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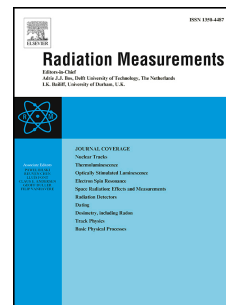
Testing single aliquot regenerative dose (SAR) protocols for violet stimulated luminescence

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Highlights:

- Compared two single aliquot regenerative dose protocols for VSL signal
- Protocols assessed to minimise recuperation and optimise signal depletion
- Partial success in dose recovery of ~400 Gy using post-blue VSL signal
- VSL signal gives 50 – 79 % underestimation of the expected D_e

Abstract

Basic assumptions of the single aliquot regenerative dose (SAR) protocol are tested using the violet stimulated luminescence (VSL) signal from quartz. The VSL signal is shown to be reduced to a sufficiently low background level between SAR steps, and the SAR protocol appears to adequately correct for sensitivity changes during measurement. The VSL SAR protocol can recover a large (405 Gy) laboratory beta dose within uncertainties, however the mean value for the dose recovery ratio is commonly 0.8 or less. This poor behaviour is echoed in the measurements of equivalent dose (D_e) for a sample with an expected D_e of ~354 Gy, which underestimates D_e by 50 – 70 %. Further investigations are required to understand the mechanisms underlying these underestimations in VSL SAR D_e values.

1. Introduction

Violet stimulated luminescence (VSL) is a recently discovered signal from quartz which samples deeper traps than those accessible by blue light (Jain 2009; Ankjærgaard et al. 2013), offering the potential to extend the upper limit of luminescence dating using a stable, non-fading signal. A recent study by Ankjærgaard et al. (2016) attempted to obtain VSL ages for the Luochuan section of the Chinese Loess Plateau, using both a single aliquot regenerative dose (SAR) method (Murray and Wintle 2000) and a modified multiple aliquot additive dose (MAAD) method (e.g. Duller 1996). The modified MAAD method used by Ankjærgaard et al. (2016) produced a relatively good correlation between their VSL ages and the independent chronology for 15 out of 23 samples. This method requires many aliquots of a sample that are divided into sets and given different additive doses. It is a method that requires similar luminescence behaviour between aliquots, and the modification used by Ankjærgaard et al. (2016) also assumes that different samples have similar behaviours. The method can therefore only reasonably be applied to homogeneous, well-bleached sediment.

In environments where heterogeneous bleaching is anticipated, a single aliquot method would typically be applied, but previous attempts to use the VSL signal with a SAR protocol have had limited success. When Ankjærgaard et al. (2016) applied the SAR method to their samples from the Chinese Loess Plateau, they underestimated the expected ages by ~50 % and concluded that the SAR method is problematic for estimation of VSL ages.

This paper explores SAR protocols using the VSL signal, and investigates the behaviour of the VSL signal under different measurement conditions. An optimized SAR protocol is identified, tested using dose recovery experiments, and applied to samples previously dated using other luminescence signals.

2. Samples, instrumentation and measurement parameters

2.1. Samples

The samples used in this study were collected from St Paul's, KwaZulu-Natal, South Africa, originally described in Botha et al. (1994). New samples were collected from the late Quaternary hillslope deposit during 2014. These samples were treated with hydrochloric acid (HCl) and hydrogen peroxide (H_2O_2) to remove carbonates and organic matter respectively. Samples were then dry sieved and further preparation was undertaken on the 180 – 212 µm diameter grain size fraction. Quartz for VSL measurements was extracted through heavy liquid separation using sodium polytungstate (SPT) at densities of 2.62 g cm^{-3} and 2.70 g cm^{-3}. Grains were etched in hydrofluoric acid (HF) to remove the alpha-irradiated outer layer and to remove any remaining feldspar grains. Two samples were selected for this study based on their D_e value, (i) a very young sample (Aber215/STP09) dated by single-grain quartz OSL giving a D_e value (minimum age model) of 1.6 ± 0.3 Gy and (ii) an old sample (Aber215/STP01) dated by single-grain feldspar post-IR $IRSL_{225}$ with an expected quartz D_e of 354 ± 30 Gy based upon the quartz dose rate and the measured feldspar D_e (minimum age model).

This older sample is beyond the range of quartz OSL dating with blue light stimulation (Colarossi 2017).

2.2. Instrument and measurement parameters

All measurements in this study were undertaken on a LexSyg Research system (Richter et al. 2013). Optical stimulation was undertaken with blue LEDs (458 nm, 100 mW cm^{-2}) and violet laser diodes (405 nm, 70 mW cm^{-2}) filtered by an Edmund Optics NT65-072 bandpass filter (centre 405 nm, FWHM 10 nm). Luminescence emitted in the UV region of the spectrum was detected by a Hamamatsu type 9235 PMT filtered by a combination of 2.5 mm Hoya U-340 and 5 mm AHF BrightLine HC340/26 interference filter. Laboratory irradiations were made using a ^{90}Sr/^{90}Y beta source, with a dose rate of 0.0912 Gy s^{-1}.

