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We investigated the infrared stimulated luminescence (IRSL) and post-infrared IRSL (pIRIR) signals emitted by K-feldspars from sedimentary samples from Asia, Europe and Africa using a single-aliquot multiple elevated temperature (MET) stimulation procedure. For separate aliquots of the same sample, we show that variation among the dose response curves (DRCs), or growth curves, constructed from the regenerative dose signal (L_x), the test dose signal (T_x , an indicator of luminescence sensitivity) and the sensitivity-corrected signal (L_x/T_x) can be largely eliminated by normalising the DRCs using one of the regenerative dose signals; we call this procedure 'regenerative-dose normalisation' or re-normalisation. Furthermore, for the MET-pIRIR signals measured at 250 °C, we find that different samples have re-normalised DRCs that follow the same growth function, despite the samples differing significantly in terms of their geological provenance, sedimentary context, equivalent dose (D_e) and luminescence sensitivity. This common feature offers the potential to establish a 'global standardised growth curve' (gSGC) for different samples of K-feldspar, and thereby enable D_e values to be estimated for a large number of single aliquots by projecting the re-normalised natural signals on to the gSGC. For the 18 samples investigated in this study, we find that D_e estimates obtained from the gSGC are consistent with those obtained using full single-aliquot regenerative dose (SAR) procedures for doses of up to ~1600 Gy. The establishment of a gSGC would greatly reduce the time required to date older samples using K-feldspar, as regenerative doses of several hundreds to a few thousands of Gy are typically delivered to each aliquot in each SAR cycle.

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Construction of a ‘global standardised growth curve’ (gSGC) for infrared stimulated luminescence dating of K-feldspar

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Abstract

We investigated the infrared stimulated luminescence (IRSL) and post-infrared IRSL (pIRIR) signals emitted by K-feldspars from sedimentary samples from Asia, Europe and Africa using a single-aliquot multiple elevated temperature (MET) stimulation procedure. For separate aliquots of the same sample, we show that variation among the dose response curves (DRCs), or growth curves, constructed from the regenerative dose signal (L_x), the test dose signal (T_x , an indicator of luminescence sensitivity) and the sensitivity-corrected signal (L_x/T_x) can be largely eliminated by normalising the DRCs using one of the regenerative dose signals; we call this procedure ‘regenerative-dose normalisation’ or re-normalisation. Furthermore, for the MET-pIRIR signals measured at 250 °C, we find that different samples have re-normalised DRCs that follow the same growth function, despite the samples differing significantly in terms of their geological provenance, sedimentary context, equivalent dose (D_e) and luminescence sensitivity. This common feature offers the potential to establish a ‘global standardised growth curve’ (gSGC) for different samples of K-feldspar, and thereby enable D_e values to be estimated for a large number of single aliquots by projecting the re-normalised natural signals on to the gSGC. For the 18 samples investigated in this study, we find that D_e estimates obtained from the gSGC are consistent with those obtained using full single-aliquot regenerative dose (SAR) procedures for doses of up to ~1600 Gy. The establishment of a gSGC would greatly reduce the time required to date older samples using K-feldspar, as regenerative doses of several hundreds to a few thousands of Gy are typically delivered to each aliquot in each SAR cycle.

Keywords: Potassium feldspar, IRSL, MET-pIRIR, dose response curves, gSGC.

1. Introduction

The single-aliquot regenerative dose (SAR) procedure (Galbraith et al., 1999; Murray and Wintle, 2000) has been widely adopted for optically stimulated luminescence (OSL) dating of quartz, and applied subsequently to infrared stimulated luminescence (IRSL) dating of potassium-rich feldspar (K-feldspar) (Wallinga et al., 2000). In the SAR procedure, a test dose is applied and the induced luminescence measured after stimulating the natural dose signal (L_n) and each of the regenerative dose signals (L_x). The corresponding test dose signals (T_n , T_x) are used to monitor and correct for any sensitivity changes induced by the laboratory treatments, which include irradiation, preheating and optical stimulation. The sensitivity-corrected signals for a series of regenerative doses (L_x/T_x) are used to construct a dose response curve (DRC), and the equivalent dose (D_e) is determined by projecting the sensitivity-corrected natural signal (L_n/T_n) on to the DRC.

Based on 6 samples of quartz from Australia and polymineral grains of loess from China, Roberts and Duller (2004) suggested that T_x not only corrected for sensitivity changes but could also be used for between-aliquot normalisation, thereby allowing a direct comparison among different aliquots of the same or different samples. This formed the basis for establishing a standardised growth curve (SGC). Roberts and Duller (2004) suggested that, once a reliable SGC had been established, D_e values may be estimated accurately based solely on measurements of L_n and T_n , thus offering a convenient means of saving on instrument time when D_e estimates are required for a large number of aliquots and/or for dating old samples.

The SGC approach was subsequently tested and applied successfully to quartz samples from a variety of regions, including agricultural and archaeological deposits in Scotland (Burbidge et al., 2006), loess from China, Germany and the Great Plains in the USA (Lai, 2006; Lai et al., 2007), sand dunes in South Africa and Florida in the USA (Telfer et al., 2008), lacustrine samples from the Qinghai–Tibetan Plateau in China (Long et al., 2010) and fluvial deposits from the Mississippi Valley in the USA (Shen and Mauz, 2011). The SGC method was

further improved by [Li et al. \(submitted\)](#), who suggested that normalisation of the DRCs using one of the regenerative doses—the so-called regenerative-dose normalisation (or re-normalisation) method—can further reduce the between-aliquot and between-sample variation among quartz DRCs. They tested the re-normalisation procedure on single aliquots of Asian, African, European and North American quartz and found that many of the samples followed a similar DRC function up to a dose of ~250 Gy, despite differing significantly in terms of their geological provenance, environmental setting and depositional age.

