

# Fractional bleaching of potassium feldspar from sediments and its role in equivalent dose determination

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## Introduction

Several methods have been developed to determine the equivalent dose (ED) of sediments that might not have been well-bleached during deposition (Berger, 1984; 1985; Berger et al., 1987; Huntley, 1985; Mejdahl, 1985; 1988; Wintle and Huntley, 1980). These all involve a short laboratory bleach which is thought to have less effect on the TL signal than that caused by light exposure prior to deposition. In a preliminary study on the thermoluminescence of eolian sands from the Lutterzand area in the eastern part of The Netherlands, Dijkmans et al. (1988) used the plateau method with additive dose pioneered by Mejdahl (1986; 1988). This method was applied to potassium feldspar grains separated from an eolian sand sample (Younger Cover Sand I). A short plateau from about 340 to 400 °C was obtained using residual signals resulting from 20 to 40 minutes exposure to a SOL 2 sunlamp. Shorter bleaching times resulted in decreasing dose values (D) with increasing glow curve temperatures, whereas longer bleaching times increased those values. The choice of the right bleaching time and therefore of the correct ED was not straightforward.

In a subsequent study twelve samples from the same site were analysed with the automated Risø TL reader using potassium feldspar separates (Dijkmans and Wintle, in prep.). The additive dose-plateau method was found to give poor plateaux in 75% of the cases (fig. 1). Several possible causes of poor plateaux are listed in Appendix A. The calculated dose (D) increased with bleaching time. For at least 50% of the samples no bleaching time could be found which would extend the plateaux to temperatures above 390 °C as recommended by Mejdahl (1988). Therefore another approach was used, based on method (a) of Wintle and Huntley (1980). The basis of this approach is reiterated here and the experimental data obtained for the set of samples were analysed using the approach of Debenham (1985).

## Method of ED determination: defining a factor f

For sediments the natural TL-intensity, I, may be separated into an unbleachable part, I<sub>0</sub>, (residual value after a long bleach) and a bleachable part, I<sub>d</sub>, (TL due to

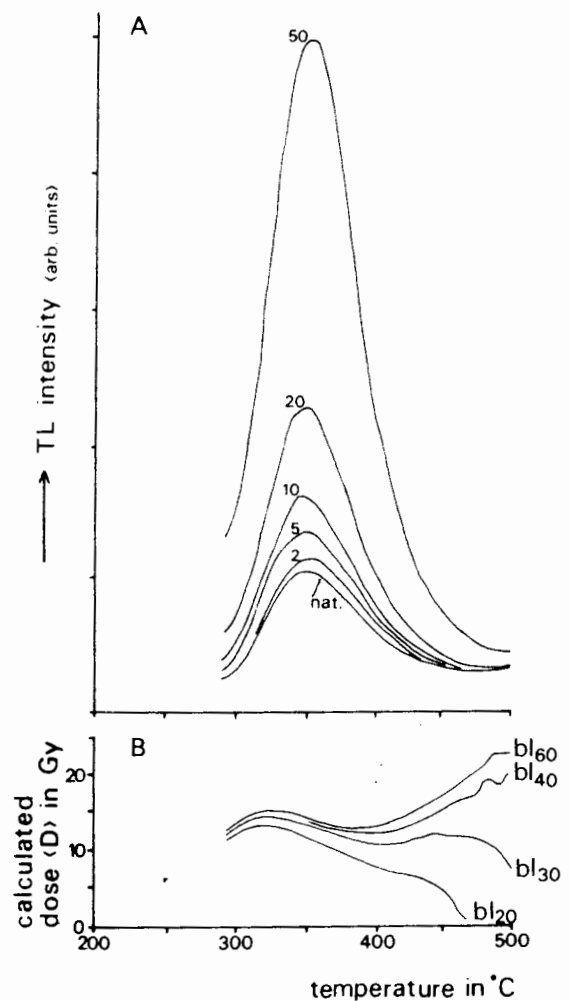


Figure 1

A) Glow curve of natural TL and five glow curves of additive dose, given in minutes of  $\beta$ -irradiation (black body has not been subtracted).