Measurements were made on the violet luminescence (VSL) signal using single aliquot regenerative dose (SAR) methods. Quartz grains (180 – 212 µm) were mounted in aluminium cups using SilkoSpray™ silicone spray and a 5 mm diameter mask. Data analysis was undertaken in Analyst V4.43.1 (Duller 2015). Dose response curves (DRCs) in all experiments were fitted with a double saturating exponential (DSE) function unless stated otherwise. Equivalent dose values were calculated by integrating the luminescence signals using the initial 2 s of the decay curve and subtracting an early background taken from the following 5 s of the decay curve (unless stated otherwise). An early background subtraction (Cunningham and Wallinga 2010) was used to isolate

the initial part of the decay curves, and to avoid the effects of re-trapping during measurement of the VSL signal (Ankjærgaard et al. 2016). Equivalent dose values were only accepted if (i) the recycling ratio was within 10 % of unity, (ii) recuperation was less than an absolute value of 2 Gy (for the young sample) or less than 5 % of the natural (for the old sample), (iii) the error on the test dose signal was less than 3 standard deviations of the background signal, and (iv) the uncertainty on the test dose signal was less than 20 %.

3. Selecting a suitable SAR protocol

A single aliquot regenerative dose (SAR) protocol was used for the initial VSL measurements by Jain (2009). Since then, basic parameters of the VSL SAR protocol have been altered in an effort to improve measurement results. Ankjærgaard et al. (2013) combined the preheat and blue bleach steps to thermally enhance bleaching of the Fast and Slow 3 (S_3) blue stimulated OSL components, and to avoid using a high preheat temperature that may induce sensitivity change. Subsequently, in an attempt to reduce recuperation, Ankjærgaard et al. (2015) added a high temperature (380 °C for 200 s) violet bleach at the end of each cycle, whilst Ankjærgaard et al. (2016) replaced the high temperature violet bleach with a TL measurement (heating to 500 °C and holding the sample at this temperature for 20 s), and Hernandez and Mercier (2015) included a violet bleach (200 °C for 500 s) after each regeneration dose (L_x) and test dose (T_x) measurement.

In this paper, two SAR protocols were tested. Protocol A has a violet bleach after L_x and T_x (Table 1, steps 5 and 10) based on Hernandez and Mercier (2015) whilst Protocol B has a high temperature clean out after the test dose measurement only (Table 1, step 9) based on Ankjærgaard et al. (2015). A preheat temperature of 280 °C was selected for this experiment due to the potential for re-trapping the VSL signal in the 220 °C and 260 °C quartz TL traps (Ankjærgaard et al. 2013), the possible change in trapping efficiency at 300 °C (Ankjærgaard et al. 2016), and to avoid overly reducing the already dim post-blue VSL signal. All other measurement parameters were kept the same in both protocols to facilitate the comparison of the results.

3.1. Signal transfer during measurement

One of the key assumptions underpinning the SAR protocol is that the luminescence signal being used for equivalent dose determination is reduced to a very low, background level at the end of each measurement cycle. This removal of signal allows a series of regeneration doses to be applied to the same aliquot to build the dose response curve (DRC). The protocol's efficiency in resetting the signal between cycles is frequently monitored by a recuperation test (Murray and Wintle, 2000). However, it is also necessary for the luminescence signal to be reset mid-SAR cycle, in-between measurement of the regenerative dose signal and the subsequent measurement of the test dose signal. It has not previously been clear how this resetting can be checked routinely. However, Colarossi et al. (2018) proposed a method of examining mid-SAR cycle signal resetting by plotting the luminescence signal from the first channel of a test

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dose measurement (T_x) as a function of the signal from the last channel of the preceding regeneration measurement (L_x) for each cycle of the SAR protocol. The intensity of the test dose signal should not depend on the magnitude of the preceding regeneration signal, so the slope of the linear regression should be close to zero. Fig. 1 shows the results of this test for the two VSL protocols investigated in this study. Data for Protocol A (Fig. 1a) have slopes of ~0.2 for three different test dose sizes suggesting that minimal charge is being carried over from the L_x measurement into the T_x measurement and the luminescence signal is being reset effectively. In contrast, the results for Protocol B (Fig. 1b) are not so straightforward, with slopes of 0.5 and 0.8 for test doses of 20 Gy and 50 Gy respectively. Furthermore, the data points representing the natural measurements (Fig. 1b, red symbols) do not plot along the regression line for their respective test dose, instead plotting at much lower levels, and implying large changes in sensitivity.

Figs. 1(c) and 1(d) show the change in test dose signal (T_x) through each protocol. In contrast to Protocol A, a large change in sensitivity is observed for Protocol B between measurement of the test dose relating to the natural (T_n), and those following the regenerative doses. The major difference between the two protocols is the high temperature treatment in Protocol B (380°C VSL measurement for 200 s, Step 9 in Table 1), and this presumably induces the large sensitivity change observed. The impact of this high temperature step upon the shape of the decay curves derived from each protocol is explored below.

