The effect of residual signal on dose measurements using MET-pIRIR signals from K-feldspar

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The effect of residual signal on dose measurements using MET-pIRIR signals from K-feldspar

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Abstract

Overestimation in dose recovery results was observed at high doses (> 1000 Gy) when a single-aliquot regenerative-dose (SAR) procedure is applied to the multiple-elevated-temperature post-IR IRSL (MET-pIRIR) signals from potassium-rich feldspar (K-feldspar) grains from the Nihewan Basin. Such overestimation results from the non-bleachable (residual) signals that were not appropriately corrected for. A signal-subtraction method was proposed to solve this problem, which involves building growth curves for the bleachable and non-bleachable signals. Here we modelled the effect of residual signal on equivalent dose ($D_e$) and found that even a small amount of residual signal may give rise to large overestimation at high doses. Our results highlight that residual signals may not only affect young samples but may lead to erroneous results for old samples. Our study also suggests that although residual signals may result in significant error in the dose recovery, they may have a very small effect on natural samples because their residual signals seem to be relatively small.

Keywords: K-feldspar, MET-pIRIR, dose recovery, signal-subtraction, residual signal

1 Introduction

Quartz has been widely used in optically stimulated luminescence (OSL) dating in the study of the Quaternary period as a sensitive and reliable natural dosimeter (e.g., Aitken, 1998; Wintle, 2008). Compared with quartz, the infrared stimulated luminescence (IRSL) signals from potassium-rich feldspar (K-feldspar) saturate at a much higher radiation dose (e.g. Li et al., 1997; Aitken, 1998; Roberts et al., 2015), but the possible presence of anomalous fading has limited its wider application (Spooner, 1994; Wintle, 1973). By applying an IR bleaching at low temperature (50°C) before measuring the IRSL at a high-temperature (>200°C), it is possible to preferentially probe traps that suffer much less or no fading (Thomsen et al., 2008; Buylaert et al., 2009; Thiel et al., 2011). Alternatively, by progressively increasing the stimulation temperature from 50 to 300°C on the
measurement of IRSL signal, the multiple elevated temperatures post-infrared IRSL (MET-pIRIR) (Li et al., 2013a; Li and Li, 2011, 2012; Li et al., 2014b) can isolate non-fading IRSL signals in feldspars and extend the dating range to the early middle Pleistocene or possibly the late early Pleistocene (e.g., Chen et al., 2015; Guo et al., 2015; Li et al., 2014b).

Although the pIRIR signals measured at elevated temperatures have significantly reduced rates of fading compared to the IRSL signal measured at low temperatures, they have also been found to be more difficult to bleach (Li et al., 2014a; Li and Li, 2011; Li et al., 2013b; Thomsen et al., 2008). Even after a prolonged bleaching period, there is still a non-bleachable (or residual) component left for the pIRIR signals. Based on published residual dose for feldspar samples, the majority of the samples studied have residual doses lower than ~60 Gy (Buylaert et al., 2012; Buylaert et al., 2011; Fu et al., 2012; Guo et al., 2016; Li et al., 2014a; Li and Li, 2011; Thomsen et al., 2008), suggesting that it is important to consider the residual doses for relatively young samples (e.g., equivalent dose ($D_e$) less than 500 Gy). For young samples, the most straightforward correction method is to measure residual doses using sunlight-bleached samples or modern analogue samples, and subtract the residual doses from the corresponding $D_e$ values (Fu et al., 2012; Li and Li, 2011; Lowick et al., 2012). Some studies also indicate that the non-bleachable signal could have been better reset in natural processes than in the laboratory conditions (i.e., solar simulator) (e.g., Thiel et al., 2011; Sohbati et al., 2012; Kars et al., 2014a). However, the signal used for building the dose response curve (DRC) include dose-dependent bleachable and non-bleachable signals. Hence, using the simple subtraction of the residual dose may give rise to $D_e$ underestimation for young samples (Li et al., 2013b). To overcome this problem, Li et al. (2013b) suggested to apply an ‘intensity-subtraction’ method, involving the construction of a DRC for the bleachable regenerative signal and then interpolation of the bleachable natural signal. In addition to the correction methods, using lower preheat and pIRIR stimulation temperatures can also minimise the residual dose problem for young samples (Madsen et al., 2011; Reimann et al., 2011). However, as fading correction (g-value) becomes unreliable in the non-linear part of the growth curve (Huntley and Lian, 2006; Li and Li, 2008), it is necessary to apply high pIRIR stimulation temperature (≥ 250°C) for isolating a non-fading component of old samples (Li and Li, 2011, 2012).

Previous studies demonstrated the importance of correcting residual doses or signals for young samples (Li et al., 2014a), simply because the residual dose is relatively large compared to the natural $D_e$. In this study, based on dose recovery tests, we demonstrated that residual signals may also result in significant effect for dating old samples.

2 Samples and facilities

Nihewan Basin is about 150 km west of Beijing in northern China (Fig. 1a). Five fluvial-lacustrine samples (DDP-3, HSP-1, HY-1, TEG-1 and ZJP-4) from different locations of the Nihewan Basin,
(Fig. 1b) were used to establish standardised growth curves (SGCs) for K-feldspar (Li et al., 2015a; Li et al., 2015b). In addition to those, three loess samples (YJG-01, YJG-08 and DDP-7), one fluvial sample (HTL-Loc1-2) and one fluvial-lacustrine sample (HY-3) were also examined to study their residual signals. The deposit type, grain size, and $D_c$ ranges for each sample are summarised in Table 1.

Samples were collected either in stainless steel tubes or as blocks. After the tubes or blocks were removed, they were immediately wrapped in light-proof plastic and transported to the Luminescence Dating Laboratory at the University of Wollongong for analysis. K-feldspar grains of 90–125 or 125–180 µm in diameter were extracted from the samples following standard mineral separation techniques (Aitken, 1998, see details in Rui et al., submitted). K-feldspar grains covered the central ~5 mm diameter portion of stainless-steel discs were used for the IRSL measurement. The measurement was performed on Risø TL/OSL-DA-15 and TL/OSL-DA-20 readers equipped with $^{90}\text{Sr}/^{90}\text{Y}$ beta sources and infrared diodes for stimulation (870 nm). IRSL signals were detected by an Electron Tubes 9235B photo-multiplier tube fitted with Schott BG-39 and Corning 7-59 filters. A solar simulator (Dr Hönle UVACUBE 400) was used for bleaching in relevant experiments.