B) Dose (D) versus temperature plots for four different bleaching times (60, 40, 30, and 20 minutes, SOL-2 lamp) for sample L11. The material used is a potassium feldspar separate; preheat 290 °C for 10 sec.; heating rate 10 °C/s; UG-11 filter. Each glow curve represents the mean of four measurements.

radiation dose since deposition) (Wintle and Huntley, 1980):

$$I = I_0 + I_d$$

One of the original methods for determination of the ED for deep-sea sediments was introduced by Wintle and Huntley as their method (a). They suggested that the effect of a relatively short exposure to light would result in a bleached signal ( $I_{bl}$ ):

$$I_{bl} = I_0 + f.I_d$$

where  $f$  is the fraction of radiation induced TL signal left after that light exposure. The unbleachable component would, of course, be unaffected. Wintle and Huntley (1980) then determined the gamma irradiation  $D$  that was needed to replicate the natural TL level. Hence:

$$I = I_0 + I_d = I_0 + f.I_d + I_D$$

where  $I_D$  is the signal due to  $D$ . This can be rewritten as

$$I_d = f.I_d + I_D$$

or

$$I_d(1 - f) = I_D$$

Assuming the TL sensitivity ( $\chi$ ) (TL per unit radiation dose) to be a constant, the equivalent dose (ED) can be expressed as

$$ED = \frac{I_d}{\chi}$$

and in a similar way

$$ED = \frac{I_D}{\chi}$$

but  $I_D = I_d(1 - f)$ , and hence

$$D = \frac{I_d(1 - f)}{\chi}$$

Combining these equations gives

$$D = \frac{I_d}{\chi}(1 - f) = ED(1 - f)$$

or

$$ED = D(1 - f)^{-1} \tag{1}$$

To determine the ED,  $f$  must also be determined experimentally. This is done by measuring the TL ( $I_{SB}$ ) left in a gamma irradiated sample after the same short bleaching time. This is then compared with the TL for the gamma irradiated sample ( $I_{B+\gamma}$ ). In each case the gamma dose is administered after the sample has been bleached for a long time to reach a base TL level  $I_B$ . This may be close to  $I_0$ , but is not necessarily identical with it. Hence  $f$  is the fraction of gamma induced TL left after the lamp exposure i.e.

$$f = \frac{I_{SB} - I_B}{I_{B+\gamma} - I_B}$$

This procedure is shown schematically in fig. 2, where  $I_{SB}$  is presented in four bleaching times (20, 30, 40 and 60 min).

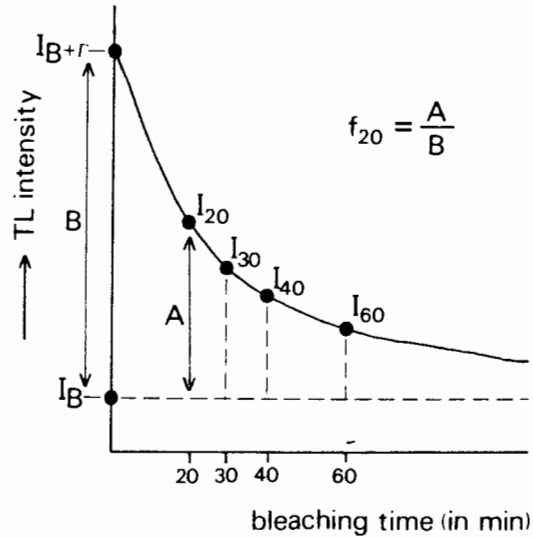


Figure 2  
Schematic diagram showing experimental procedure to determine  $f$ .

Hence, by using several bleaching times different values of  $f$  are obtained. At the same time different values of  $D$  will be obtained when the natural TL signal is regenerated after the different bleaching times. However, using equation (1) the same value of ED should be obtained for each data set!