3.2. Decay curve comparison

Comparing the natural signal (L_n) to the subsequent test dose response (T_n), data from Protocols A and B show L_n and T_n decay curves to have the same shape (Fig. 2a-b; note that all the decay curves in Fig. 2 have been normalised to the initial point to enable comparison of their shapes). However, comparing the decay curves for all the test dose responses (i.e. T_n and all subsequent T_x) reveals differences between the protocols. Whilst the decay curves for Protocol A still retain the same shape (Fig. 2c), the decay curves from Protocol B (Fig. 2d) show T_n to have a distinctly different shape from the subsequent T_x curves.

The combination of a large change in sensitivity (Fig. 1d), and a difference between the shape of the natural (L_n and T_n) versus the regenerative dose (T_x) signals, suggest that Protocol B would not be suitable for dose estimation. In Protocol A, the additional stimulation after measurement of L_x (Step 4, Table 1) reduces the luminescence signal to a sufficiently low level such that L_x does not influence the subsequent T_x measurement (Fig. 1a), thereby satisfying the signal removal requirement of the SAR protocol. Based on the results of these analyses, all further measurements reported in this paper were undertaken using Protocol A (Table 1).

4. Testing the SAR protocol

A series of experiments were undertaken on the young sample (Aber215/STP09) from St Paul's in South Africa. This sample was previously dated by single grain quartz OSL and gave

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a MAM D_e value of 1.60 ± 0.37 Gy. Although a minimum age model was used to date this sample, its OSL signal is quite well bleached, and the central age model for the single grain data set yields a D_e of 3.00 ± 0.16 Gy.

4.1. Assessing the natural equivalent dose for a young sample

In the first experiment, natural D_e values were determined using VSL Protocol A with a range of preheat temperatures (160 – 320 °C at 40 °C intervals), to determine the size of the natural dose for a young sample Aber215/STP09, which would subsequently be used for dose recovery tests (Section 4.2). Dose response curves were measured using regenerative doses of up to 40 Gy, and fitted with a single saturating exponential (SSE) function. Three D_e values per temperature were determined, and the mean D_e was calculated (Fig. 3a). In spite of the slow rate at which the VSL signal is thought to bleach in sunlight compared with the OSL signal (Ankjærgaard et al. 2013), D_e values are less than 6 Gy at all preheat temperatures. Individual D_e determinations are scattered, as would be expected for very young samples using VSL, but mean values show a monotonic increase with preheat temperature. Negative D_e values at lower preheat temperatures (see Fig. 3a) may be due to low signal intensity resulting from the early background subtraction approach.

4.2. Dose recovery tests using a large laboratory beta dose

The low natural D_e from sample Aber215/STP09 makes it ideal for undertaking dose recovery experiments using an added dose, thereby avoiding any concerns around

bleaching the natural signal in the laboratory. The second experiment was a dose recovery test using the same range of preheat temperatures as in the natural D_e measurements (Section 4.1). New discs were prepared and a laboratory beta dose of 405 Gy was added to the natural D_e. At least three discs were measured per temperature and the mean natural D_e measured at each preheat temperature during the first experiment was subtracted as a representative residual dose from the D_e value measured during this dose recovery test (this was undertaken regardless of whether the mean residual dose was positive or negative). Dose recovery ratios based upon individual aliquots are very scattered. Mean measured/given dose ratios (Fig. 3b) show that preheats at 160 and 320 °C consistently underestimate the given dose, recovering as little as 40 % of the given dose in some cases. Preheat treatments at 200, 240 and 280 °C give mean values (0.94 ± 0.18, 0.76 ± 0.31, and 0.82 ± 0.22, respectively) that consistently underestimate the given dose, but may be considered to 'pass' the dose recovery test once the relatively large uncertainties (19 to 41 % relative standard deviation) are considered.

The relative uncertainties on both sets of measurements shown in Fig. 3(a-b) are fairly large. This is due to the relatively low signal intensity, the narrow signal integration window, and the early background subtraction approach selected during analysis (Section 2.2). In an attempt to reduce the uncertainties and improve the results, the data were also analysed using a late background approach (Fig. 3c-d). This used the signal from 450 to 500 seconds for the background, and retained the 'early signal'

integration limits used in Fig. 3(a-b). Whilst this late-background approach did reduce the relative uncertainties on individual D_e measurements, and gave more reproducible data at each individual preheat temperature, it ultimately resulted in similar residual values (Fig. 3c) compared to the early-background approach (Fig. 3a) but gave less consistent measured/given dose ratios across the range of preheat temperatures (Fig. 3d) albeit with much smaller uncertainties on each datapoint. The cause of this variability in the dose recovery data is not clear. The VSL signal may be affected by re-trapping in the later part of the decay curve (Ankjærgaard et al. 2013), in which case using a late background subtraction may not be appropriate. Thus an early background subtraction would be expected to yield more appropriate, although noisy, results (e.g. Ankjærgaard et al. 2013, 2016). This appears to be the case here.

