

# Dose measurement using the fast component of LM-OSL signals from quartz

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## Abstract

A method is described for equivalent dose determination using the linearly modulated optically stimulated luminescence (LM-OSL) signal from quartz. After analysis of the LM-OSL curves, only the fast component from quartz is used for equivalent dose calculation. The results of applying the method to purified quartz and to grains without mineral separation are presented.

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**Keywords:** Quartz; LM-OSL; Fast component; Signal separation

## 1. Introduction

Since optically stimulated luminescence (OSL) dating was first introduced by [Huntley et al. \(1985\)](#), the technique has been widely applied in the dating of various archaeological and geological sediments ([Aitken, 1998](#)). The luminescence from minerals, e.g. quartz and feldspar, is used to estimate the total dose (equivalent dose  $D_e$ ) from environmental radiation following deposition of the mineral grains. The age of the sediment is given by the ratio of the accumulated dose ( $D_e$ ) to the dose rate which is estimated by measuring the content of radioactive elements in the sample itself and surrounding materials. Compared with thermoluminescence (TL) dating, OSL dating has advantages in fast and complete bleaching of the signals by sunlight. This is particularly important for sediments which may have only been bleached for a short period of time prior to deposition ([Li, 2001](#)). Several techniques for  $D_e$  determination have been suggested ([Aitken, 1998](#)). The development of the single-aliquot regenerative-dose (SAR) technique has greatly increased the precision of  $D_e$  determinations ([Murray and Roberts, 1998](#); [Murray and Wintle, 2000](#)). In the SAR technique, the initial part of the OSL signal is primarily the part of the signal that is of interest for dating.

The usual method of optical stimulation, known as continuous-wave (CW) stimulation, uses constant excitation

power. Using such stimulation, an approximately exponential decay of OSL is observed. Another way of optical stimulation is called linear modulation (LM-OSL), in which the excitation power increases linearly from zero to a maximum ([Bulur, 1996](#)). Using this technique, the OSL is observed in the form of a curve containing several peaks.

In the CW-OSL decay curves, it has been shown that there are several overlapping OSL signals, which have different optical decay rates. [Smith and Rhodes \(1994\)](#) suggested that the OSL signals from 514.5 nm laser stimulation were associated with three traps having different decay constants. [Bailey et al. \(1997\)](#) have identified three exponential components in the CW-OSL curves, namely “fast”, “medium” and “slow” components. CW-OSL is a mixture of these components, and even the OSL from the very beginning of the stimulation period contains a contribution from all components. By using the LM-OSL technique, each component with a different photoionization cross-section produces a discrete peak in the LM-OSL curve. The implementation of blue (470 nm) light-emitting diode (LED) stimulation units has enabled the study of LM-OSL to be made using an automated OSL dating system ([Bøtter-Jensen et al., 1999a,b](#)).

There are several reports on the LM-OSL signals from quartz (e.g. [Jain et al., 2003](#); [Kuhns et al., 2000](#); [Singarayer and Bailey, 2003, 2004](#)). [Bulur et al. \(2000\)](#) reported that LM-OSL of heated natural quartz can be well deconvoluted by using a linear combination of several first-order peaks, and four OSL components were identified in their study. More recently, five or six components have been recognized in the LM-OSL signals

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from different quartz samples using stimulation light centered at 470 nm (Jain et al., 2003; Singarayer and Bailey, 2003). Although different peaks were identified in these studies, the fast component was similar in them all. The photoionization cross-section of the fast component was obtained by analyzing the LM-OSL signals from sedimentary quartz using different stimulation power and ramping time. The LM-OSL of feldspar has not been studied anywhere near as intensively, though a curve for a sedimentary feldspar extract containing its natural dose was published by Wallinga et al. (2002).

If a non-fast component is present in the initial part of CW-OSL signals, especially the medium component, the equivalent dose determined with the initial signal might be problematic (Watanuki, 2002; Tsukamoto et al., 2003; Choi et al., 2003). Tsukamoto et al. (2003) reported a significant dependence of  $D_e$  on illumination time. Using an exponential form to describe the relationship between  $D_e$  and recuperation, they obtained the  $D_e$  values of the fast component by extrapolating the exponential curve to zero recuperation based on the assumption that the fast component is free from recuperation. Jain et al. (2005) proposed a differential-OSL SAR procedure for isolating the fast component, this procedure is based on the fact that the fast component is depleted by IR stimulation at 160 °C but under these conditions there is negligible depletion of the medium component and slow components (Singarayer and Bailey, 2004; Jain et al., 2003). Using the LM-OSL technique, Singarayer and Bailey (2003) explored the dose response curves of different components in quartz and their application to dating.

In this study, the LM-OSL curves were analyzed using the curve fitting programme in Origin 7.0 software. We aimed to investigate the feasibility of  $D_e$  measurement using only the fast component of the LM-OSL of quartz, and the possibility of separating the fast component of quartz LM-OSL from feldspar-contaminated samples.

## 2. Samples and apparatus

All of the samples used in this study were sediments of late Quaternary age from different locations (Table 1). These samples were previously dated with CW-OSL measurements, but the results are unpublished.