Compared to quartz OSL, however, there has been little study on the extension of the SGC method to the K-feldspar IRSL signal, except for the polymineral IRSL signals of Chinese loess examined by [Roberts and Duller \(2004\)](#). Recently, [Li et al. \(2014a\)](#) reported that K-feldspar samples from different regions of Eurasia exhibited a dose-dependency in their post-IR IRSL (pIRIR) signals and that these samples appeared to share a common DRC, hence suggesting the possibility of developing a ‘global standardised growth curve’ (gSGC) for K-feldspar. Potassium-rich feldspars have several advantages over quartz as a luminescence dosimeter, including a higher luminescence sensitivity and saturation dose limit, so there are benefits in exploring the potential to establish a gSGC for feldspars. It would prove especially useful for dating of older samples, as each SAR cycle involves the application of regenerative doses of up to several thousand Gy for each aliquot.

In this study, we examine the IRSL and pIRIR signals emitted by K-feldspar grains in sedimentary samples collected from sites in Asia, Europe and Africa. We combine a single-aliquot multiple elevated temperature (MET) stimulation procedure ([Li and Li, 2011; Li et al., 2014a](#)) with the re-normalisation method proposed for quartz by [Li et al. \(submitted\)](#), and find that this greatly reduces between-aliquot variation in DRC shape for separate aliquots of K-feldspar from the same and different samples. By developing a gSGC for K-feldspar using the MET-pIRIR signal measured at 250 °C—which has previously been shown to fade negligibly ([Li and Li, 2011](#))—we propose a practicable means of making rapid and reliable measurements of D_e for a large number of aliquots, which will be particularly advantageous for dating of Middle Pleistocene samples.

2. Sample descriptions

Eighteen sediment samples were examined in this study to assess the variability in the properties of K-feldspars from different parts of Asia (China, Georgia, India and Indonesia), Europe (France and Italy) and Africa (Libya and Kenya). Sample locations are shown in Fig. 1, and the depositional contexts, experimental measurement conditions, and D_e ranges are summarised in Table 1. Loess samples LC-110, -230 and -270 were taken from the Luochuan section on the Chinese Loess Plateau, and have been used previously for testing the MET-pIRIR method (Li and Li, 2012a, 2012b). Aeolian sand samples Sm0404, 5, 6' and 8 were collected from the Shimao section on the southeastern margin of the Mu Us Desert in central China (Sun et al., 1999), and have been used previously to investigate dose-dependent sensitivity changes (Li et al., 2013b, 2014a). Samples MTL-OSL7 and DGT-OSL1 are lacustrine sediments from the Nihewan Basin in China; TUT-OSL1 and -OSL2 are alluvial sediments from Talepu in southwest Sulawesi, Indonesia; RUP-2 and -4 are samples of alluvium from Rusinga Island, Kenya (Tryon et al., 2010); and HF12-6023 is an aeolian-colluvial sample taken from the Haua Fteah cave in northeast Libya (Douka et al., 2014). DHB2-OSL4 and PIN-OSL2 consist of colluvial and alluvial sediments deposited at the open-air archaeological sites of Dhaba in north-central India and Pinavera in the southern Caucasus, Georgia, respectively. CEP-OSL1 was collected from a colluvial-alluvial palaeosol sequence at Ceprano, Italy, and LC10-16 from a collapsed cave at Les Cottés, France (Jacobs et al., 2015). The latter four samples have also been investigated in our previous MET-pIRIR studies (Li et al., 2013a, 2014a).

3. Experimental procedures and analytical facilities

Samples were prepared for IRSL analysis using routine procedures (Aitken, 1998). First, the samples were treated with HCl acid and H₂O₂ solution to remove carbonates and organic matter, respectively, and then dried and sieved to obtain grains of 63–90, 90–125, 125–180, 150–180 and 180–212 μm in diameter. K-feldspar grains were separated from quartz and heavy minerals using a solution of sodium polytungstate with a density of 2.58 g/cm³. The separated K-feldspar grains were immersed in 10% HF acid for 40 min to etch the surfaces of the grains and remove the outer, alpha-irradiated portions, and then rinsed in HCl acid to remove any

precipitated fluorides. After drying, the etched K-feldspar grains were mounted as a monolayer on stainless steel discs of 9.8 mm diameter using “Silkospray” silicone oil as an adhesive. Grains covered the central ~5 mm diameter portion of each disc, corresponding to several hundreds to thousands of grains per aliquot.

IRSL measurements were made on an automated Risø TL-DA-20 reader equipped with IR-emitting diodes for stimulation (870 $\pm$ 40 nm). The total IR power delivered to the sample position was ~135 mW/cm² (Bøtter-Jensen et al., 2003) and laboratory irradiations were carried out on the reader using a calibrated ⁹⁰Sr/⁹⁰Y beta source. IRSL signals were detected by an Electron Tubes Ltd 9235B photomultiplier tube fitted with Schott BG-39 and Corning 7-59 filters to restrict transmission to 320–480 nm. Each IR stimulation was made for 100 s (Table 2), and the resulting signal was calculated as the sum of counts over the initial 5 s of measurement, with ‘late light’ subtraction (Aitken, 1998) of the background count rate over the final 5 s of stimulation. Prior to each IRSL measurement, an ‘IR-off’ period of up to 50 s was applied to minimise interference from the isothermal decay signal (Fu et al., 2012).

Samples were measured using the single-aliquot ‘pre-dose’ MET-pIRIR (pMET-pIRIR) procedure proposed by Li et al. (2014a) and outlined in Table 2. This procedure is similar to the standard MET-pIRIR procedure of Li and Li (2011), except that a 2 hr solar simulator bleach is given at the end of each SAR cycle, instead of a high-temperature IR bleach. The purpose of the solar bleach is to reset the dose-dependent sensitivity of the MET-pIRIR signals, based on the observations by Li et al. (2013b) that the sensitivity of these signals is reduced to a stable level after a 2 hr bleach using a UVACUBE 400 solar simulator. Li et al. (2014a) found that this bleach effectively reset the dose-dependent sensitivity between successive SAR cycles and that, consequently, both L_x and T_x signals could be used for D_e determination.