The measurement conditions selected for further tests and for subsequent dating measurements used VSL SAR Protocol A, with a preheat temperature of 280 °C, and an early signal-early background analysis on the VSL signal for dose recovery or D_e determination. Fig. 4a shows a dose response curve for the dose recovery data measured using these conditions. The curve is fitted with a double saturating exponential (DSE) function and has values of $D_{0,1}$ ~43 Gy and $D_{0,2}$ ~731 Gy. The OSL data from the blue stimulation for the same aliquot are also shown (Fig. 4b) for comparison purposes, highlighting the differences between the VSL and OSL datasets; here the OSL DRC is fitted with a single saturating exponential (SSE) and has a lower D_0 value of ~28 Gy.

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4.3 Effect of test dose size

Using the measurement and analysis conditions identified in Section 4.2 as most appropriate, the effect (if any) of the test dose size on the VSL D_e values obtained using a SAR protocol was assessed. A broad range of test doses have been applied in previous papers e.g. 30 Gy (Hernandez and Mercier 2015), 50 Gy (Porat et al. 2017), 200 Gy (Jain 2009, Ankjærgaard et al. 2013) and 540 Gy (Ankjærgaard et al. 2016), but this variable has yet to be investigated within a single study. To establish the effect of test dose size on our D_e measurements, an additional dose recovery experiment was undertaken using sample Aber215/STP09. All parameters were identical to the dose recovery experiment described in Section 4.2 using Protocol A at a preheat temperature of 280 °C, except the size of the test dose was varied. In Section 4.2 the test dose was 20 Gy, but in this experiment the size of the test dose varied from 20 to 100 Gy. The given dose was 405 Gy, and the mean measured/given dose ratios (Fig. 5) obtained using a 20 Gy and 50 Gy test dose are similar (0.82 ± 0.22, and 0.82 ± 0.12 respectively) and within 10 % uncertainty of unity, however the 50 Gy data show slightly less variation in the results. Whilst the ratios obtained using the 20 Gy and 50 Gy test doses underestimate the given dose by ~20 %, the ratios from the 100 Gy test dose underestimate by ~60 %.

The form of the dose response curve shown in Fig. 4a (and used to obtain the dose recovery data shown in Fig. 5) is a double saturating exponential ($D_{0,1}$ ~43 Gy, $D_{0,2}$ ~731 Gy). Though the origin of these two exponential components is uncertain, it is

conceivable that they arise from two defects, and that these might sensitise at different rates. In that scenario one would expect that the 100 Gy test dose would more accurately track the changes in sensitivity of the 405 Gy given dose than the much smaller 20 or 50 Gy test dose; in fact Fig. 5 shows this not to be true and the dose recovery ratio is closer to unity when using those much smaller test doses (20 or 50 Gy). Furthermore, examination of the pattern of sensitivity change seen when using test doses of 20, 50 and 100 Gy shows that they are indistinguishable from one another.

This experiment (Fig. 5) implies that for the dose range of interest in this paper, a test dose of 50 Gy or less is preferable, and that applying a large test dose (100 Gy) will result in more pronounced underestimation.

5. Natural D_e measurements

Following the dose recovery experiment on the young sample Aber215/STP09, an older sample (Aber215/STP01) with a much larger D_e was selected to test the ability of the SAR protocol to measure the natural D_e. Single grain post-IR IRSL measurements on feldspars from this sample yielded a D_e value of 463 ± 25 Gy (Colarossi 2017), and when allowance is made for the difference in dose rate between potassium-rich feldspars and quartz, this yields an expected D_e for quartz of 354 ± 30 Gy. The expected D_e value of this sample is a similar size to the dose used in the dose recovery experiment reported in Section 4.2 (405 Gy). Natural D_e measurements were undertaken using Protocol A (Table 1) with a 280 °C preheat temperature and 20 Gy test dose. DRCs were constructed using the initial signal

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from the decay curve with an early background subtraction and fitted with a DSE function. Seven aliquots were used to measure the natural D_e; four of these aliquots were rejected because the recuperation was greater than 5 % of the natural signal and data from these aliquots were excluded from the subsequent dose determination. Data from accepted aliquots gave an average D_e (93.5 ± 15.9 Gy) that was only 26% of the expected D_e value.

Although Protocol A appeared to exhibit limited sensitivity change (Fig. 1c), a possible explanation of the difficulties in routinely recovering a dose (Fig. 3b and 3d) and the underestimation seen for the natural D_e of STP01 is if sensitivity change occurs between measurement of the natural signal and all other measurements which is not corrected by measuring the response to a test dose. One method for circumventing such sensitivity change is SARA (Single Aliquot Regeneration Added dose; Mejdahl and Bøtter-Jensen 1997), where prior to their measurement using SAR a range of doses are added to a series of aliquots which retain their natural signal. A range of added beta doses (20 Gy, 50 Gy and 100 Gy) were applied to at least three aliquots per dose; results are shown in Fig. 6. One anomalously large D_e value (1038 ± 292 Gy) was measured for an aliquot in the 20 Gy added dose data set, and was excluded from analysis. The remaining data are scattered, and yield a D_e of 86.6 ± 28.4 Gy. This is smaller than the value obtained using SAR with no added dose, discussed above, and still significantly underestimates the expected D_e for this sample.