Quartz grains were separated from all of the samples after procedures of sieving, heavy liquid separation and HF acid etching in subdued red safe-light conditions. Raw samples were treated first with 20%  $H_2O_2$  and 10% HCl to remove organic materials and carbonates. Grains between 125 and 150  $\mu\text{m}$  were selected by dry sieving. Then grains with densities between 2.62 and 2.75  $\text{g}/\text{cm}^3$  were separated using sodium polytungstate heavy liquid; those separated grains were treated with 40% HF acid for 2 h to remove feldspar grains. In order to study the LM-OSL properties from K-feldspar, grains with densities from 2.58 to 2.53  $\text{g}/\text{cm}^3$  were separated as K-feldspar for samples Hld-1, Hld-4 and D4. The feldspar grains were treated with 10% HF acid for 1 h. Grains from sample Hld-1 without heavy liquid separation and HF etching were regarded as an unseparated sample. The mineral grains were mounted on 10-mm-diameter aluminum discs with Silkospray silicone oil

Table 1

Information about the samples used in this study

Sample	Type	Location	Age, ka
Hld-1	Eolian dune	Hulun Buir desert, China	12
Hld-4	Eolian dune	Hulun Buir desert, China	3.1
Rkz	Eolian sediment	Lahsa, Tibet plateau, China	15.4
Dzk	Eolian sediment	Tibet plateau, China	3
Dgw-1	Eolian sand dune	Mu Us desert, China	12.4
D4	Eolian dune	Kurqin desert, China	4.5
Dc-1	Colluvial sediment	Shanxi, China	80

Table 2

The LM-OSL single-aliquot regenerative-dose protocol

Step	Treatment
1	Give regenerative dose $Di^a$
2	Preheat at 260 °C for 10 s
3	LM-OSL measurement at 125 °C for 400 s
4	Bleaching with CW stimulation for 100 s
5	Give test dose
6	Preheat at 260 °C for 10 s
7	LM-OSL measurement at 125 °C for 400 s
8	Bleaching with CW stimulation for 100 s
9	Return to step 1

<sup>a</sup>For the natural,  $i = 0$  and  $D_0 = 0$ . The whole sequence was repeated for several regenerative doses including a zero dose and a repeat dose.

for measurement. Grains used for both CW-OSL and LM-OSL measurements were treated with the same procedures. Where appropriate the purity of quartz was tested by monitoring the presence of feldspar through measuring the IRSL signals.

OSL measurements were performed using an automated Risø TL/OSL DA-15 reader equipped with excitation units containing blue light-emitting diodes (LEDs,  $470 \pm 30 \text{ nm}$ ) (Bøtter-Jensen et al., 1999a,b). The OSL signal is detected through two 3-mm U-340 filters. Irradiation was carried out using a  $^{90}\text{Sr}/^{90}\text{Y}$  beta source built into the reader with a dose rate of 0.0899 Gy/s to quartz on aluminum discs. All LM-OSL measurements were made by ramping the stimulation power from 0 to 50  $\text{mW cm}^{-2}$  over a period of 400 s. All LM-OSL measurements were carried out at 125 °C in order to prevent retrapping of charges from the shallow trap corresponding to the 110 °C TL peak (Murray and Wintle, 1998).

For  $D_e$  determination with LM-OSL signals, the single aliquot regenerative dose (SAR) protocol, similar to that outlined by Murray and Wintle (2000), was applied in this study (Table 2). The OSL was measured using LM stimulation instead of CW stimulation. The preheating was 260 °C for 10 s. In order to ensure the degree of bleaching is the same as that in CW-OSL measurement, each LM-OSL measurement was followed by a CW stimulation at 125 °C for 100 s using 90% of the maximum optical power. Each disc underwent measurement cycles of preheating, LM-OSL measurement and CW stimulation, sensitivity measurement and irradiation. A different regenerative dose was applied in each cycle. Three regenerative doses and one repeat dose were applied to each sample disc.

### 3. Analysis of the LM-OSL curves

The natural LM-OSL signals of the quartz grains from samples Hld-1, Hld-4, Rkz, Dzk, Dgw-1 and Dc-1 are shown in Fig. 1(a). The OSL signals were normalized to the maximum intensity of each curve for comparison. A sharp peak can be identified after stimulating for 13 s for all samples, followed by slowly increasing signals with stimulating time. The similar position of the first peak of the different quartz samples implies a common detrapping rate or photoionization cross-section for the fast component in OSL signals.

The natural LM-OSL signals from K-feldspar grains of samples Hld-4, Hld-1 and D4 are shown in Fig. 1(b). Clearly, quartz and K-feldspar have significant differences in the shape of the LM-OSL decay curves. For K-feldspar the first peak appears after 35 s of stimulation, followed by a monotonic decrease in signal with increasing stimulation time. The curves from K-feldspar show broader peaks than those from quartz, as found by Wallinga et al. (2002). These indicate that K-feldspar has a lower detrapping rate in the fast component and a higher detrapping rate in the slow components than quartz.

The decay rate of LM-OSL signals is determined by the intensity of the stimulation light  $I_0$  and the photoionization cross-section (or the detrapping probability)  $\alpha$ . Assuming a single trap, a single recombination center and quasi-equilibrium conditions (Bulur, 1996; Chen and McKeever, 1997), for the first-order kinetics, the OSL curve can be described as

$$L(t) = N_0 \frac{\alpha I_0}{P} t \exp \left[ -\frac{\alpha I_0 t^2}{2P} \right], \quad (1)$$

where  $N_0$  is the initial number of trapped electrons,  $I_0$  is the maximum stimulation light intensity,  $\alpha$  is a constant proportional to the photoionization cross-section,  $P$  is the duration of stimulation and  $t$  is the stimulating time. By de-convoluting the LM-OSL curve, we can estimate the parameters, e.g. the photoionization cross-section  $\alpha$  and the initial number of trapped electrons  $N_0$ , of the traps associated with the LM-OSL signal. The parameters of the curve can be analyzed from the peak position  $t_m$  and the peak maximum  $L_m$ :

$$t_m = \sqrt{\frac{P}{\alpha I_0}} \quad (2)$$

and

$$L_m = N_0 \sqrt{\frac{\alpha I_0}{P}} \exp(-0.5) = \frac{N_0}{t_m} \exp(-0.5). \quad (3)$$

