is the case when measuring the natural signal, yet arguably this is the most important measurement of the SAR protocol. Additionally, if grains have adhered to the underside of a sample disc, or if the disc was not correctly mounted on the carousel, then poor thermal contact is likely to be most severe during the first few measurements. A potential solution is to give a small dose to the sample prior to measurement of the natural. Adding a laboratory irradiation prior to measurement of D$_e$ is not a new approach: Roberts et al. (2018) describe experiments using such an approach, and Mejdahl and
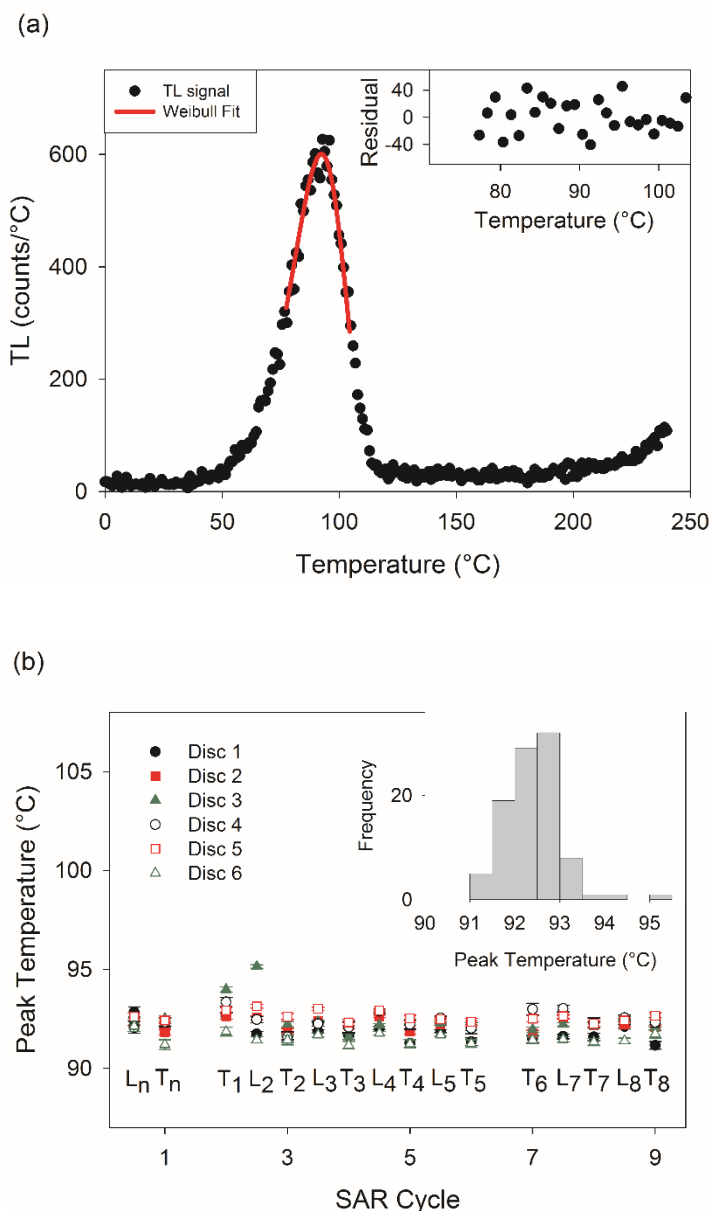
Figure 7: *Data for quartz from the beach sample used for the inter-comparison exercise of Murray et al. (2015). (a) An example of TL data collected during preheating to 240°C after adding 0.7 Gy onto the natural. The fit for the Weibull function used to calculate the peak temperature is shown in red, and the residuals to the fit are shown in the inset. (b) Peak temperatures for 6 discs of the quartz during a SAR sequence. No data can be calculated for L_1 or L_6 as the regeneration doses are zero. The inset is a histogram of the peak temperatures for all 96 TL measurements. The mean value is 92.2 ± 0.6 °C.*

Bøtter-Jensen (1994) proposed the single aliquot regeneration of additive doses (SARA) method 25 years ago. SARA involves giving a range of different additive doses prior to measurement of D_e, and has been used in a number of studies since then (e.g. Timar-Gabor and Wintle 2013; Porat et al. 2018). The approach suggested in this paper is to give a small dose prior to measurement of D_e of each aliquot, and for this value (in Gy) to be subtracted from the final D_e calculated for the aliquot, thus giving the D_e arising from irradiation during burial. Since the 110°C TL peak is very sensitive, only a small dose is required.