Table 1 lists the preheat temperatures, MET-pIRIR stimulation temperatures and sizes of test dose given to each of the study samples. Preheats were made at either 300 °C for 60 s or, for four of the samples, at 320 °C for 60 s, with the same preheat applied to the natural, regenerative and test doses. The MET-pIRIR signals were measured by stimulating with IR at successively higher temperatures, from 50 to 250 °C in steps of 50 °C. In

1 addition, for the four samples preheated to 320 °C, an extra MET-pIRIR stimulation temperature of 280 °C was
2 included. The size of the test dose ranged from 24 to 66 Gy. We show later that the shape of the DRCs is mainly
3 influenced by the IR stimulation temperature, whereas the preheat temperature and size of the test dose have
4 little effect.

5 **4. Regenerative-dose normalisation**

6 In this study, we tested whether the re-normalisation method proposed by [Li et al. \(submitted\)](#) for quartz
7 could be extended to K-feldspar. The basic idea of this method is to re-normalise the DRCs using one of the
8 regenerative dose signals to reduce the between-aliquot variation in the extent of sensitivity change from the
9 measurement of the natural or regenerative dose signals to that of the corresponding test dose signals. This can
10 be achieved by re-normalisation, as demonstrated mathematically by [Li et al. \(submitted\)](#). To briefly recap,
11 assuming a single electron trap gives rise to the luminescence signals induced by the natural, regenerative and
12 test doses (following [Chen et al., 2011](#)), then the sensitivity-corrected signal (L/T) can be described using a
13 single saturating exponential function:

$$14 \quad L/T = \left(1 - e^{-\frac{D}{D_0}}\right) / \left[k \left(1 - e^{-\frac{D_t}{D_0}}\right)\right] \quad (1)$$

15 where L is L_n or L_x , T is T_n or T_x , D is the radiation dose received, D_t is the size of the test dose, D_0 is the
16 characteristic saturation dose, and the constant k describes any change in luminescence sensitivity between
17 measurement of the natural or regenerative dose signal and stimulation of the subsequent test doses.

18 According to eqn. 1, the sensitivity-corrected luminescence signal (L/T) is dependent on D_t , D_0 and k . If
19 D_t remains the same throughout the SAR procedure and different aliquots have similar D_0 values (see below),
20 then the shape of the DRC for each aliquot is determined by its k -value. Different aliquots may have different k -
21 values, however, because their constituent grains might have experienced different irradiation, bleaching and
22 thermal treatments in the geological past. Each of these factors could influence the extent of sensitivity change

(i.e., k) induced by laboratory irradiation, preheating and bleaching, as has been reported for quartz OSL (e.g., Murray and Wintle, 1999a, 1999b; Chen et al., 2001). This effect is also expected to influence the IRSL signals from K-feldspar, because of the known dependence of the sensitivity of feldspar IRSL to these same factors (e.g., Blair et al., 2005; Li et al., 2013b).

To overcome the problem of variations in k , Li et al. (submitted) proposed an improved method to normalise DRCs for single aliquots of the same or different samples, using one of the regenerative dose signals (L_{r1}). Based on eqn. 1, it follows that the re-normalised sensitivity-corrected signal (L_x/T_x) can be expressed as:

$$\frac{L_x/T_x}{L_{r1}/T_{r1}} = \left(1 - e^{-\frac{D}{D_0}}\right) / \left(1 - e^{-\frac{D_{r1}}{D_0}}\right) \quad (2)$$

where D_{r1} is the regenerative dose chosen for re-normalisation. This method produces a DRC independent of k and, hence, it should be able to reduce the between-aliquot variation associated with the different dosing, bleaching and thermal histories of individual grains. Li et al. (submitted) noted, however, that re-normalisation is unlikely to eliminate all of the between-aliquot variation, because the D_0 value may differ among aliquots and the k -value may be dose dependent (e.g., Murray and Wintle, 2000), although the latter dependency should not influence the accuracy of D_e estimation (Wintle and Murray, 2006).

To test whether the re-normalisation method is applicable to different samples of K-feldspar, we compared the DRCs for the sensitivity-corrected MET-pIRIR 250 °C signals of samples CEP-OSL1, PIN-OSL2, MTL-OSL7 and LC-230 (Fig. 2). Of these four samples, PIN-OSL2 and CEP-OSL1 exhibited a large extent of between-aliquot scatter in L_x/T_x (black diamonds in Fig. 2a and 2b, respectively) and in L_n/T_n for PIN-OSL2 (data plotted on the y-axis). After re-normalising these DRCs using one of the regenerative dose signals (denoted by the blue arrows in Fig. 2), the scatter in L_x/T_x and L_n/T_n are reduced significantly (red circles in Fig. 2). For example, the relative standard deviation (RSD) of L_n/T_n for PIN-OSL2 is 23% ($n = 6$) and this is reduced to 13% after re-normalisation (using the regenerative dose signal at 770 Gy for each aliquot). For the same sample, the

1 RSD values for the L_x/T_x ratios at regenerative doses of 385, 770, 1210 and 1650 Gy range from 8 to 16%, which
2 are reduced to 1–4% after re-normalisation (Fig. 2b).

3 A similar trend is observed for CEP-OSL1 using the L_x/T_x signal at 1650 Gy to re-normalise each of the
4 aliquots. In this instance, the RSD of L_x/T_x for the regenerative doses fell from 4–6% to 1–2%, but the RSD of
5 L_n/T_n decreased negligibly, from 3.8 to 3.5%. The other two samples (MTL-OSL7 and LC-230) showed little
6 between-aliquot scatter in L_x/T_x and L_n/T_n (i.e., RSD values of 1–2 % for L_x/T_x), so no significant improvements
7 were gained after re-normalising (Fig. 2c and 2d). We attribute the small scatter in L_x/T_x and L_n/T_n for these two
8 samples to the smaller grain size used (63–90 μm): this results in each aliquot containing more grains, so there is
9 a larger grain-averaging effect and, hence, reduced between-aliquot variation.