Curve fitting for typical LM-OSL signals from quartz and feldspar are shown in Fig. 2(a) and (b), respectively. As shown in the figures, at least four components, namely fast, medium, S1 and S2, are necessary to fit the LM-OSL signals. It is noted that three components of quartz, fast, medium and S1, were depleted to a negligible level within the measurement period of 400 s. The peak position  $t_m$  of the fast and medium components of quartz are given as 13 and 30 s, respectively, whereas the peaks of the fast and medium components from the feldspar grains appear around 33 and 60 s. Hence, the fast component

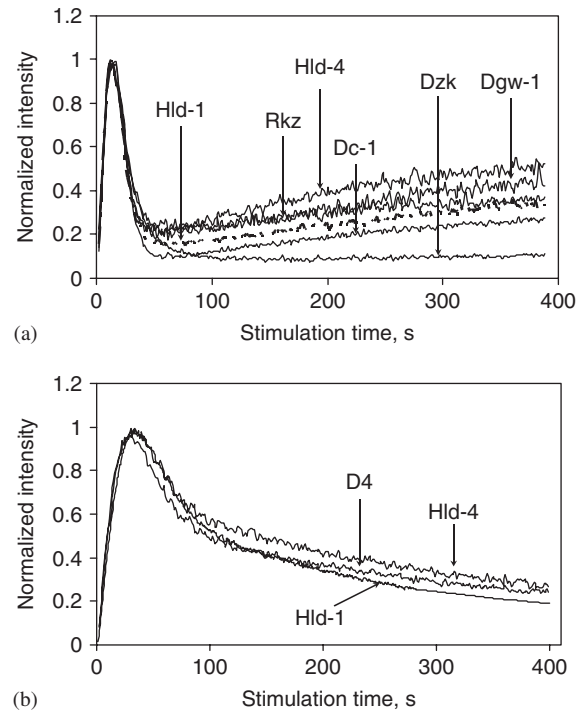


Fig. 1. (a) Natural LM-OSL curves of quartz fractions for sediments from different regions. (b) Natural LM-OSL curves of feldspar fractions. The LM-OSL was measured at 125 °C for 400 s after preheating at 260 °C for 10 s. The photon counts were normalized to the first peak for comparison.

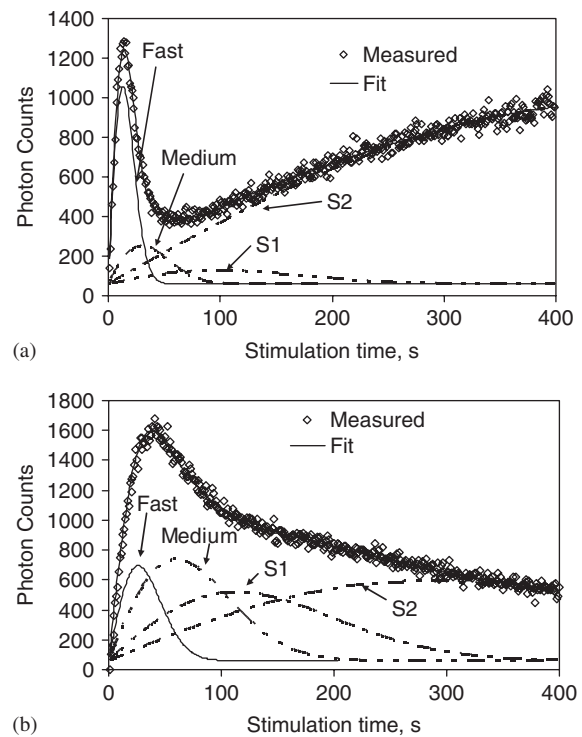


Fig. 2. (a) Curve fitting of a typical LM-OSL curve from quartz. (b) Curve fitting of a typical LM-OSL from K-feldspar. Four components, namely fast, medium, S1 and S2, are deduced by curve fitting based on Eq. (1) (see text).

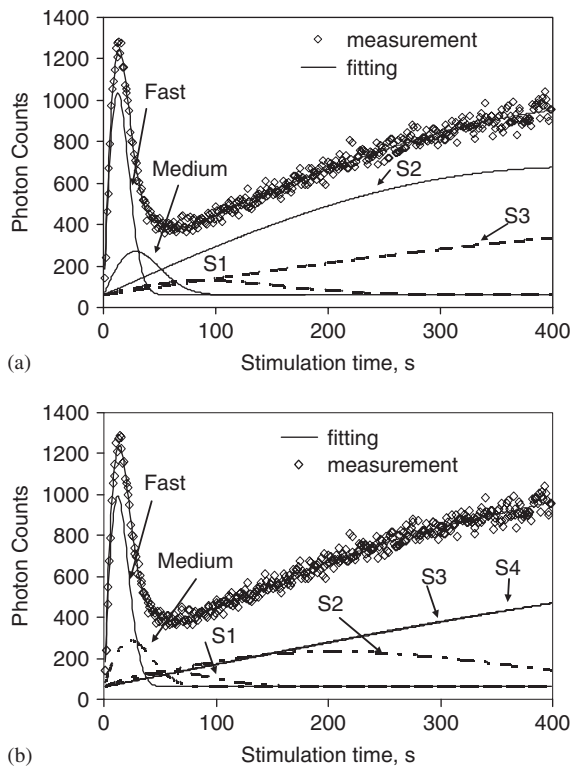


Fig. 3. (a) Curve fitting of typical LM-OSL from quartz using five components. (b) Curve fitting of the same LM-OSL curve as (a) using six components. The components, namely fast, medium, S1, S2, S3 and/or S4, are deduced based on Eq. (1). In (b), the curve S3 overlaps with curve S4 so that only five components are recognized in fitting with six components.

of the LM-OSL signals from quartz can be easily distinguished from that from K-feldspar according to the difference between the shapes of the LM-OSL curves and the peak positions, whereas the medium quartz and fast feldspar components are probably indistinguishable.