6. Summary and Conclusions

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A recent paper by Ankjærgaard et al. (2016) showed that the natural VSL signal (L_n) in quartz grows in the natural environment over the range from 200 – 2000 Gy, and using a modified multiple aliquot additive dose method they obtained ages in agreement with independent age control up to 600 ka, beyond the limit of quartz blue stimulated OSL dating. In the present paper, two SAR protocols were tested, to develop methods that could be applied where multiple aliquot methods would not be appropriate (e.g. inhomogeneous bleaching). Protocol A (based on Hernandez and Mercier, 2015), appears to be more appropriate than Protocol B (based on Ankjærgaard et al., 2015) as the former exhibits less carry over of charge from the measurement of the regeneration dose (L_x) to the measurement of the test dose (T_x) (Fig. 1a-b), has much lower sensitivity change (Fig. 1c-d), and has greater similarity between the shape of the decay curve for L_n and that for all laboratory regenerative dose signals (Fig. 2).

However, dose recovery data obtained using Protocol A are poor. If the definition of success for a dose recovery test is for data to be within the range 0.9 to 1.1, then although some of the data (Fig. 3) do pass within uncertainties, the mean value for this ratio at various preheat temperatures is commonly 0.8 or less. The reason for this poor behaviour is not clear, but it does not appear to be related to sensitivity change. Measurement of the natural D_e from a much older sample, yielded a SAR D_e that was 74 % lower than expected, and measurement using a SARA approach failed to improve this.

Dose underestimation using SAR protocols with the VSL signal have been noted previously. For example Ankjærgaard et al. (2015) reported age underestimates of 50 % or more for sediments in the age range 1.6 to 0.7 Ma. In the present study, it is not clear whether the difficulties encountered in the dose recovery experiment are the same as those underlying the underestimation observed in the natural D_e, or if more than one problem remains to be solved. Further investigation is required to understand the mechanism(s) causing such problems. Nevertheless, the growth of the VSL signal in nature remains an enticing prospect for dating.

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Table 1: The two SAR measurement protocols tested in this study. Protocol A was selected for further measurements (see text for detail). Differences between the two protocols are highlighted in bold.

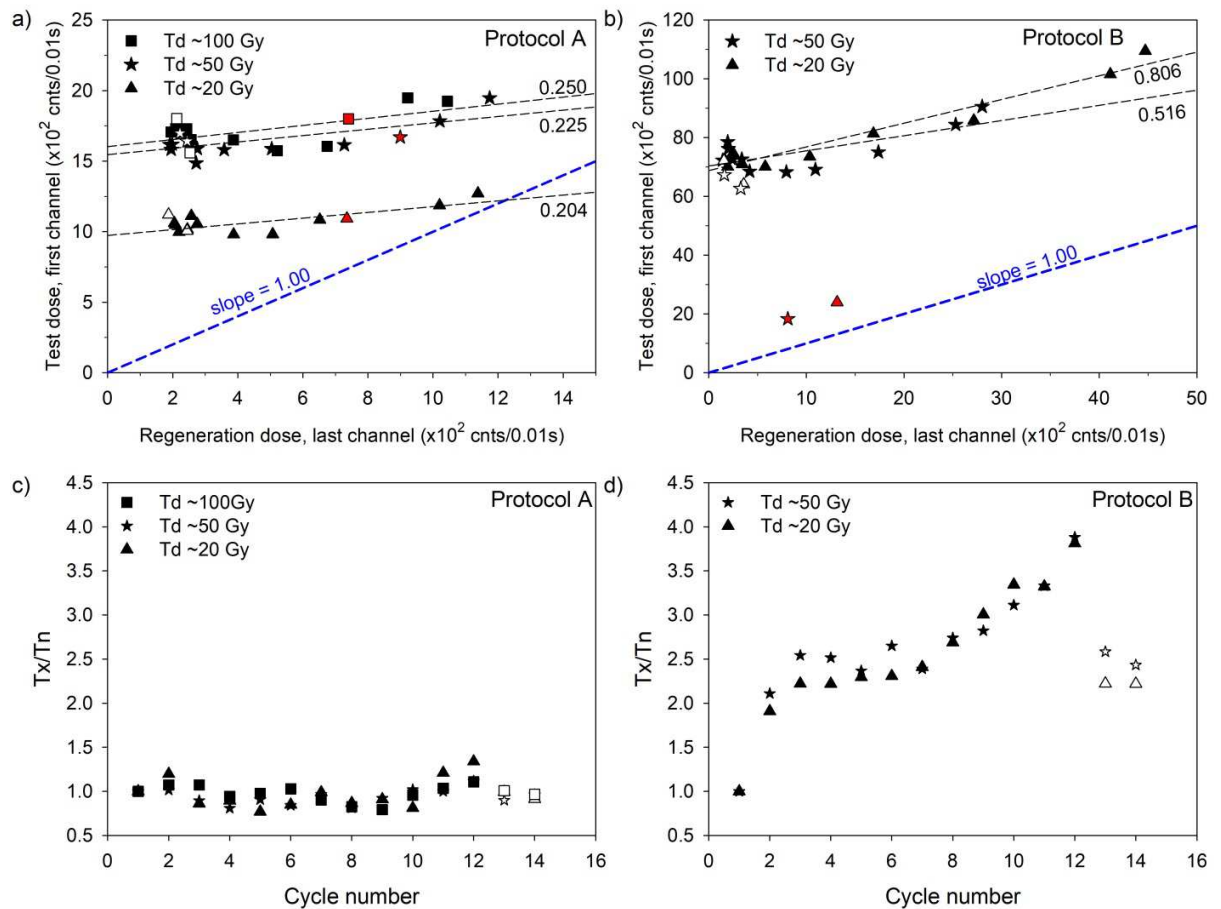| Step	Protocol A		Protocol B	
1	Regeneration Dose		Regeneration Dose	
2	Preheat at 280 °C for 10 s		Preheat at 280 °C for 10 s	
3	BSL at 125 °C for 40 s		BSL at 125 °C for 40 s	
4	VSL at 30 °C for 500 s	L_x	VSL at 30 °C for 500 s	L_x
5	**VSL at 200 °C for 500 s**		Test dose	
6	Test dose		Preheat at 280 °C for 10 s	
7	Preheat at 280 °C for 10 s		BSL at 125 °C for 40 s	
8	BSL at 125 °C for 40 s		VSL at 30 °C for 500 s	T_x
9	VSL at 30 °C for 500 s	T_x	**VSL at 380 °C for 200 s**	
10	**VSL at 200 °C for 500 s**		Return to step 1	
11	Return to step 1			