### 3 Results

In this study, $D_c$ estimates were determined based on SGCs using a single- aliquot regenerative-dose (SAR) MET-pIRIR procedure (Table S1 and Group A in Fig. 2) of Li et al. (2014b). To reset the pre-dose ‘memory’ from the preceding cycle, the aliquots were bleached for ~4 h in the solar simulator at the end of each SAR cycle (Li et al., 2014b). For the MET-pIRIR measurements, infrared stimulation was performed successively at six temperatures (50, 100, 150, 200, 250 and 290°C) for 100 s. The net pIRIR signals were calculated as the sum of counts in the first 10 s of pIRIR decay minus a ‘late light’ background estimated from the mean count rate over the final 10 s. A series of regenerative doses, ranging from 0 to ~2400 Gy, were given to each aliquot and the corresponding MET-pIRIR signals were measured. We first established growth curves of sensitivity-corrected signals ($L_x/T_x$) using different aliquots from different samples. As shown in Fig. 3a–f, there are large between-aliquot variations in the growth curve data. To reduce the scatter, the least-squares normalisation (LS-normalisation) method (Li et al., 2016) was applied by using the built-in function `lsNORM()` provided in the R-package ‘numOSL’ (Peng et al., 2013; Peng and Li, 2017). In the function `lsNORM()`, a general order kinetic (GOK) function (Guralnik et al., 2015) in the form $f(x) = a[1 - (1 + b/cx)^{-(c/d)}] + d$ is used for fitting, where $x$ is the dose and the parameters $a$, $b$, $c$ and $d$ are constants. All SAR SGCs were then normalised using the signal corresponding to a regenerative dose of 373 Gy. It can be seen that the between-aliquot variation was significantly reduced after LS-normalisation (Fig. 3g–l), indicating that different samples and aliquots share the same growth curve. This observation is consistent with those reported in previous studies (Jacobs et al., 2019; Li et al.,...
2015b). The parameters $a$, $b$, $c$ and $d$ for the best-fit function at 290°C stimulation temperature (Fig. 3l) are 1.70, 0.003, 1.20 and 0.13, respectively. This SGC was subsequently used for $D_e$ estimation for all the studied samples.

3.1 Dose recovery and residual tests

3.1.1 Dose recovery and residual details and results

Dose recovery and residual tests were conducted using natural aliquots bleached by solar simulator. We first studied the bleachability of the MET-pIRIR signals for our samples. Three groups of natural aliquots from DDP-3 were prepared and each group consists of 4 aliquots. Different groups were bleached using the solar simulator for 4 h, 8 h and 16 h, respectively. For each aliquot, the natural signal ($L_n$) and the corresponding test dose signal ($T_n$) were measured and then bleached for 4 h in the solar simulator. After that a regenerative dose of 373 Gy and a corresponding test dose were given and measured (Table 2). This regenerative dose signal ($L_r/T_r$) was used to normalise the ‘natural’ signal, so that the re-normalised signal ($\frac{L_n/T_n}{L_r/T_r}$) can be projected directly onto the corresponding SGC to estimate equivalent doses, as the SGCs were also normalised to unity at 373 Gy.

The average of the re-normalised ratios relative to the natural signal values (no bleaching) are shown in Fig. 4. It shows that after 16 h bleaching, only 0.8% of the initial signal was left for the IRSL at 50°C, while 8.7% was remaining for the signal measured at 290°C. As the remaining signal determined after 8 h bleaching (10.9 ± 1.5 % at 1σ) is consistent at 2σ with that obtained after 16 h bleaching (8.7 ± 0.5 % at 1σ) at 290°C, a 8 h bleaching was applied to all our samples for the recovery and residual signal measurement.

Dose recovery tests were conducted on samples DDP-3 and HSP-1, with natural dose of 1048 and 1478 Gy, respectively. Four aliquots of DDP-3 and three aliquots of HSP-1 were first bleached for 8 h using a solar simulator and then given doses of 1000 and 1800 Gy as surrogate ‘natural’ dose, respectively. The $L_n/T_n$ value of the recovery signal and the $L_r/T_r$ value of the regenerative dose of 373 Gy were also determined using the MET-pIRIR procedure. The averages of the re-normalised ratios were projected onto the SAR SGCs (Fig. 3g–3l) to estimate the recovered $D_e$ values. Fig. 5 shows the temperature dependence of the recovered $D_e$ (open square). For both samples, the recovered $D_e$ of the 50°C IRSL are smallest and an increasing trend with MET-pIRIR signals at elevated temperatures was observed. For the recovered result of DDP-3, the measured doses at higher temperatures (>100°C) are statistically consistent with the given dose at 2σ. However, for the sample HSP-1 (given dose = 1800 Gy), significant overestimation was obtained for the MET-pIRIR signals at 250°C and above. The 250°C MET-pIRIR signal yielded a $D_e$ value of 2808 ± 105 Gy while no finite result can be obtained for the MET-pIRIR$_{290}$ signal because its signal is higher than the maximum signal level of the corresponding SGC.
The residual signals of samples DDP-3 and HSP-1 after 8 h bleaching were also measured using the protocol mentioned above. The averages of the re-normalised residual signals were projected on to the SAR SGCs (Fig. 3g–3l) to estimate the residual doses, respectively (Fig. 5 filled circle). The residual doses of DDP-3 and HSP-1 at the 290°C stimulation temperature are 25.6 ± 4.8 and 23.0 ± 3.0 Gy, respectively. Such residual doses are substantially smaller than the dose recovery results, suggesting that the overestimation in dose recovery at high stimulation temperatures for sample HSP-1 cannot be corrected by subtracting the residual dose from the \( D_e \) value.

### 3.1.2 Effect of residual signal

As reported by Li et al. (2013b), both the natural and regenerative signals may contain bleachable and non-bleachable signals, which are expected to be originated from different traps. Since the residual dose is obtained by interpolating the non-bleachable natural signal onto the DRC which consists of both bleachable and non-bleachable signals, this may result in unreliable \( D_e \) estimation for young samples if the residual dose is simply subtracted from the \( D_e \) value. In order to avoid this problem, Li et al. (2013b) proposed a signal subtraction method, in which the residual signals are measured for both natural and regenerative doses to estimate their corresponding bleachable signals. \( D_e \) evaluation can then be achieved by interpolating the bleachable signal onto the dose response curve of the bleachable signal.