In contrast to the minimal changes in peak position of the fast component, the slow components for quartz differ significantly from sample to sample when four components were used in fitting the LM-OSL curves of 400 s. Due to the overlapping and slow detrapping rates of the slower components, their peaks cannot be separated clearly. This makes it difficult to determine the number of peaks in the slower part of the LM-OSL curve. In order to evaluate the influence of the number of peaks on curve fitting of the fast component, different numbers of peaks were used to fit the same LM-OSL curves from quartz, and are shown in Fig. 3(a) (five components) and (b) (six components). The components were named as fast, medium, S1, S2 and S3, etc. Comparing with the four components fitted in Fig. 2(a), the fast and medium components reach their maximum value at 13 and 29 s, respectively, indistinguishable from those found in Fig. 2(a). However, significant differences are found between the peak positions and magnitudes of the slower components, especially the slowest components (S2 and S3). The position of the fast components shows insignificant differences when different number of components are used in curve fitting.

Using a similar method of analysis for LM-OSL curves taken over 5000 s stimulation, and using different quartz samples, Jain et al. (2003) suggested that at least six components were needed to fit their curves; this large number of components is not justified in our data, presumably because of the much shorter stimulation time, and the correspondingly reduced total stimulation power delivered. Another possible reason for the large variation in the parameters of the slow components is that the assumption of first-order kinetics for all OSL components is not satisfied, particularly for the slow components. For instance, Chen and Leung (2003) suggested that the OSL decay curve can be described using a single stretched-exponential equation rather than a single exponential decay based on their theoretical and numerical simulation.

#### 4. Equivalent dose measurement of quartz using the fast component

##### 4.1. $D_e$ determination using quartz LM-OSL

In order to test the validity of  $D_e$  determination using the fast component of LM-OSL from quartz, sample Hld-1, the  $D_e$  value previously measured with a CW-OSL SAR technique was  $28.7 \pm 0.8$  ( $n = 9$ ) Gy, was selected. The LM-OSL  $D_e$  measurements were carried with a modified SAR dose procedure (Murray and Wintle, 2000) (Table 2). Backgrounds were subtracted from the LM-OSL signals by measuring blank discs under the same experimental conditions as discs with samples. This approach was employed as the measured background signal changes with stimulating power.

Four components were used to fit the LM-OSL curves. The test dose created LM-OSL curves were fitted using the same way. Only the fast component was used for sensitivity correction. The average of the first peak position  $t_m$  is  $13.0 \pm 1.0$  s for all of the LM-OSL curves. Using Eq. (2), a detrapping probability of  $2.4 \pm 0.2$  s<sup>-1</sup> corresponding to a photoionization cross-section of  $2.3 \pm 0.2 \times 10^{-17}$  cm<sup>2</sup> was obtained. This value agrees well with the value obtained by Jain et al. (2003) ( $2.32 \pm 0.16 \times 10^{-17}$  cm<sup>2</sup>) and is also very close to that obtained by Singarayer and Bailey (2004) ( $2.5 \pm 0.3 \times 10^{-17}$  cm<sup>2</sup>). The  $D_e$  values and recycling ratios obtained using fixed peak at 13 s for six aliquots are shown in Fig. 4. The average  $D_e$  value is  $30.3 \pm 2.6$  ( $n = 6$ ) Gy, similar to the value of  $28.7 \pm 0.8$  Gy obtained by CW-OSL. The average of the recycling ratios is  $0.98 \pm 0.06$ , suggesting that sensitivity change is well corrected. Similar results were found in samples of Rkz, Dgw-1 and Dzk.

In order to examine the influence of the first peak position variation on  $D_e$  calculation, two fitting approaches were tried on the same LM-OSL curves from one aliquot. One way is to fix the first peak position at 13 s when fitting the curves. The other is to allow peak position to vary. The regenerative dose growth curves from the same LM-OSL curves are shown in Fig. 5(a) and (b) for fixed and unfixed peak positions, respectively. It is clear that the growth curve derived from fixing the first peak position has good linearity and small deviations, whereas that obtained without fixing the first peak position has significant deviations from the best fit line. A poor recycling ratio was



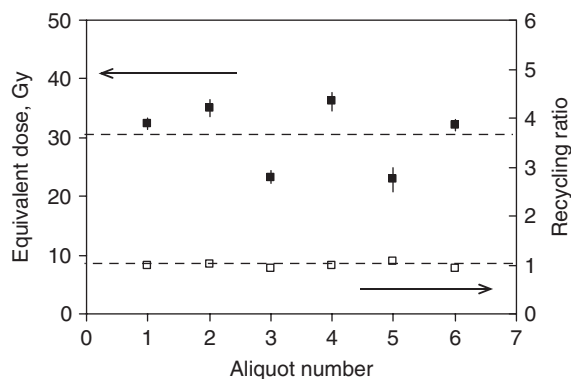


Fig. 4.  $D_e$  values (filled squares) and recycling ratios (open squares) obtained from six quartz aliquots of sample Hld-1 using SAR technique. The dashed lines represent the averages in two data sets.