Figure 7 shows data obtained using this approach for quartz from a beach ridge in Denmark that was used in a recent inter-laboratory dating comparison exercise. The sample has a mean D_e (based on data 22 determinations by participating laboratories) of 4.48 ± 0.12 Gy (Murray et al. 2015). Six aliquots of the quartz were given a 0.7 Gy dose prior to the start of a SAR protocol. This meant that a 110°C TL peak could be observed in the preheat data acquired prior to measurement of the natural OSL signal, as well as prior to all subsequent test dose and regenerative dose responses. The TL signal obtained after a dose of 0.7 Gy was well fitted with the Weibull function, with no structure to the residuals (Fig. 7a), and yielded a peak temperature of 92.4 ± 0.2 °C (uncertainty is derived from curve fitting). Values of the peak temperature are consistent both within the measurements for a single aliquot and between aliquots (Fig 7b). The D_e values obtained for these six aliquots showed little overdispersion (8%), and after subtraction of the 0.7 Gy added to the sample prior to measurement of the natural signal, yielded a value of 4.18 ± 0.20 Gy, consistent with the value of 4.48 ± 0.12 Gy obtained during the intercomparison exercise (Murray et al. 2015). Finally, it is interesting to note that the peak temperature values for all 96 measurements made on these six aliquots of the intercomparison sample have a mean of 92.2 ± 0.6 °C (standard deviation), close to the mean value from the calibration quartz on this instrument (93.7 ± 0.6 °C, Fig 3).

6. Implications for other types of luminescence measurements

This paper has focussed on monitoring the reproducibility of heating during preheating operations as part of a SAR sequence. The same technique is applicable to many experimental designs involving heating quartz, such as those used for kinetic analysis. Temperature control is also important during measurement of the OSL signal itself, as many authors have demonstrated (e.g. Spooner, 1994; McKeever et al. 1997). Changes in the temperature of the sample during optical stimulation influence thermal assistance, and optical cross-sections.

Data from a short interval of optical stimulation at the start of an OSL decay is normally summed to characterise the intensity of the signal. However, Jain et al. (2007) have shown that because of thermal lag, when an aliquot of quartz mounted on a sample disc is heated to its measurement temperature (e.g. 125°C), the quartz will only reach the designated temperature sometime after the hotplate. If optical stimulation begins too soon, the grains on the sample holder may still be increasing in temperature during the first part of the OSL measurement. This will make the form of the decay curve complex, and can sometimes be observed as a rise in the intensity of the OSL signal in the first few seconds of optical stimulation. Additionally, if the contact between the hotplate and the sample varies through a measurement sequence (as implied for some aliquots in the measurements shown above) then this will cause additional scatter in the data. Jain et al. (2007) recommended pausing for 15 seconds after raising the hotplate to the measurement temperature required, in order to ensure that the sample reaches a stable temperature. Whilst this is important for quartz OSL measurements, the IRSL signal and the pIR-IRSL signal are even more strongly temperature dependent (e.g. Duller and Wintle 1991), and so these pauses are especially important. Jain et al. (2007) also noted that the impact of thermal lag could be reduced by using low heating rates (e.g. 1° C/s).

Rock slices (between 0.4 and 1.2 mm in thickness) are likely to be especially prone to thermal lag because of their mass (e.g. Fig 1a of Sellwood et al. 2019), and both of the approaches described

here have been adopted to mitigate this problem (e.g. Sohbati et al 2015 use a pause of 30s between reaching the desired measurement temperature and starting optical stimulation, while Jenkins et al. (2018) used a heating rate of 1°C/s and then waited for 100 seconds prior to the start of optical stimulation).

Another signal that is likely to be very sensitive to the sort of variations in thermal treatment shown here is thermally-transferred OSL (TT-OSL; Duller and Wintle 2012). The transfer of charge into the fast OSL trap is achieved by heating the sample, evicting charge from defects and allowing transfer to other traps. The strong temperature dependence of the magnitude of the TT-OSL signal (e.g. Fig 4a of Wang et al. 2006) makes this type of measurement very sensitive to small variations in thermal contact. Measurement of the 110°C TL peak is feasible in TT-OSL measurements, and so this approach of monitoring the variability in heating should also be of value here.