To measure the unbleachable signals from regenerative doses, a relative young sample (HTL-Loc1-2, \( D_e = 35.3 ± 3.2 \) Gy) was selected and bleached by 290°C IR stimulation to remove their natural signals. Following the IR bleaching, a total of 15 aliquots divided into 5 groups were given different regenerative doses, ranging from 0 to 2000 Gy. They were then bleached for 8 h using solar simulator to remove all the bleachable signal and measured using the MET-pIRIR procedure in Table 2 (see also Fig. 2 Group B). For each aliquot, the residual signal and the corresponding test dose signal were measured and then a regenerative dose (\( D_r = 373 \) Gy) was applied and measured to normalise the between-aliquot variation. The re-normalised residual signals are shown in Fig. 3m–3r (red dots). The residual signals show a clear dose dependency for higher-temperature stimulation (e.g., 250 and 290°C). These residual signals were subtracted from the total signals (Fig. 3m–3r, black dashed lines) to calculate the bleachable signals (red dashed lines). Fig. 3m shows that the non-bleachable (residual) component is a small fraction of the IRSL measured at 50°C (0.5% at 1500 Gy) and that the proportion of the non-bleachable component increases with the stimulation temperature, accounting for ~8.5% (at 1500 Gy) of the total MET-pIRIR signal at 290°C (Fig. 3r).

To estimate the bleachable signals for the ‘natural’ aliquots, four discs of DDP-3 and HSP-1 were bleached for 8 h by solar simulator and then given a dose of 1000 and 1800 Gy, respectively. These discs were then bleached ~8 h again to remove all bleachable signals and measured to get the re-normalised \( L_n/T_n \) ratios. This ‘natural’ residual signal was then subtracted from the ‘natural’ signal of the aliquot used for dose recovery tests to estimate the bleachable signal generated by the given dose.
Fig. 6a and 6b show the temperature dependence of the total signal (red filled square), the residual 
signal (red filled circle), and the corresponding bleachable signal (residual-signal-subtracted signal, 
red open square) obtained using the signal-subtraction method for DDP-3 and HSP-1, respectively. 
The residual-corrected bleachable signal is consistent with the expected signal (black open square) at 
250 and 290°C at 2σ; the latter was obtained by interpolating the corresponding given doses on to the 
bleachable SGCs. For the signals at lower temperatures (50–150°C), however, the measured 
bleachable signals are still lower than the corresponding expected signal for DDP-3, which can be 
attributed to the initial sensitivity changes that were not appropriately corrected for using the test dose 
signals (see section 3.1.4). This result suggests that the ‘natural’ signal intensity at high temperature 
can be recovered successfully after applying the signal-subtraction method to correct for residual 
signals. It also confirms that the overestimation in the dose recovery is caused by the high residual 
signals after solar simulator bleaching.

3.1.3 Effect of charge carry-over

Previous studies suggested that the failures of dose recovery could also be attributed to the charge 
carry-over effect from the natural/regenerative doses to the subsequent test doses, which can be 
reduced by applying a large test dose thereby decreasing the relative size of the charge carried over 
(Nian et al., 2016; Yi et al., 2016; Colarossi et al., 2018). To test this effect for our samples, we have 
conducted a simplified dose recovery test using a larger test dose (200 Gy). Three aliquots of HY-1 
and TEG-1 were firstly bleached by the solar simulator for 8 h to erase their natural signals. They 
were then subjected to two repeated $L_x/T_x$ measurements with the same regenerative dose (1800 Gy 
for HY-1 and 2200 Gy for TEG-1). We then compare the $L_x/T_x$ values obtained from the two cycles 
(Fig. 7); a successful dose recovery should yield indistinguishable $L_x/T_x$ values for the two cycles. As 
shown in Fig. 7, the sensitivity-corrected signals from the first cycle ($L_1/T_1$) are systematically higher 
than those from the second cycle ($L_2/T_2$), implying a failure in dose recovery. We, therefore, 
concluded that the charge carry-over effect is not the main contribution of the failure of the dose 
recovery for our samples.

3.1.4 Effect of initial sensitivity change

Another reason for the failure in dose recovery has been attributed to the larger sensitivity change 
that occurs during the measurement of $L_n$, which results in a significant difference between the 
luminescence efficiency of the natural dose (or given dose) and the subsequent test dose ($T_n$) (e.g., 
Wallinga et al., 2000; Chen et al., 2013; Li et al., 2013a; Kars et al., 2014b; Zhang, 2018; Qin et al., 
2018).

To study this effect for our samples, two groups of natural aliquots from sample HY-1 were 
bleached by solar simulator for 8 h to erase the natural signal. Three repeated $L_x/T_x$ measurements 
with a fixed regenerative dose (1000 Gy) and test dose (60 Gy) were performed on one group (Group 
A). For the other group (Group B), after being given the regenerative doses, the aliquots were
bleached using solar simulator for 8 h before \( L_x/T_x \) measurements to assess the corresponding residual signal. In order to allow for a comparison between the two groups, following the repeated \( L_x/T_x \) measurements, a regenerative dose of 373 Gy and the corresponding test dose were given and measured \( (L_x/T_x) \) for each group. All the \( L_x/T_x \) values were normalised by the corresponding \( L_x/T_x \) values and plotted with the repeating cycles (Fig. 8). The bleachable component for each cycle (Fig. 8, black empty square) can be calculated by subtracting the re-normalised residual signal (Group B) (Fig. 8, red filled circle) from the re-normalised total regenerative signal (Group A) (Fig. 8, black filled square). For the signal of the MET-pIRIR290 (Fig. 8b), the re-normalised total \( L_x/T_x \) value of the second cycle was smaller than the first cycle, indicating the sensitivity may change between the first and second cycle. However, after correcting the residual signals, the bleachable \( L_x/T_x \) value remains unchanged for each cycle, indicating that the sensitivity was stable for the repeated measurement. Therefore, we conclude that the failure of the dose recovery test for the pIRIR290 signals is mainly due to the effect of the residual signal rather than the initial sensitivity changes. For the IRSL50, however, whose residual signal is negligible (Fig. 8a), the re-normalised \( L_x/T_x \) values of the first cycle were significantly smaller than the second for both the total and the bleachable signal; the latter two are indistinguishable from each other. This supports that the low-temperature IRSL suffers from the initial sensitivity change, resulting in underestimation in \( D_e \), as has already been demonstrated in previous studies (Li and Li, 2011; Li et al., 2017).