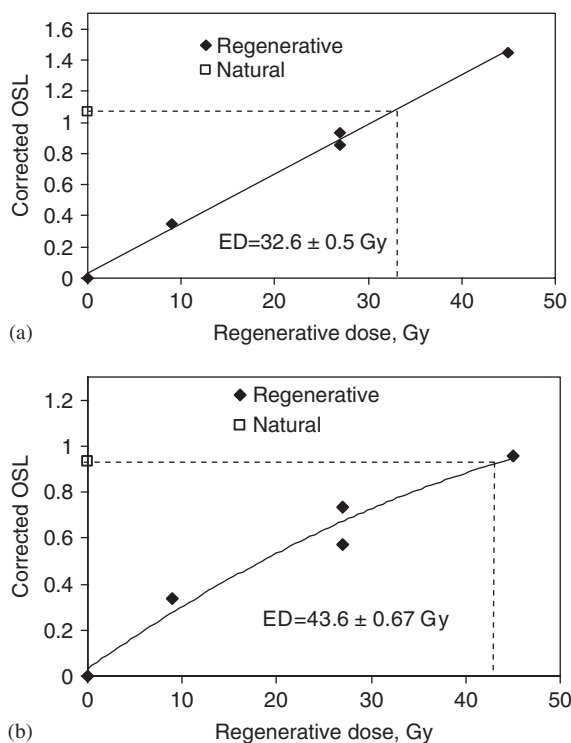


Fig. 5. (a) Regenerative dose growth curve using the fast component of quartz from sample Hld-1 with the first peak position fixed at 13 s. (b) Regenerative dose growth curve using the same data as (a) without fixing the first peak position. The solid lines are the best fitted line to the data.

found using the latter method, suggesting that the signal from the repeated regenerative dose was not recovered well without fixing the first peak position. It is concluded that fixing the peak position of the fast component gives improved reproducibility and the estimates of peak intensity are more accurate.

Because the choice of fixing the first peak position at 13 s is based on the average value obtained by fitting limited curves, it is necessary to examine the impact on the  $D_e$  estimation of choosing a given peak position. For one set of LM-OSL curves of one aliquot of sample Hld-1, the  $D_e$  value was calculated by fixing the first peak at 12, 13 and 14 s, respectively, when

fitting the LM-OSL curves. Note that these three peak positions are within the range of  $13.0 \pm 1.0$  s obtained by fitting a large number of curves from different sediment samples. The fitted area of the first peak, i.e. the fast component, and the corresponding  $D_e$  values are listed in Table 3. The  $D_e$  values are coincident with each other, despite differences among the fitted areas. When the first peak is fixed to either 11 or 15 s, no growth curve can be constructed because the LM-OSL curves could not be fitted properly. It is suggested that fixing the first peak position in this range affected each curve equally.

#### 4.2. Preheating plateau and dose recovery tests

With sample Hld-1, the  $D_e$  was determined using different preheat temperatures. The same SAR sequence as described in Section 4.1 was followed, except that a range of different preheat temperatures were used. A total of 28 aliquots in 7 groups were analyzed. Each of the 7 groups was preheated at a temperature (ranging from 160 to 280 °C) for 10 s. The curve fitting for LM-OSL was made by fixing the first peak position at 13 s. All the LM-OSL curves were fitted with four peaks. The  $D_e$  values and the recycling ratios obtained at each preheat temperature are shown in Fig. 6. The average  $D_e$  value for all preheat temperatures is  $30.2 \pm 0.8$  ( $n=28$ ) Gy, which agrees well with the value obtained using the CW-OSL technique. No dependence of  $D_e$  on preheating temperature was observed, suggesting that thermal transfer has no influence on the fast component of LM-OSL signals. Jain et al. (2003) also reached a similar conclusion. There is no significant deviation from unity in the recycling ratio except for data obtained at a preheat of 180 °C (0.82). The average recycling ratio is  $0.95 \pm 0.06$ , suggesting that the sensitivity correction is successful and independent on preheat temperature.

Another eight aliquots of quartz from sample Hld-1 were used for a ‘dose recovery’ test. They were bleached by the blue light from the Risø reader for 200 s at 125 °C to remove the natural signals and then given a dose of 27.0 Gy. The irradiated aliquots were then treated as the natural to determine the  $D_e$  using the LM-OSL SAR technique, with the same sequence and regenerative doses as described in Section 4.1 (Table 2). The LM-OSL curves from these aliquots were fitted by four peaks with the first peak position being fixed at 13 s. The average  $D_e$  value is  $26.3 \pm 0.7$  Gy, demonstrating that it is possible to accurately recover a laboratory dose of 27 Gy.

#### 5. Equivalent dose measurement of an unseparated sample

Although quartz grains can normally be extracted from sediment after heavy liquid separation and HF acid etching, feldspar may still be present if it appears in the form of inclusions (Fragoulis and Readhead, 1991). The presence of such feldspar grains can give rise to incorrect results in  $D_e$  determination due to anomalous fading (Huntley and Lamothe, 2001) and other effects (Wallinga et al., 2000). Hence, it is important to ensure the OSL signals that are analyzed originate only from quartz.

The feasibility of distinguishing the fast component of quartz from the mixed signals of feldspar and quartz was explored

Table 3

Results of curve fitting by fixing the first peak position at 12, 13 and 14 s

First peak position (s)	Area under the first peak (counts)	Equivalent dose (Gy)	Recycling ratio
12	4709	$28.4 \pm 0.41$	0.93
13	5449	$28.3 \pm 0.38$	0.96
14	5933	$28.8 \pm 0.37$	0.98

The fitting was carried out using the same set of LM-OSL curves from a quartz aliquot of sample Hld-1. The areas are the fitted area of natural signals.