Figure 1: Assessing the amount of signal carried over from the regeneration dose measurements (L_x) into the test dose measurements (T_x) from a) Protocol A and b) Protocol B. Data from the first channel of T_x are plotted against the last channel of the preceding L_x measurement. Red-filled symbols show the data from the first cycle (i.e. the natural measurements) and open symbols from repeated regeneration doses. Values are shown for the slope of each dashed line, constructed using a linear regression function. Also shown is the change in sensitivity during the SAR cycle for (c) Protocol A and (d) Protocol B.
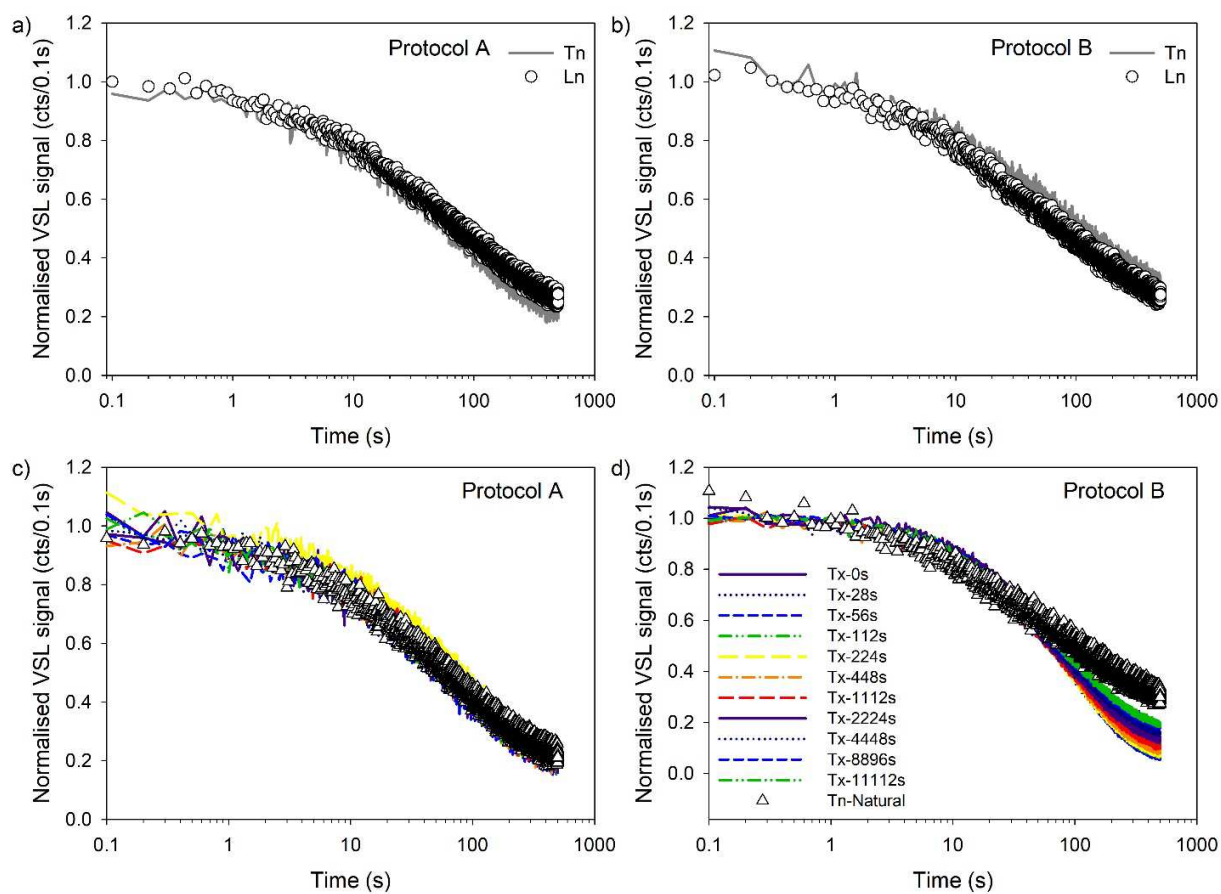
Figure 2: Comparison of normalised decay curves for (a-b) L_n vs T_n and (c-d) T_n vs T_x for SAR Protocols A and B, as outlined in Table 1, using a test dose of 50 Gy.