3.2 Simulating the effect of residual signal

Based on the dose recovery tests above, we show that the residual signal may cause significant problems for old samples with doses in the non-linear range. Supposing that the natural sample has a residual signal prior to burial and the bleachable and residual signals follow a single saturating exponential function, the natural signal of the sample can be expressed as

\[
L_N = A \left( 1 - e^{-\frac{D}{D_0}} \right) + B \left( 1 - e^{-\frac{\Delta D + D}{D_R}} \right) \tag{1}
\]

where \( A \) and \( D_0 \) are the maximum intensity and the characteristic saturating dose of the bleachable signal, respectively, \( D \) is the irradiation dose, \( B \) and \( D_R \) are the maximum intensity and the characteristic saturating dose of the residual signal, respectively, and \( \Delta D \) is the residual dose prior to burial. The first term on the right-hand side of Eq. (1) is the bleachable signal intensity \( (L_N\text{-bleachable in Fig. 2}) \) and the second term is the hard-to-bleach or residual signal intensity \( (L_N\text{-residual }+ \text{L}_{RN} \text{ in Fig. 2}) \). It then follows that the residual signal prior to burial is

\[
L_{RN} = B \left( 1 - e^{-\frac{\Delta D}{D_R}} \right) \tag{2}
\]

For the regenerative doses, since the residual signal is expected to be fully reset by IR stimulation during SAR measurements, the regenerative signal \( (L_R) \) can be written as
Because $D_e$ is estimated by comparing $L_N$ and $L_R$, correct results can only be obtained when $\Delta D$ is equal to zero, i.e., there was no residual signal prior to burial.

In order to test the effect of the residual signal on the accuracy of $D_e$, we modelled the $D_e$ estimates for different scenarios with different magnitudes in $L_{RN}$, $B$, and $D_R$. To determine how these parameters might affect the accuracy of $D_e$ estimation, we modelled a range of samples with paleodoses ($P$) ranging from 0 to 2000 Gy. We first used a single saturating exponential function to fit the experimentally obtained dose response curve data from the bleachable signal (red dashed curve in Fig. 3r) and the residual signal (red solid curve in Fig. 3r) at 290°C, which yielded estimates of the parameters $A$ (1.28), $D_0$ (344 Gy), $B$ (0.21) and $D_R$ (4682 Gy).

To study the effect of $B$ (maximum residual signal), we changed the $B$ value relative to different fractions of $A$ (i.e., 0.04A, 0.08A, 0.12A and 0.16A) and fixed the values of $A$, $D_0$ and $D_R$. For each of the assumed $B$ values, we modelled five scenarios, in which the samples have different residual signals before burial (i.e., $L_{RN}$ is set at 0, 0.2B, 0.4B, 0.6B and 0.8B, respectively). The natural signal $L_N$ can be calculated for each $P$ value based on Eq. (1). By setting $L_N = L_R$, the corresponding $D_e$ can be calculated based on Eq. (3).

Fig. 9 shows modelled $D_e/P$ ratios calculated using different combinations of $B$ and $L_{RN}$. As expected, $D_e$ values obtained for the case of $L_{RN} = 0$ are consistent with $P$. However, the $D_e$ values are systematically overestimated when there is a residual signal prior to burial ($L_{RN} > 0$). Such an effect is especially prominent in the very small dose range, as well as in the high dose range. For $B = 0.04 * A$ (i.e., the maximum residual signal is only 4% of that of bleachable signal), an overestimation of 59% is obtained for $P = 20$ Gy ($L_{RN} = 0.8 * B$). The extent of overestimation decreases with dose, so that the $D_e$ values are very close to $P$ between 160 and 700 Gy, with overestimation generally less than 10% (Fig. 9a). However, the extent of overestimation increases beyond ~700 Gy and a significant overestimation of 140% is obtained for $P = 1500$ Gy for the case $L_{RN} = 0.8 * B$. Such an effect is even more severe for relatively larger maximum residual intensities ($B$) (Fig. 9b–d).

We also studied the effect of $D_R$ by fixing the value of $A$, $D_0$ and $B$ and assuming different values of $D_R$ (i.e., 1000, 2000, 3000 and 4000 Gy). For each $D_R$ value, the $L_{RN}$ value is also assumed to be different proportions of $B$ (i.e., 0, 0.2B, 0.4B, 0.6B and 0.8B). Fig. 10 shows the $D_e$ values calculated using different combinations of $D_R$ and $L_{RN}$. A similar pattern to that of Fig. 9 is observed. It shows that in the case of significant residual signal prior to burial, there would be a significant overestimation of $D_e$ estimation. Such an effect would be increased when the residual signal has a higher characteristic saturation dose ($D_R$).
If the dose-subtraction method is applied for $D_e$ correction, the residual of the natural signal is $L'_{RN} = B \left( 1 - e^{-\frac{\Delta D + D}{D_R}} \right)$ and the residual dose can be calculated by setting $L'_{RN} = L_R$. Residual-dose-corrected $D_e/P$ ratios are shown in Fig. S1 and S2. The dose-subtraction correction appears to yield reliable results for young samples only when the maximum intensity of the residual signal ($B$) is small (Fig. S1a). However, considerable overestimation can still be obtained in the high dose range (Fig. S1 and S2). For $B = 0.04 \times A$, the corrected $D_e$ values are consistent with $P$ up to 700 Gy, while an overestimation of 139% is still obtained for $P = 1500$ Gy (Fig. S1a).

### 3.3 Effect of residual signal on $D_e$ estimation

As demonstrated in the previous section, the amount of residual signal prior to burial plays an important role in $D_e$ estimation. One of the critical requirements for dating older samples is that there should be negligible residual signal prior to burial. One of the commonly adopted methods to estimate residual signal is to bleach the natural samples in the laboratory using either sunlight or a solar simulator (e.g., Li et al., 2014a). However, previous studies have suggested that the residual signal observed by bleaching the natural samples may not represent the true residual signal prior to burial, based on the observation that there is a positive correlation between residual dose and $D_e$ (e.g., Buylaert et al., 2012; Lauer et al., 2017; Schatz et al., 2012; Sohbati et al., 2012). This suggested that there might be negligible residual for natural samples. To further investigate this issue for samples from our study area, six samples (YJG-01, YJG-08, DDP-7, HTL-Loc1-2, HY-3, TEG-1) with a range of natural doses between ~5 Gy and ~1478 Gy, were selected to test their natural residual signal.