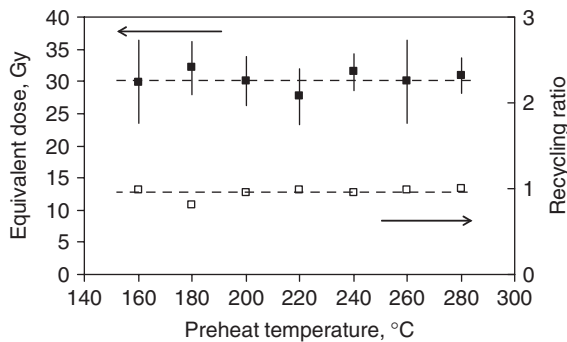


Fig. 6. Preheat plateau for quartz from Hld-1. The  $D_e$  value from each temperature is the average of four aliquots using the fast component and shown as filled squares. The average over all temperatures,  $30.2 \pm 0.8$  Gy, is shown as dashed line. The open squares represent the recycling ratio of each preheat temperature. The average of all recycling ratio is  $0.95 \pm 0.06$  and shown as a dashed line.

by deconvoluting the LM-OSL signals from an unseparated sample. There is a significant difference in the detrapping rates of the fast components of the LM-OSL signals of quartz and feldspar. For 400 s stimulation, the quartz fast component peaks at around 13 s, whereas for feldspar, the fast component peaks at around 33 s. Sample Hld-1 was chosen for study. The sample was treated as described previously, but without heavy liquid separation and HF etching. Because of the overwhelming OSL signals from feldspar compared to the quartz signals, infrared (IR) bleaching for 100 s at room temperature was used prior to each LM-OSL measurement to reduce the magnitude of the OSL signals from feldspar. Duller and Bøtter-Jensen (1993) demonstrated that using IR at room temperature reduced the blue-stimulated signal from coarse-grained feldspars, and Jain and Singhvi (2001) investigated the effect of IR on the OSL signal when the feldspars were held at different temperatures. Wallinga et al. (2002) built on these studies and demonstrated the effect of IR stimulation on LM-OSL signals from both quartz and feldspars. A typical natural LM-OSL curve following IR bleaching for 100 s is shown in Fig. 7(a). At least four peaks were necessary to fit the signals (shown as 1, 2, 3 and 4 in Fig. 7(a)). A sharp peak around 13 s was observed and it was followed by a broad peak of overlapping signals. Based on the peak time, the sharp peak is associated with the fast component of quartz. For LM-OSL curve without IR bleaching (Fig. 7(b)), no peak around 13 s can be identified; thus IR bleaching is necessary to reduce the interference of feldspar signals with the fast component of quartz OSL obtained under blue stimulation.

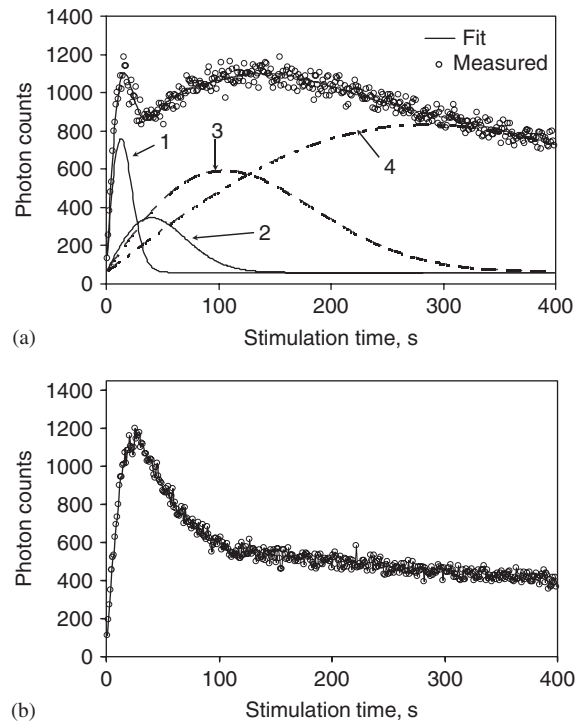


Fig. 7. Natural LM-OSL curve from the unseparated grains of sample Hld-1. (a) The LM-OSL curve measured following IR bleach for 100 s. Four peaks, 1, 2, 3 and 4, are deduced by curve fitting based on Eq. (1) (see text). (b) The LM-OSL curve measured without IR bleach. The LM-OSL was measured at 125 °C for 400 s after preheating at 260 °C for 10 s.

$D_e$  determination for unseparated material from sample Hld-1 was achieved using the fast component of the LM-OSL signals with the position of the first peak fixed in the curve fitting. Six aliquots were measured using the same SAR technique as described in Section 4, with the addition of an IR bleach prior to each LM-OSL measurement. The LM-OSL curves were fitted with four peaks and the first peak position was fixed at 13 s. The  $D_e$  value, obtained from the first peak, and recycling ratio from each aliquot are shown in Fig. 8. The average  $D_e$  value obtained is  $28.8 \pm 1.0$  ( $n = 6$ ) Gy, agreeing well with the previous result of  $30.2 \pm 0.8$  Gy from pure quartz. There is also insignificant deviation in the recycling ratios from 1, suggesting that the sensitivity correction is successful. This result indicates that the fast component of quartz signals can be separated successfully.

In order to explore the interferences in curve fitting between the signals from feldspar and quartz, the second peak (peak 2, medium component) was also used to calculate the  $D_e$  value.

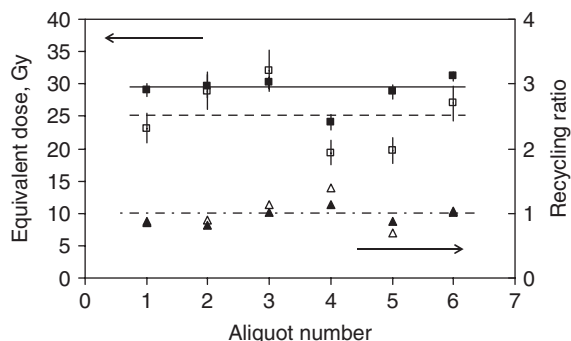


Fig. 8.  $D_e$  values obtained from unseparated grains of sample Hld-1. The values of  $D_e$  estimated from the area of peak 1 are shown as filled squares and the average value is shown as the solid line. The  $D_e$  values estimated from the area of peak 2 are shown as open squares and their average is shown as the dashed line. The filled triangles represent the recycling ratio when using peak 1 to estimate the equivalent dose. The dash dot is the average of recycling ratios. The open triangles represent the recycling ratio when using peak 2 to estimate the equivalent dose.