It is important to appreciate that thermal lag is very strongly influenced by the user. Taking care to avoid grains on the sides or base of the sample holder, and on the hotplate, is critical. Using sample holders that are not deformed is also important, as is ensuring that grains form a monolayer on the sample holder. Identifying these problems can sometimes be challenging, and the approach described here for monitoring the apparent peak temperature provides a routine way of checking for such problems. It is also important that the thermocouple and heating system in instruments are calibrated (e.g. Thomsen et al. 2007) so that the temperature of samples is not only reproducibly precise, but also accurate.

7. Conclusions

Thermal control of samples during luminescence measurements should not be taken for granted, and the method outlined here based on monitoring the 110 °C TL peak in quartz provides a method for routinely checking the reproducibility of heating. The method requires no additional

measurements to those normally undertaken for SAR (the only exception is if a dose is added to the natural as described in section 5, but this is not essential). A number of different factors can cause scatter within replicate measurements on a single aliquot, between aliquots, or between instruments. Grains between the hotplate and the sample holder, or distortion of the sample holder may cause variation in heating within an aliquot or between aliquots. Such effects will also be seen when comparing measurements between instruments, but systematic differences in heating between instruments is most likely to result from differences in the calibration of the heater plate.

The results described above from applying the method outlined in this paper to 8 instruments gives some indication of the reproducibility that is achievable. In replicate measurements of quartz used for beta source calibration, 42 out of 48 aliquots had apparent peak temperatures that could be repeatedly measured within 3°C for an individual aliquot, and for 37 out of the 48 it was less than 2°C. Measurements of 6 aliquots on each instrument gave a range of between 3.3 and 9.7°C and this presumably reflects some differences in thermal lag between discs, possibly due to variations in the flatness of the discs. Looking at data from all 8 instruments, the range in apparent peak temperatures increases only slightly to 10.0°C, implying that the calibration of the hotplates for these 8 instruments are consistent.

Measurements of the apparent peak temperature of two widely available samples have been described, and could form the basis for other laboratories to assess their own data. Quartz distributed for a laboratory inter-comparison (Murray et al. 2015) gave a peak temperature of 92.2 ± 0.6 °C when mounted on ~0.095g aluminium discs and measured at $5°C.s^{-1}$ on instrument B, similar to the value for the calibration quartz (93.7 ± 0.6 °C) on that instrument (Fig. 3). When combining data from all 8 instruments the average peak temperature for the calibration quartz was 94.8 ± 2.0 °C.

A number of quality control measures (e.g. recycling ratio, recuperation, IR-OSL depletion ratio (Duller 2003)) have been proposed in order to assess whether data collected using a SAR protocol should be accepted or not (Jacobs et al. 2006). It would be possible to define threshold values for the reproducibility of the thermal treatment of aliquots that was expected if the D_e data from an aliquot were to be accepted. However, defining such thresholds may be complex, and it is not entirely clear what parameter would be the best to choose. One approach would be to assess the maximum range in apparent peak temperature for replicate measurements on a single aliquot as the key parameter. From the data shown in Fig. 3a, Fig. 7b, and the discussion around Fig. 6, it appears that it should be routinely possible for the range in peak temperature for an individual aliquot to be less than 5°C. A second parameter to assess would be the consistency in the mean apparent peak temperature between aliquots, to ensure that heating of each aliquot is similar. Data in Fig. 3c suggests that within a single reader average apparent peak temperature should vary less than ~5-10°C, but further research is needed to ensure that such variations between aliquots are not reflecting variations in kinetic parameters. At this stage, it would seem prudent to focus on reproducibility between replicate measurements of the 110°C TL peak on a single aliquot, and a variation of 5°C or less is recommended for acceptance of an aliquot.

The impact of variability in preheating upon the OSL signal from quartz may be minimised by using preheats of 220°C or lower (Fig. 6 and discussion in section 4.2), though some samples need the higher preheats to pass appropriate acceptance criteria. Change in luminescence signals due to variability in the measurement temperature and preheating is likely to be greater in feldspars than quartz (Roberts 2007), and so the approach described in this paper would be valuable for that mineral as well. Unfortunately the TL signal of feldspars does not have a narrow first order peak like the 110°C peak in quartz, but it may be possible to monitor the TL emission arising from the test dose in SAR sequences for feldspars to give some indication of reproducibility of heating. Additionally, the good practice learned from improving the reproducibility of making

measurements of quartz should help to improve the reproducibility and accuracy of heating across other materials where reproducible heating is equally, if not more important.

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