Three to four aliquots of each natural sample were bleached for 8 h by solar simulator. After bleaching, we estimated the residual signal using the protocol mentioned above. Fig. 11a shows the residual doses (based on total SGCs) for different samples as a function of IR stimulation temperatures. For all samples, the smallest residual doses were obtained at an IR stimulation temperature of 50°C (2–6 Gy), and the size of the residual doses increase with an increase in stimulation temperature (e.g., the residual doses range between ~9 and 44 Gy for the MET-pIRIR290).

The residual signals of the MET-pIRIR290 signal are plotted against the corresponding natural signals of the samples in Fig. 11b. A positive correlation is observed between the residual signal and the natural signal; the latter is a reflection of natural dose. The youngest sample YJG-01 ($L_n/T_n = 0.16 \pm 0.02$) has the smallest residual signal (0.06 ± 0.01), and the oldest sample TEG-1 ($L_n/T_n = 1.46 \pm 0.05$) has the largest residual signal (0.33 ± 0.03), indicating that the residual signal increases with the natural doses.

To further test whether the residual signal is dose dependent (i.e., growing with dose), the two youngest samples (YJG-01 and YJG-08) were studied. For each sample, two groups of natural aliquots were given a large dose (1000 Gy) on top of their natural doses (~5 and ~36 Gy for YJG-01 and YJG-08, respectively). One group (Group A) was then measured to get the re-normalised $L_n/T_n$. 
The other group (Group B) was bleached by solar simulator for 8 h to estimate their residual signal. A significant increase in the residual signal can be observed for both samples after giving 1000 Gy (Fig. 11b). This result further supports that the residual signal is dose-dependent. Therefore, the fact that young samples have relative lower residual signals indicates that the residual signal observed in the laboratory is actually bleachable in nature.

To further test the effect of residual signal on $D_e$ determination, the $D_e$ of Group A samples (natural plus laboratory doses) were estimated by projecting their re-normalised ‘natural’ signals onto the corresponding SGCs. For sample YJG-01 (whose $D_e$ is around 5 Gy), the expected $D_e$ of natural dose plus 1000 Gy is 1005 Gy. A $D_e$ value of $1128 \pm 75$ Gy was obtained for the 290°C MET-pIRIR signal, which is consistent with the expected value (1005 Gy) at 2σ. For sample YJG-08 (whose $D_e$ is ~36 Gy), the expected dose of Group A is 1036 Gy. A $D_e$ of $1809 \pm 158$ Gy was obtained, which is significantly overestimated by ~70%. However, if the signal-subtraction method is used to correct for the residual signal, a residual-corrected $D_e$ value of $1116 \pm 98$ Gy is obtained, which is in good agreement with the estimated dose (1036 Gy). The different behaviours between YJG-01 and YJG-08 can thus be explained by their different residual signals prior to burial. The overestimation of YJG-08 is caused by its relatively large residual signal, as expected by our modelling results (Figs. 9 and 10).

4 Discussion

Our study shows that the hard-to-bleach pIRIR signal is dose dependent. For natural samples, the traps associated with the hard-to-bleach signal are expected to have reached saturation due to the long geological history of the grains, as the sunlight may not fully reset the non-bleachable signal. Our simulation shows that the presence of the residual signal prior to burial may lead to $D_e$ overestimation for old samples (Figs. 9 and 10). This is experimentally confirmed by our dose recovery test, which demonstrated that the dose recovery ratio could be significantly overestimated if there is a relatively large residual signal. Failed dose recovery tests on old samples were also reported in previous studies. In the study of samples from the Carpathian Basin (Stevens et al., 2011), significant overestimation in dose recovery ratios (1.37 ± 0.05 and 1.42 ± 0.07) of the 290°C pIRIR signal were obtained when the given dose was 1000 Gy, while they were consistent with unity at 2σ for smaller given doses (<150 Gy). Dose recovery ratios ranging from 1.20 ± 0.11 to 1.60 ± 0.12 with given doses between 200 and 800 Gy were also reported by Li et al. (2018) for their samples from central Europe using the 290°C pIRIR signal. All the above studies suggested that such an overestimation cannot be solved by applying a simple dose-subtraction method. Instead, we showed that a signal-subtraction method could resolve this issue for our samples. We, therefore, conclude that the failure of dose recovery ratios at high given doses may be a result of incorrect correction of the residual signal.

The failure of the dose recovery can also be affected by the charge carry-over from natural/regenerative doses to the subsequent test doses (e.g., Nian et al., 2016; Yi et al., 2016;
Colarossi et al., 2019) and the initial sensitivity change during the measurement of natural signals (e.g., Wallinga et al., 2000; Kars et al., 2014b; Zhang, 2018; Qin et al., 2018). In this study, we found that increasing the size of test dose to 200 Gy did not improve the dose recovery results at large doses (Fig. 7). Furthermore, for the same given/regenerative dose, the effect of charge carry-over should be similar and independent of measurement cycles, which is not the case, as shown in Fig. 8b, where the $L_x/T_x$ values are identical for the 2nd and 3rd measurement cycles but both differ from the 1st cycle.

Therefore, we conclude that the effect of the charge carry-over cannot explain the failure of dose recovery for our samples. Instead, we show that, after residual signal correction, the values of repeated $L_x/T_x$ measurements of equal doses remain unchanged for the MET-pIRIR$_{290}$ signal, further confirming that the effect of initial sensitivity change is not an issue for the high-temperature MET-pIRIR signals, although it did affect the lower-temperature signals.

Our study also suggested that an overestimation in the dose recovery test does not necessarily mean that the $D_e$ estimation is also wrong. This is because the reliability of $D_e$ estimation depends on the magnitude of the residual signal prior to sample burial. If the residual signal has been well reset during natural processes before burial, the $D_e$ estimates should be reliable. Fortunately, this appears to be the case for many samples and sites, including our samples. For example, Thiel et al (2011) found for sediments from Japanese loess that the dose recovery ratios were consistent with unity at 290°C pIRIR signal for smaller given doses (< 100 Gy), while overestimation was observed for larger given dose. Despite this, the dating results are reliable up to 500 Gy, when comparing with independent ages.