10 The results in Fig. 2 suggest that re-normalisation is especially useful for reducing between-aliquot
11 variations in the DRCs for samples with a large scatter in L_x/T_x , such as CEP-OSL1 and PIN-OSL2. In such
12 cases, k evidently varies among aliquots of the same sample, but this variation can be largely removed by re-
13 normalising using one of the regenerative dose signals. In the next section, we compare the DRCs of different
14 samples after re-normalisation.

15 **5. Between-sample comparisons of DRCs**

16 To test whether the re-normalisation procedure could also reduce inter-sample variations in DRC shape,
17 we measured the IRSL and MET-pIRIR signals for a range of samples from around the world (Table 1). The re-
18 normalised DRCs for the L_x/T_x , L_x and T_x signals (measured at different stimulation temperatures) are shown for
19 these samples in Fig. 3, 4 and 5, respectively. Sixteen of the samples were measured using the single-aliquot
20 pMET-pIRIR procedure (Table 2), and Sm0404 and DGT-OSL1 were measured using the multiple-aliquot
21 pMET-pIRIR procedure of [Li et al. \(2013b\)](#) to enable a comparison of the DRCs obtained using single- and
22 multiple-aliquot methods.

Each of the data points in Fig. 3–5 is based on the average of 4–6 aliquots; the associated relative standard errors are typically only a few percent and are omitted for clarity. Fig. 3a–e shows the DRCs for the re-normalised L_x/T_x signals measured at stimulation temperatures of 50–250 °C. The DRCs have been multiplied by a scaling factor, such that the regenerative dose DRC for each sample passes through a re-normalised L_x/T_x value of unity at 770 Gy; this is equivalent to normalising each data set using the regenerative dose signal at 770 Gy. Unlike the DRCs from quartz OSL, which can be fitted by either a single saturating exponential function or a saturating exponential plus linear function (see Li et al., submitted), the compiled DRCs from the K-feldspars investigated in this study appear to be more homogeneous and are best represented by a double saturating exponential function:

$$y = y_1 * \left(1 - e^{-\frac{x}{D_1}}\right) + y_2 * \left(1 - e^{-\frac{x}{D_2}}\right) + y_0$$

where y_0 , y_1 and y_2 are constants, and D_1 and D_2 are the characteristic saturation doses of the two exponential components. We note that, as in previous studies (e.g., Thomsen et al., 2011), our fitting with two exponential components is purely empirical and no physical justification is provided. The best-fitted DRCs are shown as solid lines in Fig. 3a–d and the values of the fitting parameters are listed in Table 3. For the 250 °C MET-pIRIR signal (Fig. 3e), the best-fitted curve is shown as a dashed line and, for comparison, the same dashed curve is displayed in Fig. 3a–d. The following features are revealed in Fig. 3:

- a) The 250 °C L_x/T_x signal saturates at a lower dose than those measured at lower stimulation temperatures, consistent with the smaller D_0 values estimated from curve fitting (Table 3).
- b) There appears to be a dose-dependency in the shape of the DRCs for the L_x/T_x signals measured at 50, 100, 150 and 200 °C for the different samples. At 50 °C, for example, the samples have similar IRSL DRCs up to a dose of ~1000 Gy, but then diverge (Fig. 3a). A similar trend is observed for the 100 and 150 °C MET-pIRIR signals (Fig. 3b and 3c).
- c) The 200 and 250 °C MET-pIRIR L_x/T_x signals (Fig. 3d and 3e) are more similar in shape for the different samples. At 200 °C, the signals deviate only above a dose ~1500 Gy (Fig. 3d), while the

250 °C signals are indistinguishable up to the maximum applied dose of 3600 Gy (Fig. 3e). The DRCs obtained using the single-aliquot pMET-pIRIR procedure (Table 2) appear to be identical with those of samples Sm0404 and DGT-OSL1, which were measured using the multiple-aliquot pMET-pIRIR procedure (Li et al., 2013b).

- d) The similarity in the DRCs for the different samples suggests that there is little dependence on either the preheat temperature or the size of the test dose, both of which varied between samples (Table 1).
- e) The D_0 values of the DRCs in Fig. 3 are consistent with the observations of Li and Li (2011), who suggested an upper dose limit of ~800–1000 Gy for the sensitivity-corrected (L_x/T_x) MET-pIRIR signals.

Fig. 4a–e shows the re-normalised DRCs for the sensitivity-uncorrected (L_x) MET-pIRIR signals of these same samples; the DRCs are, as in Fig. 3, re-normalised to unity at ~770 Gy and fitted by a double saturating exponential function. Li et al. (2014a) suggested that the L_x pMET-pIRIR signals are more reproducible than the L_x/T_x counterparts and they calculated a D_0 value of ~850 Gy for the 6 samples investigated. On this basis, they suggested that L_x could be used to measure much larger doses than can be accomplished with L_x/T_x , thereby significantly extending the dating range into the Middle Pleistocene. A similar result is obtained in this study, where we have investigated an even more diverse set of samples and applied the re-normalisation procedure. The following features are apparent in Fig. 4:

- a) The L_x signals saturate at a much larger dose than do the corresponding L_x/T_x signal (Fig. 3), as demonstrated by their greater D_0 values (Table 3).
- b) For the 50 °C IRSL signal (Fig. 4a), the DRCs for the different samples appear to diverge above a dose of ~1500 Gy. This divergence is less prominent for the MET-pIRIR L_x signals measured at higher temperatures (Fig. 4b–e), and all of the samples appear to share a similar DRC shape for the 250 °C MET-pIRIR signal (Fig. 4e). For the latter signal, the DRCs obtained using the single-aliquot procedure are indistinguishable from those obtained for samples Sm0404 and DGT-OSL1 using the multiple-aliquot procedure.