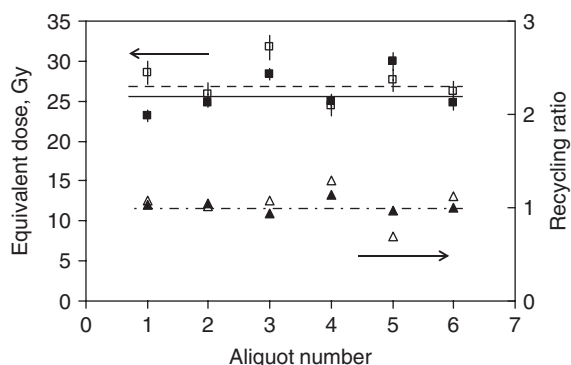


Fig. 9. Recovered doses for the unseparated sample from Hld-1. The  $D_e$  values estimated using peak 1 are shown as filled squares with the average value shown as solid line. The recycling ratios when using peak 1 for  $D_e$  calculation are shown as filled triangles with the average value shown as dash dot. The open squares represent the equivalent dose estimated from peak 2 and their average is shown as dashed line. The open triangles represent the recycling ratio when using peak 2 to estimate the equivalent dose.

As shown as open squares in Fig. 8, the average  $D_e$  value given from peak 2 is  $25.0 \pm 2.1$  ( $n = 6$ ) Gy, smaller than the value of  $28.8 \pm 1.0$  Gy given by peak 1, but higher than the value of  $21.8 \pm 1.2$  ( $n = 6$ ) Gy obtained from the K-feldspar fraction measured with the SAR CW-OSL technique. Using this second peak, the data showed large standard error in  $D_e$  values and poor recycling ratios are found with this component. These results demonstrate that peak 2 of unseparated samples is significantly affected by the mixing of signals from feldspar and quartz, and uncertainty of peak number and positions.

A 'dose recovery' test was also applied to unseparated material from Hld-1. The  $D_e$  determination procedures were similar to those described in Section 4.2. The  $D_e$  values estimated using peaks 1 and 2 are shown in Fig. 9. The average  $D_e$  value given by peak 1 was  $26.0 \pm 1.1$  ( $n = 6$ ) Gy, demonstrating a good recovery of the known laboratory dose of 27.0 Gy. A similar average  $D_e$  value of  $27.4 \pm 1.0$  ( $n = 6$ ) Gy was given by peak 2. The similarity in the  $D_e$  values from peaks 1 and 2

is presumably due to the short delay between irradiation and measurement so that any anomalous fading of feldspar signals in peak 2 is then negligible. The dose recovery measurement is not affected, though peak 2 contains signals from quartz and feldspar.

## 6. Discussion

There are both advantages and disadvantages for dose determination using the method described earlier in the paper. Besides being able to be applied to quartz samples that are contaminated by feldspar inclusions, it can also be used for samples of mixed minerals for which quartz separation is complicated, e.g. fine grained polyminerals. It may also be advantageous for quartz samples which show a weak fast OSL component (e.g. Choi et al., 2003). Since the fast quartz component is also more rapidly bleached in nature, dating using LM-OSL would also be advantageous for poorly bleached samples, as previously suggested by Larsen et al. (2000). The main disadvantage in applying the method is that the procedures are time consuming, both because each LM-OSL curve needs to be fitted and because the data collection time is longer than for CW-OSL measurements. The latter problem could be overcome by measuring CW-OSL signals with a non-constant time interval for data collection. This results in peaks that can be subjected to the same analysis (Poolton et al., 2003). In addition, if one was also interested in the slower components, it should be pointed out that the signal-to-noise ratio decreases with increased stimulation time.

## 7. Conclusions

From this study of the LM-OSL signals from quartz, K-feldspar and unseparated fractions of several sediment samples of different ages and geological settings, the following conclusions can be made:

Firstly, quartz LM-OSL curves exhibit a common fast component, which differs significantly from feldspars. This fast component from quartz shows a sharp peak around 13 s in LM-OSL curves obtained with maximum illumination of 400 s by blue light from LEDs with maximum power of 50 mW/cm<sup>2</sup>. K-feldspar has a broad peak, occurring at around 35 s.

Secondly, the quartz and feldspar fast components can be separated from LM-OSL signals by curve fitting. At least four components are necessary to describe both the LM-OSL signals from quartz and feldspar. The number of peaks used in curve fitting has an insignificant effect on the parameters of the fast component, but has a significant influence on slower components.

Thirdly, equivalent dose determination by the SAR technique is achieved by using only the fast component of quartz LM-OSL signals separated by curve fitting. For purified quartz, the  $D_e$  values obtained using the LM-OSL technique agree well with those obtained from the CW-OSL technique. Fixing the position of the first peak reduces the error in curve fitting. Changing the position of this fixed peak in the range from 12 to 14 s does not affect the  $D_e$  determination. The results

from such a modified SAR LM-OSL technique can be checked using ‘preheat plateau’, ‘recycling ratio’ and ‘dose recovery’ tests.

Fourthly, the fast component from quartz can be successfully separated from samples without mineral separation, and used for  $D_e$  determination using the SAR technique. It is necessary to add an infrared bleach prior to each LM-OSL measurement to reduce the overwhelming OSL signal from feldspars. The  $D_e$  given by using the first peak, corresponding to the quartz fast component, of the LM-OSL signals from unseparated sample agrees with that obtained from pure quartz.