Although we can estimate the hard-to-bleach signal component in dose recovery tests and apply a signal-subtraction method to correct for a residual problem, it is difficult to apply this method to natural samples because the residual signal prior to burial is unknown. Fortunately, it appears that the natural process can bleach the hard-to-bleach signals, according to the observation of a positive correlation between residual signal and natural signal obtained in our study (Fig. 11b) and in previous studies (e.g., Buylaert et al., 2012; Lauer et al., 2017; Schatz et al., 2012; Sohbati et al., 2012). It is likely that the so-called non-bleachable or residual signals could have been better reset in natural processes than in laboratory conditions (i.e., solar simulator) (e.g., Thiel et al., 2011; Sohbati et al., 2012; Kars et al., 2014a). As a result, subtracting the residual signal observed by bleaching the natural sample in the laboratory from natural signal may result in underestimation in $D_e$, and, hence, it should only be applied to correct for residual in dose recovery tests but not for $D_e$ estimation. The best way to estimate the residual signal prior to burial is to measure modern analogues or check the relationship between the residual signal and the natural signal using a series of samples with different ages or $D_e$ (e.g., Sohbati et al., 2012). In the case that there is no modern analogue or a series of samples to estimate the residual signals for natural samples, reliable $D_e$ can only be obtained by estimating the easy-to-bleach component and hard-to-bleach component for both the natural and regenerative
signals, and interpolating the bleachable natural signal onto the corresponding dose response curves (Fig. 3m–r).

5 Conclusion

Severe $D_r$ overestimation in dose recovery tests was observed when the simple dose-subtraction method was applied for old samples. This is attributed to the effect of the dose-dependent residual signal, which can be corrected using a signal-subtraction method. Our study suggests that, apart from young samples, caution is needed for the residual correction for old samples.

Acknowledgements

This study was supported by an Australian Research Council Future Fellowship to Bo Li (FT140100384), grants from the National Natural Science Foundation of China to Yujie Guo (No. 41702192) and postgraduate scholarships from the China Scholarship Council and the University of Wollongong to Xue Rui (201506010345). We thank Jiafu Zhang, Baoyin Yuan, Yanyan Yan and Junkang Wang for help with the field investigations and the collection of luminescence samples, Richard G. Roberts, Zenobia Jacobs, Yasaman Jafari and Terry Lachlan for essential support in the luminescence dating laboratory, Mariana Sontag-González for proofreading and Tony Reimann and another anonymous reviewer for their helpful comments.


Wintle, A.G., 2008. Luminescence dating: where it has been and where it is going. Boreas 37, 471–482.


Table 1

Summary of depositional contexts, grain sizes, and equivalent dose ($D_e$) estimates for the studied samples.

<table>
<thead>
<tr>
<th>Sample code</th>
<th>Section</th>
<th>Deposit type</th>
<th>Grain size ($\mu$m)</th>
<th>Re-normalized $L_n/T_n$ (1σ error)$^a$</th>
<th>Measured equivalent dose (Gy)$^b$</th>
</tr>
</thead>
<tbody>
<tr>
<td>YJG-01</td>
<td>YJG</td>
<td>Loess</td>
<td>125–180</td>
<td>0.16 ± 0.02</td>
<td>5.2 ± 4.1</td>
</tr>
<tr>
<td>YJG-08</td>
<td>YJG</td>
<td>Loess</td>
<td>125–180</td>
<td>0.30 ± 0.01</td>
<td>35.7 ± 2.5</td>
</tr>
<tr>
<td>HTL-Loc1-2</td>
<td>HTL-Loc1</td>
<td>Fluvial</td>
<td>125–180</td>
<td>0.30 ± 0.01</td>
<td>35.3 ± 3.2</td>
</tr>
<tr>
<td>DDP-3</td>
<td>DDP</td>
<td>Fluvial-lacustrine</td>
<td>90–125</td>
<td>1.38 ± 0.01</td>
<td>1048.5 ± 38.9</td>
</tr>
<tr>
<td>DDP-7</td>
<td>DDP</td>
<td>Loess</td>
<td>90–125</td>
<td>0.82 ± 0.01</td>
<td>236.6 ± 6.5</td>
</tr>
<tr>
<td>TEG-1</td>
<td>TEG</td>
<td>Fluvial-lacustrine</td>
<td>90–125</td>
<td>1.46 ± 0.05</td>
<td>1477.9 ± 281.0</td>
</tr>
<tr>
<td>HSP-1</td>
<td>HSP</td>
<td>Fluvial-lacustrine</td>
<td>90–125</td>
<td>1.46 ± 0.04</td>
<td>1477.7 ± 223.6</td>
</tr>
<tr>
<td>HY-1</td>
<td>HY</td>
<td>Fluvial-lacustrine</td>
<td>90–125</td>
<td>1.46 ± 0.01</td>
<td>1442.5 ± 79.8</td>
</tr>
<tr>
<td>HY-3</td>
<td>HY</td>
<td>Fluvial-lacustrine</td>
<td>90–125</td>
<td>1.38 ± 0.01</td>
<td>1096.9 ± 47.5</td>
</tr>
<tr>
<td>ZJP-4</td>
<td>ZJP</td>
<td>Fluvial-lacustrine</td>
<td>90–125</td>
<td>1.31 ± 0.02</td>
<td>860.4 ± 50.8</td>
</tr>
</tbody>
</table>

$^a$ The natural signal ($L_n$), a single regenerative-dose signal ($L_r$, given dose 373 Gy) and the corresponding test dose signals ($T_n$ and $T_r$) were measured for aliquots from each sample. The central age model (CAM) was applied to the re-normalised ratios ($L_n/T_n$) to get the central value.

$^b$ The equivalent dose of each sample was obtained by projecting the central value of the re-normalized $L_n/T_n$ ratios of the MET-pIRIR290 onto the corresponding SAR SGC without any residual correction.
Table 2

Multiple-aliquot regenerative-dose (MAR) procedure for MET-pIRIR measurements (Li et al., 2017).