This concept can be taken further, as first suggested by Debenham (1985) for the data he obtained by applying the regeneration method using a 16 hour sunlamp bleach and assuming that  $I_0 = I_B$ . However, laboratory lamp exposures do not usually attain the residual level at deposition (i.e.  $I_0 = I_B$ ). Hence one can write

$$I_B = I_0 + f.I_d$$

but by definition

$$I = I_0 + I_d$$

Substituting for  $I_d$ :

$$I_B = I_0 + f(I - I_0) = f.I + (1 - f)I_0 \tag{2}$$

A plot of  $I_B$  versus  $I$  would thus have a slope  $f$  and an intercept on the y-axis of  $(1 - f)I_0$ . To demonstrate the fact that  $f=0$  Debenham (1985) plotted the value of the residual level,  $I_B$ , (in terms of their equivalent dose) after a 16 hour bleach against natural intensities,  $I$ , for the same samples. He used fine grained samples from four different sites covering a timespan from 10 to 700

ka and obtained the slopes of this plot at two particular temperatures. *f* was about 0.05 for the TL at 300 - 310 °C - an indication that 5% of the bleachable TL in the fine grain samples survived the 16 hour sunlamp exposure. The same approach has been applied in the present paper to the 12 eolian sand samples from the Lutterzand.

**Experimental procedure and results**

Twenty mg weighed samples were measured using a UG-11 filter and employing a pre-heat of 10 seconds at 290 °C as originally recommended by Mejdahl and Winther-Nielsen (1982). Typical glow curves are given in fig. 1A.

Because of the problems with obtaining reproducible plots of dose, *D*, versus glow curve temperatures (as mentioned earlier), the area beneath the peak, defined by the half height, was used. The procedure differs from that of Wintle and Huntley (1980) in that additive dose was used instead of regeneration. In table 1A values of *D* are given for four different bleaching times (20, 30, 40, and 60 minutes) for three samples which were examined in detail. It can be seen that *D* increases systematically with increasing bleaching time.

Further experiments were carried out on these samples in order to calculate the factor *f* (again using the temperature range between the half peak height), as defined in the previous section. Potassium feldspar separates of the samples were bleached for 5 hours using the SOL 2 lamp. A small part of each sample was measured (*I<sub>B</sub>* in fig. 2) and the rest was given a gamma dose of about 39 Gy. Part of this was measured (*I<sub>B+γ</sub>* in fig. 2). Subsamples of the irradiated material were exposed to the lamp for 20, 30, 40, and 60 minutes respectively and subsequently measured (fig. 2). For these measurements 20mg samples were used with 4 samples for each data point. Second glow normalization was used to improve the reproducibility.

Glow curves from several stages of the experiment are given for one sample in fig. 3. The values of *f* are given in table 2 and the resulting values of ED, obtained from putting *D* and *f* in equation (1), in table 1B for the three samples.