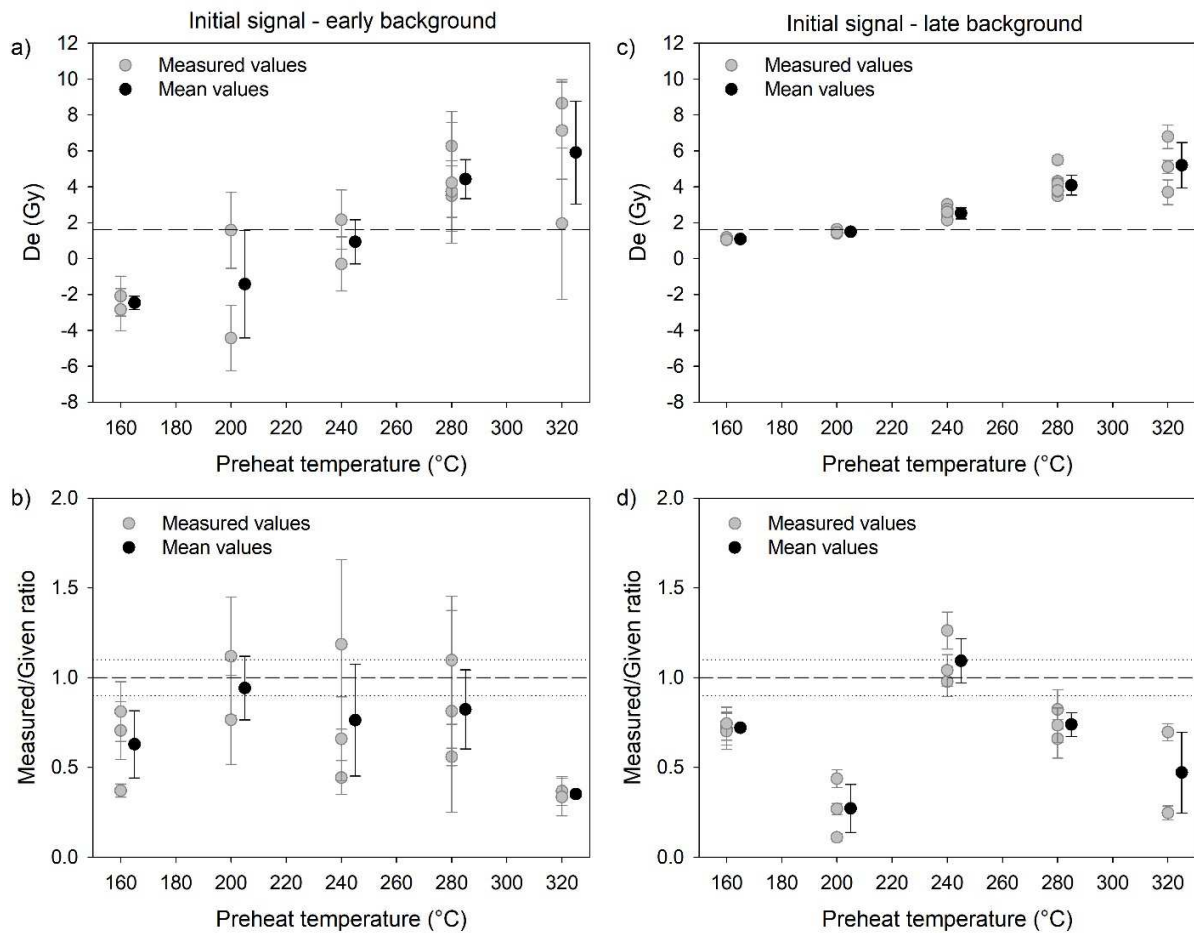
Figure 3: a) Natural D_e measurements for a young sample (Aber215/STP09) using Protocol A and various preheat temperatures. The expected D_e for the sample is denoted by the dashed line. b) Dose recovery experiment for the same sample after adding a laboratory beta dose (405 Gy) onto the natural D_e. Data points in grey represent aliquot-specific values, and the mean ± standard deviation is shown in black offset by 5 °C. c-d) The data from a) and b) reanalysed using a late background subtraction for comparison.
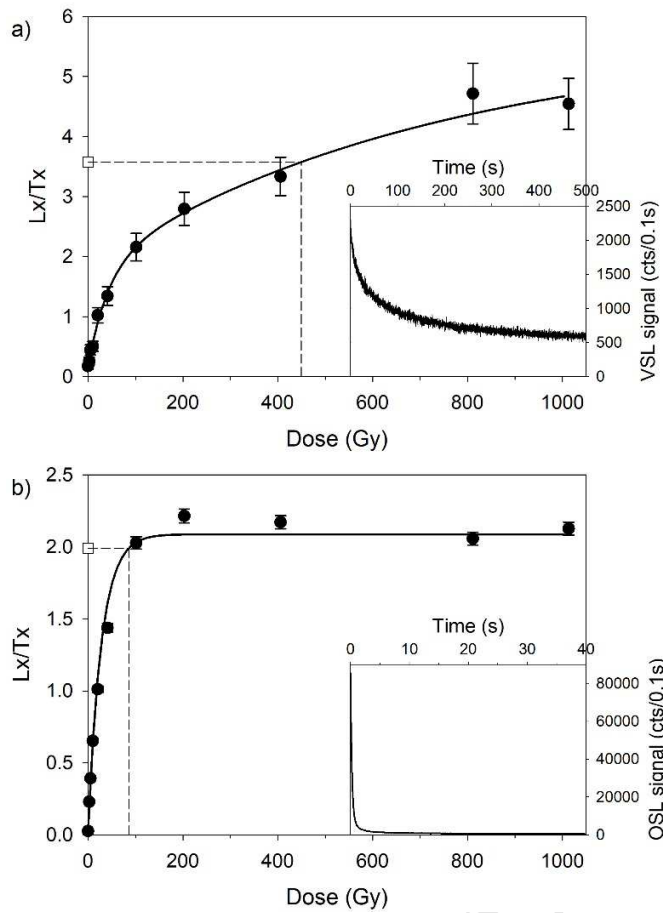
Figure 4: a) Example dose response curve for one aliquot of Aber215/STP09 from the dose recovery experiment where a laboratory beta dose of 405 Gy was added onto the natural D_e prior to measurement and a 280 °C preheat was used. The DRC is fitted with a DSE