<table>
<thead>
<tr>
<th>Step</th>
<th>Treatment</th>
<th>Signal</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Give regenerative dose, $D_i^a$</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Preheat at 320°C for 60 s</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>IRSL measurement at 50°C for 100 s</td>
<td>$L_{x(50)}$</td>
</tr>
<tr>
<td>4</td>
<td>IRSL measurement at 100°C for 100 s</td>
<td>$L_{x(100)}$</td>
</tr>
<tr>
<td>5</td>
<td>IRSL measurement at 150°C for 100 s</td>
<td>$L_{x(150)}$</td>
</tr>
<tr>
<td>6</td>
<td>IRSL measurement at 200°C for 100 s</td>
<td>$L_{x(200)}$</td>
</tr>
<tr>
<td>7</td>
<td>IRSL measurement at 250°C for 100 s</td>
<td>$L_{x(250)}$</td>
</tr>
<tr>
<td>8</td>
<td>IRSL measurement at 290°C for 100 s</td>
<td>$L_{x(290)}$</td>
</tr>
<tr>
<td>9</td>
<td>Give test dose, 60 Gy</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Preheat at 320°C for 60 s</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>IRSL measurement at 50°C for 100 s</td>
<td>$T_{x(50)}$</td>
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<td>12</td>
<td>IRSL measurement at 100°C for 100 s</td>
<td>$T_{x(100)}$</td>
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<tr>
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<td>IRSL measurement at 150°C for 100 s</td>
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<td>14</td>
<td>IRSL measurement at 200°C for 100 s</td>
<td>$T_{x(200)}$</td>
</tr>
<tr>
<td>15</td>
<td>IRSL measurement at 250°C for 100 s</td>
<td>$T_{x(250)}$</td>
</tr>
<tr>
<td>16</td>
<td>IRSL measurement at 290°C for 100 s</td>
<td>$T_{x(290)}$</td>
</tr>
<tr>
<td>17</td>
<td>Solar simulator bleach for 4 h</td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>Give normalisation dose, $D_r$</td>
<td></td>
</tr>
<tr>
<td>19</td>
<td>Preheat at 320°C for 60 s</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>IRSL measurement at 50°C for 100 s</td>
<td>$L_{r(50)}$</td>
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<td>24</td>
<td>IRSL measurement at 250°C for 100 s</td>
<td>$L_{r(250)}$</td>
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<tr>
<td>25</td>
<td>IRSL measurement at 290°C for 100 s</td>
<td>$L_{r(290)}$</td>
</tr>
<tr>
<td>26</td>
<td>Give test dose, 60 Gy</td>
<td></td>
</tr>
<tr>
<td>27</td>
<td>Preheat at 320°C for 60 s</td>
<td></td>
</tr>
<tr>
<td>28</td>
<td>IRSL measurement at 50°C for 100 s</td>
<td>$T_{r(50)}$</td>
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<td>29</td>
<td>IRSL measurement at 100°C for 100 s</td>
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<td>IRSL measurement at 150°C for 100 s</td>
<td>$T_{r(150)}$</td>
</tr>
<tr>
<td>31</td>
<td>IRSL measurement at 200°C for 100 s</td>
<td>$T_{r(200)}$</td>
</tr>
<tr>
<td>32</td>
<td>IRSL measurement at 250°C for 100 s</td>
<td>$T_{r(250)}$</td>
</tr>
<tr>
<td>33</td>
<td>IRSL measurement at 290°C for 100 s</td>
<td>$T_{r(290)}$</td>
</tr>
</tbody>
</table>

*a For the natural sample, $i = 0$ and $D_i = 0$ Gy, and the observed signals are denoted as $L_n$ and $T_n$. 
Table S1

The single-aliquot regenerative-dose (SAR) procedure for multiple elevated temperature post-infrared IRSL (MET-pIRIR) measurements (Li et al., 2014b).

<table>
<thead>
<tr>
<th>Step</th>
<th>Treatment</th>
<th>Signal</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Give regenerative dose, $D_i$</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>Preheat at 320°C for 60 s</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>IRSL measurement at 50°C for 100 s</td>
<td>$L_x(50)$</td>
</tr>
<tr>
<td>4</td>
<td>IRSL measurement at 100°C for 100 s</td>
<td>$L_x(100)$</td>
</tr>
<tr>
<td>5</td>
<td>IRSL measurement at 150°C for 100 s</td>
<td>$L_x(150)$</td>
</tr>
<tr>
<td>6</td>
<td>IRSL measurement at 200°C for 100 s</td>
<td>$L_x(200)$</td>
</tr>
<tr>
<td>7</td>
<td>IRSL measurement at 250°C for 100 s</td>
<td>$L_x(250)$</td>
</tr>
<tr>
<td>8</td>
<td>IRSL measurement at 290°C for 100 s</td>
<td>$L_x(290)$</td>
</tr>
<tr>
<td>9</td>
<td>Give test dose, 60 Gy</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Preheat at 320°C for 60 s</td>
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<td>11</td>
<td>IRSL measurement at 50°C for 100 s</td>
<td>$T_x(50)$</td>
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<td>IRSL measurement at 100°C for 100 s</td>
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</tr>
<tr>
<td>15</td>
<td>IRSL measurement at 250°C for 100 s</td>
<td>$T_x(250)$</td>
</tr>
<tr>
<td>16</td>
<td>IRSL measurement at 290°C for 100 s</td>
<td>$T_x(290)$</td>
</tr>
<tr>
<td>17</td>
<td>Solar simulator bleach for 4 h</td>
<td></td>
</tr>
<tr>
<td>18</td>
<td>Repeat step 1–17 for different $D_i$</td>
<td></td>
</tr>
</tbody>
</table>

*For the natural sample, $i = 0$ and $D_i = 0$ Gy, and the observed signals are denoted as $L_n$ and $T_n$.\[553\]
Figures’ captions

Fig. 1. (a) Map showing the locations of the Nihewan Basin (red rectangle). (b) DEM map showing the east of the Nihewan Basin, the Sanggan River and the sample locations: Dadaopo (DDP), Heshangping (HSP), Hutoulang locality 1 (HTL-Loc1), Hongya (HY), Taiergou (TEG), Yujiagou (YJG), Zhaojiaping (ZJP).

Fig. 2. Procedures to build total (Group A) and residual (Group B) standardised growth curves (SGCs).