- c) As the DRCs for the various IRSL and MET-pIRIR L_x signals are indistinguishable up to ~1500 Gy, a common DRC can be established up to this dose limit. Above this dose, however, the 250 °C L_x signal saturates earlier than the L_x signals stimulated at lower temperatures.
- d) As with the L_x/T_x data, we can discern no dependence of the shape of the DRCs on either preheat temperature or the size of the test dose.
- e) The D_0 values of the DRCs in Fig. 4 suggest an upper dose limit of ~1600–2000 Gy for the L_x signal.

Finally, the DRCs for the test dose (T_x) signal—a proxy for sample sensitivity—are shown for the IRSL and MET-pIRIR signals of the different samples in Fig. 5. The DRCs have been re-normalised to unity at ~770 Gy and fitted using a double saturating exponential function. The DRCs for the different samples are widely scattered when the T_x signal is stimulated at 50 °C (Fig. 5a), but this scatter is reduced at higher stimulation temperatures (Fig. 5b–e). Furthermore, the dose dependence (i.e., the ratio between the signal intensities at high and low doses) is greater for the MET-pIRIR signals measured at higher stimulation temperatures. The least scatter in the DRCs is observed for the 250 °C MET-pIRIR signal, but there remains considerable scatter in the low-dose range (<300 Gy) even at this elevated temperature, which would prevent the reliable estimation of D_e for young samples using the T_x signal. The D_0 values of the DRCs in Fig. 5 suggest an upper dose limit of ~2000–2300 Gy for the T_x signal.

6. Towards a global standardised growth curve for K-feldspar

We have found that different samples from around the world conform to a highly reproducible DRC when the high temperature MET-pIRIR signals, especially those measured at 250 °, are combined with the re-normalisation procedure proposed by Li et al. (submitted). On the basis of this common feature, we now examine the prospects of developing a global standardised growth curve (gSGC) to facilitate the rapid estimation of D_e for single aliquots of K-feldspar.

The test-dose normalised SGC method proposed previously (e.g., [Roberts and Duller, 2004](#); [Lai, 2006](#); [Lai et al., 2007](#)) required measurements of only L_n and T_n for each aliquot, and the L_x/T_x ratios were then projected on to the SGC. Burbidge et al. (2006) recommended that one additional regenerative dose response could be measured to check the validity of the SGC. The procedure proposed here is similar to their suggestion, but the additional regenerative dose signal (L_{r1}) and corresponding test dose signal (T_{r1}) are measured for each aliquot in order to re-normalise the DRCs. With this information, the D_e of the re-normalised L_x/T_x signal can be calculated by solving the following equation using an iterative method:

$$\frac{f(D_{r1})}{L_{r1}/T_{r1}} = \frac{f(D_e)}{L_n/T_n} \quad (3)$$

where $f(D)$ is the gSGC function for the L_x/T_x signal and D_{r1} is the regenerative dose used for re-normalisation. The form of this equation is equally applicable to L_x and T_x (as the denominators) and the fitting parameters of the DRCs plotted in Fig. 3 are listed in Table 3. Li et al. (submitted) explain how eqn. 3 is derived and recommend that D_{r1} should be close in size to the D_e to minimise the scatter about the gSGC remaining after re-normalisation.

To test the reliability of D_e estimation using the gSGC functions established for the 250 °C MET-pIRIR L_x/T_x , L_x and T_x signals (Fig. 3e, 4e and 5e, respectively), 84 individual aliquots from 14 samples were analysed using the method described above. Here we focus on the 250 °C MET-pIRIR signal for D_e determination, because it has the most reproducible DRC for the different stimulation temperatures tested (Fig. 3–5) and has a negligible fading rate ([Li and Li, 2011](#)). In Fig. 6, the D_e values obtained for the L_x/T_x , L_x and T_x signals using the gSGC are compared against those determined by constructing full SAR growth curves for each aliquot. Reliable D_e estimates cannot be obtained from the L_x/T_x signal beyond a dose of ~1000 Gy (Fig. 6a) due to the substantial uncertainties associated with projecting L_x/T_x ratios on to the gSGC as it approaches saturation (Fig. 3e). By contrast, the L_x signal has much smaller uncertainties for D_e values greater than 1000 Gy (Fig. 6c), owing to this signal having a much higher saturation dose level (Fig. 4e). For the L_x signal, the gSGC and individual DRCs

yield consistent D_e values up to a dose of ~1600 Gy (Fig. 6c and 6d), and the T_x signal gives a similar result (Fig. 6e and 6f). We did not estimate D_e values smaller than 250 Gy for the T_x signal, because of its limited dose-dependency and the large between-sample variation in the low-dose range (Fig. 5e).

The D_e estimates for the L_x/T_x signal obtained from the gSGC and from individual DRCs are consistent at 2σ , up to a dose of ~1000 Gy. That is, the relationship between the two data sets is statistically consistent with the 1:1 line in Fig. 6a and 6b, and most of the points deviate no more than 10% from this line. Similar results were obtained for the L_x and T_x signals, with both of these relationships extending to much higher doses (Fig. 6c–f). The D_e values obtained from the gSGC and individual DRCs are displayed as ratios in Fig. 7 for the L_x/T_x , L_x and T_x signals. The ratios are plotted against the D_e values estimated from individual DRCs in the left-hand panels, and as radial plots in the right-hand panels. Most of the ratios are consistent with unity at 2σ , suggesting that D_e estimates as accurate as those obtained from full DRCs can be obtained by using the gSGC for the 250 °C MET-pIRIR signals.