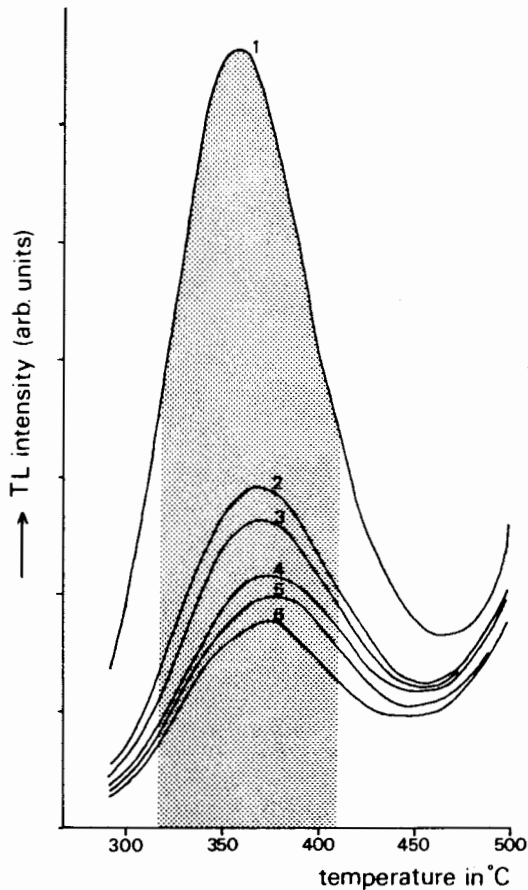
Besides obtaining *f* for the integral about the peak, *f* was also obtained as a function of temperature. *f*<sub>20</sub> versus temperature is plotted in fig. 4A for the three samples and for the four bleaching times for one of the samples in fig. 4B. For all three samples the values of *f* increases with increasing glow curve temperature. This effect was also reported by Wintle and Huntley (1980) who concluded that it was brought about by TL sensitivity changes which occur after exposure to the lamp and to a different extent for different glow curve temperatures. Another reason for the variation of *f* with temperature could be the particular bleaching response at different temperatures for the lamp used. This may be different from sunlight and requires further investigation.

**Table 1.** Values of *D* and ED (in Gy) obtained for three samples for each of four bleaching times. The ED is calculated both by using: B) mean *f* values for individual samples, C) values of *D* and *f* both obtained as a function of temperature, and D) graphically-derived *f* values as shown in table 2. A preliminary dose rate calibration of 1.64 Gy/min was used.

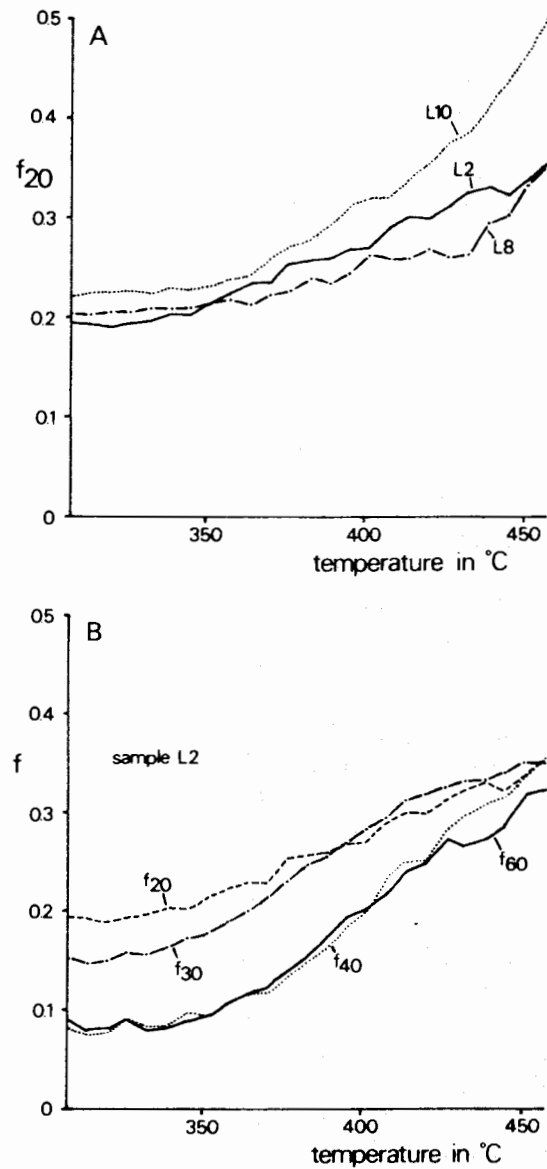
	Sample		
	L2	L8	L10
A) Dose ( <i>D</i> ) Obtained by extrapolation to <i>I<sub>B</sub></i>			
<i>D</i> <sub>20</sub>	12.32	18.40	12.62
<i>D</i> <sub>30</sub>	12.89	19.50	13.37
<i>D</i> <sub>40</sub>	13.69	20.37	13.89
<i>D</i> <sub>60</sub>	14.33	20.93	14.45
mean ED	13.31	19.80	13.58
± 1σ	± 0.88	± 1.10	± 0.78
B) Equivalent dose obtained from <i>D</i> using <i>f</i> values of individual samples.			
ED <sub>20</sub>	16.02	23.75	16.17
ED <sub>30</sub>	15.88	23.85	16.74
ED <sub>40</sub>	15.76	24.11	16.68
ED <sub>60</sub>	16.09	23.65	17.06
mean ED	15.94	23.84	16.66
± 1σ	± 0.15	± 0.20	± 0.37
C) Equivalent dose obtained from individual values of <i>D</i> and <i>f</i> obtained as a function of temperature (fig. 5)			
ED <sub>20</sub>	15.61	23.42	16.60
ED <sub>30</sub>	15.73		
ED <sub>40</sub>	15.27		
ED <sub>60</sub>	16.04		
mean ED	15.66		
± 1σ	± 0.32		
D) Equivalent dose obtained from <i>D</i> using graphically derived <i>f</i> values (fig. 6)			
ED <sub>20</sub>	16.40	24.50	16.79
ED <sub>30</sub>	16.50	24.97	17.12
ED <sub>40</sub>	16.88	25.12	17.13
ED <sub>60</sub>	17.10	24.98	17.24
mean ED	16.72	24.89	17.07
± 1σ	± 0.33	± 0.27	± 0.19