Fig. 3. $L_x/T_x$ ratios (total signals, a–f), standardised growth curves (SGCs) for the LS-normalised total signals (g–l), and SGCs for the normalised total, residual and bleachable signals (m–r) at different stimulation temperatures. The total signals were measured using the SAR MET-pIRIR procedure listed in Table S1. The residual signals were obtained using aliquots bleached by solar simulator for 8 h and measured using the MAR MET-pIRIR listed in Table 2. Both the total SGCs (black lines in g–l, as well black dashed lines in m–r) and the residual SGCs (red lines in m–r) have been normalised to unity at a dose of 373 Gy. The bleachable SGCs (red dashed lines in m–r) were obtained by subtracting the residual SGCs (red lines) from the total SGCs (black dashed lines).

Fig. 4. Residual signal measured after different bleaching time using solar simulator for sample DDP-3. All signals are normalized to unity at 0 h bleaching time. Each data point represents the average of results from four aliquots.

Fig. 5. The dose recovery and residual results for IRSL signals at different temperatures from (a) DDP-3 and (b) HSP-1. The corrected $D_e$ values (open triangle) were obtained by subtracting the residual doses (filled circle) from the measured recovered doses (open square). The grey lines represent the corresponding given dose for each sample. The error bar is 1σ.

Fig. 6. Comparison of the dose recovery results corrected by the signal-subtraction method with the corresponding estimated intensity for (a) DDP-3 and (b) HSP-1. The recovery signal with the bleachable component (red open square) was obtained by subtracting the residual signal (red filled circle) from the total recovery signal (red filled square). The estimated signal (black open square) was obtained by interpolating the corresponding given dose onto the bleachable SGC. The error bar is 1σ.

Fig. 7. Re-normalised $L_x/T_x$ of repeated pIRIR measurements on multiple aliquots with the test dose of 200 Gy. The error bar is 1σ.

Fig. 8. The $L_x/T_x$ signal (total signal, filled black square) and the corresponding residual signal (filled red circle) from the repeated measurements with a fixed regenerative dose (1000 Gy) for sample HY-1 with (a) 50°C and (b) 290°C. The bleachable signal (open square) was obtained by subtracting the residual signal from the total signal.
Fig. 9. Modelled $D_e$ values to paleodose ($P$) ratio plotted against $P$. The data in each panel are based on different magnitudes of $B$ (maximum intensity of the residual signal) relative to $A$ (maximum intensity of the bleachable signal) (i.e., 0.04$A$, 0.08$A$, 0.12$A$ and 0.16$A$). For each of the assumed $B$ values, the $L_{RN}$ value (residual signal before burial) is set at 0, 0.2$B$, 0.4$B$, 0.6$B$ and 0.8$B$. The different magnitudes of $L_{RN}$ and $B$ used for these simulations are shown in each panel.

Fig. 10. Modelled $D_e$ values to $P$ ratio plotted against the $P$. The data in each panel are based on different value of $D_R$ (the characteristic saturating dose of the residual signal) (1000, 2000, 3000, 4000 Gy). For each of the assumed $D_R$ value, the $L_{RN}$ value is set at 0, 0.2$B$, 0.4$B$, 0.6$B$ and 0.8$B$. The different magnitude of $L_{RN}$ and $D_R$ used for these simulations are shown in each panel.

Fig. 11. (a) Residual doses for the MET-pIRIR signals for different samples from the Nihewan Basin against IR stimulation temperature. Some data are missing as the measured signal is lower than the zero-dose of the corresponding SAR SGC. (b) The relationship between residual signal and the corresponding natural signal for the MET-pIRIR$_{290}$ of different samples. The filled symbols are the residual signals of the natural signals. The open symbols are the residual signals of the natural aliquots with given dose (YJG-01 and 08).

Fig. S1. Dose-corrected modelled $D_e$ values to paleodose ($P$) ratio plotted against $P$. The data in each panel are based on different magnitudes of $B$ (maximum intensity of the residual signal) relative to $A$ (maximum intensity of the bleachable signal) (i.e., 0.04$A$, 0.08$A$, 0.12$A$ and 0.16$A$). For each of the assumed $B$ values, the $L_{RN}$ value (residual signal before burial) is set at 0, 0.2$B$, 0.4$B$, 0.6$B$ and 0.8$B$. The different magnitudes of $L_{RN}$ and $B$ used for these simulations are shown in each panel. The residual dose for dose-subtraction correction is obtained by setting $L_{RN}$(residual of natural signal) = $L_R$ (regenerative signal).

Fig. S2. Dose-corrected modelled $D_e$ values to $P$ ratio plotted against $P$. The data in each panel are based on different values of $D_R$ (characteristic saturating dose of the residual signal) (1000, 2000, 3000, 4000 Gy). For each of the assumed $D_R$ value, the $L_{RN}$ value is set at 0, 0.2$B$, 0.4$B$, 0.6$B$ and 0.8$B$. The different magnitudes of $L_{RN}$ and $D_R$ used for these simulations are shown in each panel. The residual dose for dose-subtraction correction is obtained by setting $L_{RN} = L_R$. 
Fig. 4

![Graph showing the relationship between solar simulator bleaching time and normalized residual IRSL signal for different temperatures. The graph includes data points for 50°C, 100°C, 150°C, 200°C, 250°C, and 290°C.](image)
Fig. 5

(a) DDP-3

(b) HSP-1

---

IRSL temperature, °C

---

Equivalent dose, Gy

---

Recovery
Residual
Dose_subtraction
Fig. 6

(a) DDP-3

(b) HSP-1
Fig. 8

(a) 50 °C

Re-normalised Lx/Tx

- Total_signal
- Residual_signal
- Corrected_signal

(b) 290 °C

Re-normalised Lx/Tx

- Total_signal
- Residual_signal
- Corrected_signal

Repeating cycles
Fig. 9

(a) $B = 0.04 \times A$

(b) $B = 0.08 \times A$

(c) $B = 0.12 \times A$

(d) $B = 0.16 \times A$
Fig. 11

(a) Residual dose, Gy vs. Stimulation temperature, °C

(b) Residual signal vs. Natural signal
Fig. S1

(a) B = 0.04 * A

(b) B = 0.08 * A

(c) B = 0.12 * A

(d) B = 0.16 * A
Fig. S2

(a) $D_R = 1000$

(b) $D_R = 2000$

(c) $D_R = 3000$

(d) $D_R = 4000$