7. Discussion

Li et al. (submitted) have suggested that the shapes of the DRCs constructed from L_x/T_x signals are controlled by two factors: k and D_0 (see eqn. 1). The k -value is determined by sensitivity changes occurring between the natural or regenerative dose measurement and the corresponding test dose measurement, and is expected to vary among samples, aliquots and grains. As a result, the specific experimental conditions adopted for stimulation, irradiation and preheating may induce different k -values and, hence, differences in the shapes of the DRCs. Individual grains are likely to have experienced different histories of irradiation, bleaching and perhaps heating before burial, any of which may cause a change in their luminescence sensitivity, so there is likely to be natural variation in k -values among single grains. This variation will increase further if the grains also respond differently to the experimental treatments given in the laboratory, even if all of the grains are measured using identical procedures. This predicted variability is supported by our results for the MET-pIRIR L_x/T_x signal measured at 250 °C using the SAR procedure, which yielded large between-aliquot variation for two

1 of the four samples tested (Fig. 2a and 2b). We have shown that the re-normalisation procedure can substantially
2 reduce such variation in the L_x/T_x signal by producing a DRC independent of k (see eqn. 2).

3 However, re-normalisation is of greatest benefit when the sensitivity-uncorrected signal (L_x) is used for
4 D_e determination (Li et al., 2014a). The sensitivity may vary significantly between aliquots, but the variation can
5 be largely eliminated using the re-normalisation procedure. Fig. 8 presents such an example using sample MTL-
6 OSL7. In Fig. 8a, the 250 °C MET-pIRIR L_x signals for 6 aliquots are plotted against the corresponding
7 regenerative doses. Large variations in L_x and L_n (the latter shown on the y-axis) are observed for the different
8 aliquots, but re-normalising these signals using the regenerative dose signal at 638 Gy effectively eliminates this
9 scatter (Fig. 8b) and enables a well-defined DRC to be fitted to the re-normalised L_x signals.

10 The samples investigated in this study were collected from a variety of geological settings in various
11 parts of Asia, Europe and Africa, so they are expected to have different luminescence properties and pre-burial
12 histories of irradiation, bleaching and heating, resulting in a range of k -values. In addition, the inherent IRSL
13 sensitivity (T_n) of these samples is highly variable, as shown in Fig. 9 by the distribution of the 250 °C MET-
14 pIRIR T_n signal intensities for the 84 aliquots included in Fig. 6 and 7. The sensitivity varies over 3 orders of
15 magnitude, from less than 10 counts/Gy to more than 5,000 counts/Gy. Yet despite such large variation in T_n , the
16 re-normalisation method allows the DRCs for different samples to be compared and then combined to form a
17 gSGC.

18 Furthermore, we have shown that although these samples were measured using a variety of experimental
19 conditions (e.g., different preheat conditions and sizes of test dose), they yield essentially indistinguishable
20 DRCs after re-normalisation (Fig. 3–5). By largely eliminating the variation caused by differences in
21 measurement conditions and in luminescence properties of K-feldspars from different regions of the world, it is
22 possible to establish a gSGC based on the re-normalised DRCs for the MET-pIRIR signals—and the 250 °C
23 signal in particular. We have confirmed that D_e values based on the gSGC are consistent with those obtained

1 from individual DRCs, so we consider the gSGC as a robust and rapid means of generating multiple estimates of
2 D_e for K-feldspars, especially for older samples that require the application of large laboratory doses.

3 We expect that the gSGC established here will be applicable to other K-feldspar samples measured using
4 the same MET-pIRIR signals, but we suggest that a few aliquots (e.g., 4–6) from each sample or each site should
5 be measured routinely using a full SAR procedure to test if the re-normalised DRCs have the same shape as the
6 gSGC proposed here. The existence of a gSGC for the MET-pIRIR signals also foreshadows the probable
7 existence of a common DRC for other pIRIR signals, including two-step pIRIR signals (e.g., [Thomsen et al.,](#)
8 [2011](#)). However, as DRC shapes can differ significantly for different pIRIR signals (Fig. 3–5 and [Li et al.,](#)
9 [2014b](#)), it may be necessary to construct the re-normalised DRC specific to each signal.

10 Finally, although we have demonstrated that a gSGC may be established for single aliquots of K-
11 feldspar extracted from a variety of samples, the existence of a common DRC does not necessarily imply that
12 reliable age estimates will be obtained, because the natural signals are measured separately. The latter caveat
13 applies equally to the D_e values estimated using the full SAR procedure. For this reason, dose recovery tests
14 (Roberts et al., 1999), fading tests, residual signal measurements and age checks against independently dated
15 samples also need to be performed to demonstrate that a series of known doses can be accurately determined
16 from the re-normalised DRC. Furthermore, to maximise the benefits of establishing a gSGC for D_e
17 determination, future research could usefully focus on investigating the variability in the re-normalised DRCs of
18 individual grains of K-feldspar from the same and different samples. Incorporation of this knowledge into single-
19 grain pIRIR dating procedures may make it practicable to obtain reliable D_e values from a large number of
20 individual grains, thereby enabling questions of pre-depositional bleaching and post-depositional mixing to be
21 readily investigated.

22 8. Conclusions

The IRSL and MET-pIRIR signals emitted by single aliquots of K-feldspar from a variety of geological regions appear to share a common dose response curve—which we refer to as a global standardised growth curve (gSGC)—when one of the regenerative dose signals is used as a normalisation device to reduce the between-aliquot variation in the shapes of the individual DRCs. Once the gSGC has been constructed, equivalent doses can be rapidly estimated for other aliquots by measuring only the natural signal, one regenerative dose signal (for normalisation) and the corresponding test dose signals. We have provided the gSGC fitting parameters for the IRSL signal measured at 50 °C and the MET-pIRIR signals stimulated at 100, 150, 200 and 250 °C. The D_e values estimated for 84 single aliquots using the gSGC determined for the 250 °C signal are consistent at 2σ with those obtained from the individual DRC of each aliquot. The gSGC greatly reduces the time required to measure a large number of aliquots and is, therefore, particularly useful for pIRIR dating of K-feldspar samples with large D_e values.

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Figure captions

Figure 1: Map showing the geographic locations of the sites and samples investigated.