**Table 2.** *f* factor for four bleaching times of the three samples. Each data point is represented by four individual measurements and one standard deviation is indicated. *f* as calculated by linear regression (see fig. 5) using the data of twelve samples are also shown.

Sample	<i>f</i> factor for four bleaching times $\pm 1\sigma$			
	<i>f</i> <sub>20</sub>	<i>f</i> <sub>30</sub>	<i>f</i> <sub>40</sub>	<i>f</i> <sub>60</sub>
L2	0.231 $\pm$ 0.018	0.188 $\pm$ 0.010	0.131 $\pm$ 0.018	0.109 $\pm$ 0.007
L8	0.225 $\pm$ 0.018	0.182 $\pm$ 0.016	0.155 $\pm$ 0.014	0.115 $\pm$ 0.010
L10	0.220 $\pm$ 0.017	0.202 $\pm$ 0.013	0.167 $\pm$ 0.013	0.153 $\pm$ 0.011
L1 - L12 (linear regression)	0.249 $\pm$ 0.022	0.219 $\pm$ 0.037	0.189 $\pm$ 0.024	0.162 $\pm$ 0.026



**Figure 3**  
Glow of sample L2 at various stages of the experiment to determine *f*. 1)  $I_B + \gamma$ ; 2)  $I_B + \gamma + bI_{20}$ ; 3)  $I_B + \gamma + bI_{30}$ ; 4)  $I_B + \gamma + bI_{40}$ ; 5)  $I_B + \gamma + bI_{60}$ ; 6)  $I_B$ . Only the relevant part of the temperature range is shown. The shaded part represents the area between the half peak height. Preheat 290 °C for 10 sec.; heating rate 10 °C/s; UG-11 filter.



**Figure 4.**  
A: The fraction of gamma induced TL, *f*, left after 20 minutes exposure to the SO1 2 lamp as a function of temperature for the samples L2, L8 and L10.  
B: *f* as a function of temperature for four different bleaching times (sample L2).

**Implication of the factor *f* for equivalent dose determination**

The values of ED obtained for the different bleaching times (as shown in table 1B) are in good agreement with each other for all of the samples used in the experiment. This is exemplified by the low standard deviation of the mean of the four ED values for each sample, as compared with the standard deviation of the mean *D* (cf. table 1A and B). Moreover there is no systematic increase (or decrease) in ED with bleaching time. For these reasons it can be argued that these values of the ED are the correct values to be used for TL fading of these samples. Combining values of *D* and *f*, both obtained as a function of temperature, in equation (1) permits the calculation of ED as a function of temperature. Values of ED are shown for four bleaching times for sample L2 alongside the values of *D* (fig. 5). The mean values of ED for the temperature range 308-392 °C (half peak height) are given in table 1C, as are the results for the 20 minutes bleach for L8 and L10.