function. Inset: The measured VSL decay curve for the laboratory given dose (L_n). b) Data from the blue stimulation prior to the VSL measurement for the same aliquot. The DRC is fitted with a SSE. Inset: The measured OSL decay curve for the laboratory given dose (L_n).
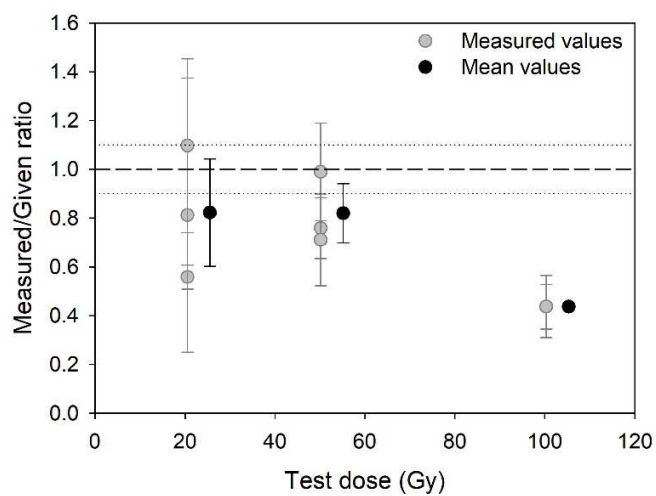
Figure 5: Dose recovery experiment using Protocol A with a preheat of 280 °C. A known beta dose of 405 Gy was added to the natural signal and the size of the test dose was varied. Measured/given dose ratios calculated are shown in grey and the mean ratio for each test dose size in black (offset by 5 Gy).
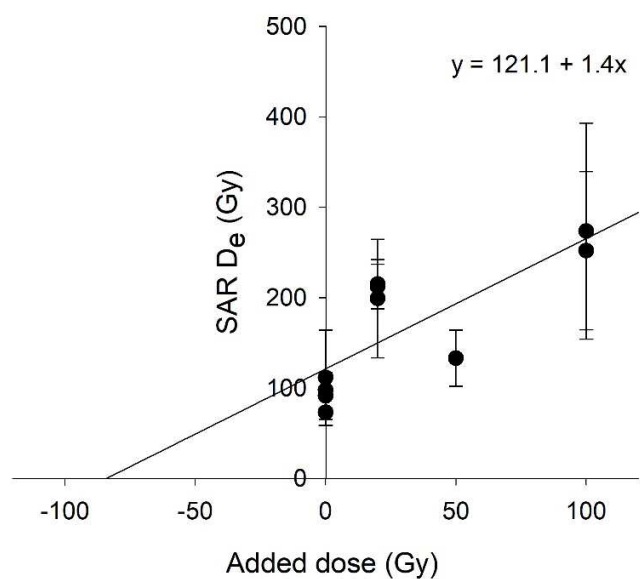
Figure 6: Natural D_e measurements using SAR Protocol A on an old sample Aber215/STP01. A laboratory beta dose between 0 Gy and 100 Gy was added to the natural D_e prior to measurement.