Figure 2: The sensitivity-corrected regenerative dose (L_x/T_x) signals (black diamonds, left-hand axis) and their re-normalised counterparts (red circles, right-hand axis) for samples CEP-OSL1 (a), PIN-OSL2 (b), MTL-OSL7 (c) and LC-230 (d). The sensitivity-corrected natural (L_n/T_n) signals, before and after re-normalisation, are plotted on the y-axis. The re-normalised data were calculated using the procedure described in the text, in which one of the L_x/T_x signals (indicated by the blue arrow) is chosen for each sample. Note that the re-normalised data are offset slightly to the right on the x-axis for clarity.

Figure 3: Dose response data for the re-normalised IRSL and MET-pIRIR L_x/T_x signals from 18 samples of K-feldspar (see Table 1 for details). These sensitivity-corrected signals were measured at stimulation temperatures of 50 °C (a), 100 °C (b), 150 °C (c), 200 °C (d) and 250 °C (e), and each data set has been normalised to unity at 770 Gy for comparison. Each data point is the average results of 4–6 aliquots and the standard errors are only a few percent. The solid lines in (a)–(d) and the dashed line in (e) are the best-fit curves to the data for all 18 samples using a double saturating exponential function. The same dashed line is also shown in (a)–(d). The symbols in (a)–(e) are the same and the legend is shown in the bottom right-hand corner of the figure.

Figure 4: Dose response data for the re-normalised IRSL and MET-pIRIR L_x signals from the same 18 samples as in Fig. 3. The sensitivity-uncorrected signals were measured at stimulation temperatures of 50 °C (a) to 250 °C (e), and the data and best-fit curves are displayed in the same manner as in Fig. 3.

Figure 5: Dose response data for the re-normalised IRSL and MET-pIRIR T_x signals from the same 18 samples shown in Fig. 3 and 4 (see text for details). These test dose signals represent the luminescence sensitivity of the different samples when stimulated at temperatures of 50 °C (a) to 250 °C (e). The data and DRCs are displayed in the same manner as in Fig. 3 and 4.

Figure 6: Comparisons of the 250 °C MET-pIRIR D_e values estimated for 84 single aliquots using their L_x/T_x (a, b), L_x (c, d) and T_x (e, f) signals and the gSGC procedure (y-axis) or the full SAR growth curve for each

aliquot (x -axis). In each panel, the solid line shows the 1:1 relationship and the dashed lines shows the 10% deviation from the 1:1 line. Panels (a), (c) and (e) show the full range of D_e values, while panels (b), (d) and (f) show those smaller than 500 Gy. The 84 aliquots consist of 5 aliquots from DHB2-OSL4 and MTL-OSL7; 6 aliquots from each of samples RUP-2, RUP-4, Sm5, Sm6', Sm8, PIN-OSL2, LC-110, LC-230, LC-270, CEP-OSL1 and LC10-16; and 8 aliquots from TUT-OSL1.

Figure 7: The same data as presented in Fig. 6, but displayed as the ratios between the gSGC and individual DRC D_e values obtained for 84 single aliquots from the L_x/T_x , L_x and T_x signals, plotted against the individual DRC D_e values in panels (a), (c) and (e), respectively. The corresponding radial plots of these D_e ratios are shown in panels (b), (d) and (f).

Figure 8: Intensities of the sensitivity-uncorrected (L_n) 250 °C MET-pIRIR signals emitted by 6 aliquots of sample MTL-OSL7, plotted against the corresponding regenerative doses (a). The same data as in (a) but re-normalised using the regenerative dose signal at 638 Gy (b). In both plots, the L_n signals are shown on the y -axis.

Figure 9: Natural sensitivities of the 250 °C MET-pIRIR signals for the 84 single aliquots of 14 samples (listed in the legend) shown in Fig. 6 and 7. The sensitivities were calculated from the T_n signal intensities divided by the test dose (in Gy), and are ranked in order of increasing sensitivity (i.e., T_n intensity per unit dose).

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Table 1: Summary of sample locations, depositional contexts, measurement conditions and equivalent dose (D_e) estimates for the study samples.

Sample code	Country of origin	Depositional context	Grain size (μm)	Preheat ($^{\circ}\text{C}$)	MET-pIRIR temp. ($^{\circ}\text{C}$)	Test dose (Gy)	Method of D_e determination ^a	Approximate age ^b	D_e range ^c (Gy)
LC-110	China ^d	Loess	63–90	320	50–250	33	SA	LP	260–360
LC-230	China ^d	Loess	63–90	320	50–250	33	SA	MP	820–950
LC-270	China ^d	Loess	63–90	320	50–250	33	SA	MP	880–1100
Sm0404	China ^e	Aeolian sand	90–125	300	50–250	33	MA	LP	360–450
Sm5	China ^e	Aeolian sand	180–212	300	50–250	48	SA	MP	390–440
Sm6'	China ^e	Aeolian sand	90–125	300	50–250	48	SA	MP	1100–1250
Sm8	China ^e	Aeolian sand	150–180	300	50–250	33	SA	MP	1350–1600
DGT-OSL1	China ^f	Lake	63–90	320	50–280	51	MA	EP	>2000 ^g
MTL-OSL7	China ^f	Lake	63–90	320	50–280	51	SA	MP	1000–1100
LC10-16	France	Colluvial–alluvial	90–125	300	50–250	24	SA	LP	80–120
PIN-OSL2	Georgia	Colluvial–alluvial	90–180	300	50–250	33	SA	MP	600–900
DHB2-OSL4	India	Colluvial–alluvial	125–180	300	50–250	24	SA	LP	150–190
TUT-OSL1	Indonesia	Alluvial	180–212	320	50–250	55	SA	LP	350–700
TUT-OSL2	Indonesia	Alluvial	180–212	320	50–250	55	SA	MP	400–900
CEP-OSL1	Italy	Colluvial–alluvial	125–180	300	50–250	33	SA	MP	1100–1450
RUP-2	Kenya	Alluvial	180–212	320	50–280	44	SA	LP	170–310
RUP-4	Kenya	Alluvial	180–212	320	50–280	44	SA	LP	220–800
HF12-6023	Libya	Aeolian–colluvial	180–212	300	50–250	66	SA	LP	250–450

^a Single aliquot (SA) or multiple aliquot (MA).