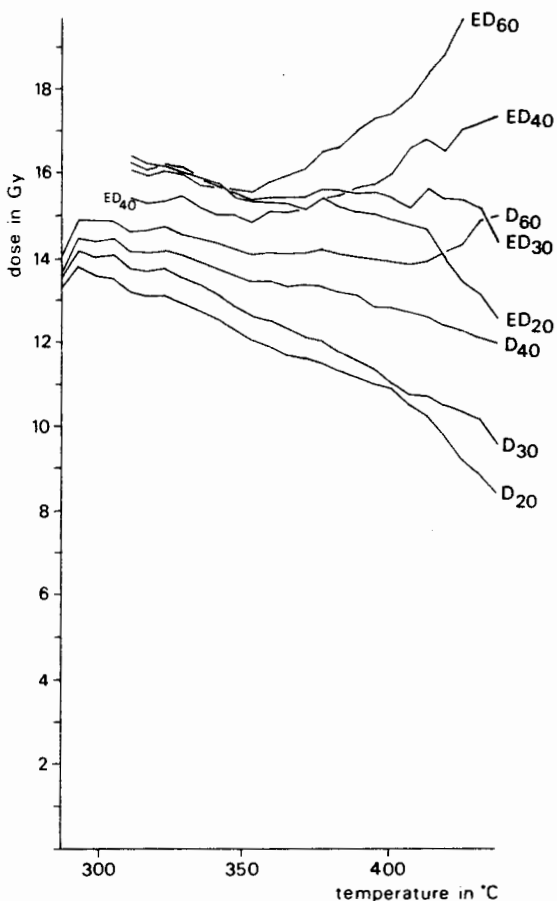


Figure 5. Dose (*D*) and equivalent dose (*ED*) versus temperature plot for four different bleaching times (in minutes) of sample L2. The values of the *ED* are calculated using both *D* and *f* obtained as a function of temperature. A preliminary dose rate calibration of 1.64 Gy/min was used.

The data also suggest that none of these bleaching times were sufficient to match the residual level at deposition. This point of view can also be supported by following the same approach as Debenham (1985), which was explained in detail in a previous section. The absolute value of the natural TL after a short bleaching  $I_{bl}$  is given by equation (2): i.e.

$$I_{bl} = I_0 + f.I_d$$

and hence

$$I_{bl} = f.I + (1 - f)I_0.$$

Therefore a plot of  $I_{bl}$  for a single bleaching time versus the natural TL intensity, *I*, will have a slope *f*.

Plots of  $I_{bl}$  versus *I* were obtained for the twelve eolian sand samples from the Lutterzand area which range in age from a few hundred to about fifteen years (fig. 6). Four plots were obtained, one for each bleaching time. Individual data points are given for both the 20 and 60 minutes bleaching times and the straight lines represent linear regression for the four data sets.

The *f* values obtained in this way can be compared with the *f* values obtained for the individual samples L2, L8 and L12 as shown in table 2. There is good agreement between the individual values of *f* and those obtained from fig. 6 (most values agree within one standard deviation) considering no normalization based on absolute TL sensitivity between the twelve different samples was employed. The data imply that potassium feldspars from The Lutterzand area are very similar, in spite of their different ages. This agrees with the geological evidence. They also imply that the values of *f* obtained from the linear regression using the twelve samples could be applied to the values of *D* obtained for any eolian sand samples in the Lutterzand area to obtain values of the ED. Using this approach for the three samples used in the experiment, the values of the ED obtained for the different bleaching times (table 1D) also come together without an increasing or decreasing trend with bleaching time. Moreover they are in agreement with the ED values obtained from individual *f* values (cf. table 1B and 1D). This would justify the use of the graphically derived *f* values for a group of homogeneous samples in at least a limited geographical area. This approach would involve a considerable reduction in laboratory and analytical work (and sample material) compared with calculating individual *f* values for each sample to be dated. One must take into consideration that to obtain a reliable value of *f* from linear regression, a data set is needed with a large variety of natural TL intensities (i.e. use both young and older samples).