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## References

- Aitken, M.J., 1998. *An Introduction to Optical Dating*. Oxford University Press, Oxford.
- Bailey, R.M., Smith, B.W., Rhodes, E.J., 1997. Partial bleaching and the decay form characteristics of quartz OSL. *Radiat. Meas.* 27, 123–136.
- Bøtter-Jensen, L., Duller, G.A.T., Murray, A.S., Banerjee, D., 1999a. Blue light emitting diodes for optical stimulation of quartz in retrospective dosimetry and dating. *Radiat. Prot. Dosim.* 84, 335–340.
- Bøtter-Jensen, L., Mejdahl, V., Murray, A.S., 1999b. New light on OSL. *Quat. Sci. Rev.* 18, 303–309.
- Bulur, E., 1996. An alternative technique for optically stimulated luminescence (OSL) experiment. *Radiat. Meas.* 26, 701–709.
- Bulur, E., Bøtter-Jensen, L., Murray, A.S., 2000. Optically stimulated luminescence from quartz measured using the linear modulation technique. *Radiat. Meas.* 32, 407–411.
- Chen, R., Leung, P.L., 2003. The decay of OSL signals as stretched-exponential functions. *Radiat. Meas.* 37, 519–526.
- Chen, R., McKeever, S.W.S., 1997. *Theory of Thermoluminescence and Related Phenomena*. World Scientific, Singapore.
- Choi, J.H., Murray, A.S., Cheong, C.S., Hong, D.G., Chang, H.W., 2003. The resolution of stratigraphic inconsistency in the luminescence ages of marine terrace sediments from Korea. *Quat. Sci. Rev.* 22, 1201–1206.
- Duller, G.A.T., Bøtter-Jensen, L., 1993. Luminescence from potassium feldspars stimulated by infrared and green light. *Radiat. Prot. Dosim.* 47, 683–688.
- Fragoulis, D.V., Readhead, M.L., 1991. Feldspar inclusions and the enhancement of thermoluminescence in quartz grains. *Nucl. Tracks Radiat. Meas.* 18, 291–296.
- Huntley, D.J., Lamothe, M., 2001. Ubiquity of anomalous fading in K-feldspars and the measurement and correction for it in optical dating. *Can. J. Earth Sci.* 38, 1093–1106.
- Huntley, D.J., Godfrey-Smith, D.I., Thewalt, M.L.W., 1985. Optical dating of sediments. *Nature* 313, 105–107.
- Jain, M., Singhvi, A.K., 2001. Limits to the depletion of green-blue light stimulated luminescence in feldspars: implications for quartz dating. *Radiat. Meas.* 33, 883–892.
- Jain, M., Murray, A.S., Bøtter-Jensen, L., 2003. Characterisation of blue-light stimulated luminescence components in different quartz samples: implications for dose measurement. *Radiat. Meas.* 37, 441–449.
- Jain, M., Murray, A.S., Bøtter-Jensen, L., Wintle, A.G., 2005. A single-aliquot regenerative-dose method based on IR(1.49 eV) bleaching of the fast OSL component in quartz. *Radiat. Meas.* 39, 309–318.
- Kuhns, C.K., Larsen, N.A., McKeever, S.W.S., 2000. Characteristics of LM-OSL from several different types of quartz. *Radiat. Meas.* 32, 413–418.
- Larsen, N.A., Bulur, E., Bøtter-Jensen, L., McKeever, S.W.S., 2000. Use of the LM-OSL technique for the detection of partial bleaching in quartz. *Radiat. Meas.* 32, 419–425.
- Li, S.H., 2001. Identification of well-bleached grains in the optical dating of quartz. *Quat. Sci. Rev.* 20, 1365–1370.
- Murray, A.S., Roberts, R.G., 1998. Measurement of the equivalent dose in quartz using a regenerative-dose single-aliquot protocol. *Radiat. Meas.* 29, 503–515.
- Murray, A.S., Wintle, A.G., 1998. Factors controlling the shape of the OSL decay curve in quartz. *Radiat. Meas.* 29, 65–79.
- Murray, A.S., Wintle, A.G., 2000. Luminescence dating of quartz using an improved single-aliquot regenerative-dose protocol. *Radiat. Meas.* 32, 57–73.
- Poolton, N.R.J., Bøtter-Jensen, L., Andersen, C.E., Jain, M., Murray, A.S., Malins, A.E.R., Quinn, F.M., 2003. Measuring modulated luminescence using non-modulated stimulation: ramping the sample period. *Radiat. Meas.* 32, 639–645.
- Singarayer, J.S., Bailey, R.M., 2003. Further investigations of the quartz optically stimulated luminescence components using linear modulation. *Radiat. Meas.* 37, 451–458.
- Singarayer, J.S., Bailey, R.M., 2004. Component-resolved bleaching spectra of quartz optically stimulated luminescence: preliminary results and implications for dating. *Radiat. Meas.* 38, 111–118.
- Smith, B.W., Rhodes, E.J., 1994. Charge movements in quartz and their relevance to optical dating. *Radiat. Meas.* 23, 593–600.
- Tsukamoto, S., Rink, W.J., Watanuki, T., 2003. OSL of tephric loess and volcanic quartz in Japan and an alternative procedure for estimation  $D_e$  from a fast component. *Radiat. Meas.* 37, 459–465.
- Wallinga, J., Murray, A., Wintle, A., 2000. The single-aliquot regenerative-dose (SAR) protocol applied to coarse-grain feldspar. *Radiat. Meas.* 32, 529–533.
- Wallinga, J., Murray, A.S., Bøtter-Jensen, L., 2002. Measurement of the dose in quartz in the presence of feldspar contamination. *Radiat. Prot. Dosim.* 101, 367–370.
- Watanuki, T., 2002. Chronological study of loess palaeosol by improved method of luminescence dating and application to reconstruct past environmental changes. Ph.D. Thesis, Tokyo Metropolitan University, unpublished.