^b Early Pleistocene (EP), Middle Pleistocene (MP) or Late Pleistocene (LP).

^c D_e estimates based on gSGC results for the sensitivity-uncorrected MET-pIRIR 250 $^{\circ}\text{C}$ signals (L_n and L_x) measured for 4–6 aliquots of each sample.

^d Samples from the Chinese Loess Plateau.

^e Samples from the Mu Us Desert.

^f Samples from the Nihewan Basin.

^g Finite D_e estimates could not be obtained as this sample was in dose saturation.

Table 2: The ‘pre-dose’ multiple elevated temperature pIRIR (pMET-pIRIR) procedure.

Step	Treatment	Observed
1	Give regenerative dose, D_i	
2	Preheat at 300 or 320 °C for 60 s	
3	IRSL measurement at 50 °C for 100 s	$L_{x(50)}$
4	IRSL measurement at 100 °C for 100 s	$L_{x(100)}$
5	IRSL measurement at 150 °C for 100 s	$L_{x(150)}$
6	IRSL measurement at 200 °C for 100 s	$L_{x(200)}$
7	IRSL measurement at 250 °C for 100 s	$L_{x(250)}$
8	IRSL measurement at 280 °C for 100 s ^a	$L_{x(280)}$
9	Give test dose, D_t	
10	Preheat at 300 or 320 °C for 60 s	
11	IRSL measurement at 50 °C for 100 s	$T_{x(50)}$
12	IRSL measurement at 100 °C for 100 s	$T_{x(100)}$
13	IRSL measurement at 150 °C for 100 s	$T_{x(150)}$
14	IRSL measurement at 200 °C for 100 s	$T_{x(200)}$
15	IRSL measurement at 250 °C for 100 s	$T_{x(250)}$
16	IRSL measurement at 280 °C for 100 s ^a	$T_{x(280)}$
17	Solar simulator bleach for 2 hr	
18	Return to step 1	

^a Steps 8 and 16 were used only for samples DGT-OSL1, MTL-OSL7, RUP-2 and RUP-4 (see Table 1). For the other 14 samples, the highest IR stimulation temperature used was 250 °C.

Table 3: Summary of the parameters for the best-fit gSGC functions shown in Fig. 3, 4 and 5 for the L_x/T_x , L_x and T_x signals measured at various IR stimulation temperatures.

IR stimulation temp. (°C)	Signal	Parameters of the gSGC function $f(D)$ ^a					2.3 * D_0 (Gy) ^b	3 * D_0 (Gy) ^b
		y_1	D_1 (Gy)	y_2	D_2 (Gy)	y_0		
50	L_x/T_x	0.57 ± 0.19	241 ± 66	0.88 ± 0.15	1115 ± 344	0.008 ± 0.009	2000	2780
	L_x	0.36 ± 0.25	276 ± 137	1.37 ± 0.19	1187 ± 287	0.003 ± 0.009	2450	3280
	T_x	0.20 ± 0.24	259 ± 279	0.34 ± 0.15	1462 ± 191	0.685 ± 0.014	1420	2520
100	L_x/T_x	0.39 ± 0.12	159 ± 48	0.91 ± 0.10	741 ± 105	0.018 ± 0.008	1420	1950
	L_x	0.41 ± 0.28	304 ± 131	1.22 ± 0.23	1146 ± 288	0.010 ± 0.008	2300	3100
	T_x	0.28 ± 0.22	310 ± 220	0.42 ± 0.26	2715 ± 639	0.640 ± 0.015	2900	4960
150	L_x/T_x	0.43 ± 0.11	177 ± 41	0.88 ± 0.09	801 ± 109	0.026 ± 0.006	1500	2060
	L_x	0.40 ± 0.27	327 ± 132	1.26 ± 0.23	1157 ± 263	0.015 ± 0.007	2340	3130
	T_x	0.31 ± 0.33	372 ± 300	0.45 ± 0.18	2533 ± 608	0.610 ± 0.014	2880	4760
200	L_x/T_x	0.30 ± 0.06	119 ± 32	0.95 ± 0.05	661 ± 49	0.034 ± 0.006	1320	1780
	L_x	0.34 ± 0.28	316 ± 145	1.24 ± 0.25	1023 ± 205	0.020 ± 0.006	2080	2800
	T_x	0.37 ± 0.28	372 ± 218	0.43 ± 0.24	2792 ± 709	0.573 ± 0.013	2980	5120
250	L_x/T_x	0.37 ± 0.10	142 ± 35	0.74 ± 0.09	545 ± 62	0.062 ± 0.005	1000	1380
	L_x	0.17 ± 0.12	197 ± 116	1.32 ± 0.10	830 ± 79	0.028 ± 0.005	1780	2360
	T_x	0.48 ± 0.37	410 ± 210	0.45 ± 0.15	2175 ± 420	0.455 ± 0.011	2500	4060

^a The dose response data were fitted using a double saturating exponential function of the form $f(D) = y_1 * \left(1 - e^{-\frac{x}{D_1}}\right) + y_2 * \left(1 - e^{-\frac{x}{D_2}}\right) + y_0$, with the best-fit parameters of y_1 , D_1 , y_2 , D_2 and y_0 listed above.

^b The 2.3 * D_0 and 3 * D_0 values correspond to the doses at 90% and 95%, respectively, of the saturation level for a growth curve fitted using a single saturating exponential function – that is, the doses at 10% and 5% below the saturation level, respectively. An arbitrary 2 * D_0 value, which corresponds to the dose at 86% of the saturation level, has been suggested as a safe upper limit for reliable D_e estimation (Wintle and Murray, 2006), but if the shape of the growth curve is characterised accurately then much higher D_e values can be estimated (Galbraith and Roberts, 2012). The D_e estimates have increasingly large standard errors at values above 3 * D_0 , as the natural signals impinge on the saturated region of the growth curves.