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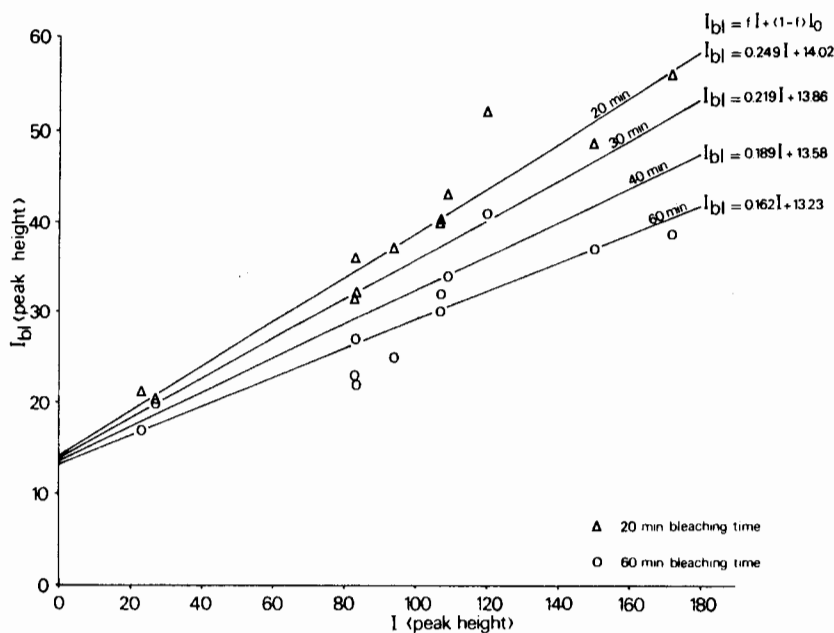


Figure 6. Levels of TL remaining after specific SOL 2 lamp exposure times ( $I_b$ ) plotted against the natural TL intensities ( $I$ ), for 12 sand samples from the Lutterzand. Values represent arbitrary units of the peak height at about 360 °C.

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## Appendix A: Possible causes of poor plateaux in the additive dose method

1. Non-linear growth of TL signal - not found to be a problem for these samples when linear and exponential fits were compared.
2. At higher glow curve temperatures the TL signals are smaller because of lower sensitivity (fig. 1), and hence there is a greater potential for error.
3. Second order kinetic behaviour causing peak shifts resulting in supralinearity (Chen et al., 1983; Levy, 1982).
4. Inappropriate choice of light source (SOL-2 lamp) as proposed by Mejdahl (1988), but this is unlikely in our experience (Dijkmans et al., 1988, fig. 7).

## PR. Reviewer's comments (Helen Rendell)

This paper sets out to explore the logical implications of Wintle and Huntley's (1980) method (a) for ED determination. In this method both D, the gamma dose required to match exactly the natural TL, and f, the fraction of radiation induced TL left after a short exposure to light, are determined for a range of glow-curve temperatures. Some of the underlying assumptions, notably that TL sensitivity to dose will be unaffected by bleaching, are inevitably problematic. One might also suspect that the value of f obtained will be a function of both bleaching spectrum and the wavelength range of TL emissions monitored. Nevertheless the results quoted should encourage other workers to explore this method of ED determination.

One small point on experimental method: the heating rate claimed (10 °C/s). Am I alone in being worried by the implications of such (apparently) rapid heating? Perhaps a plot of  $\ln(T^2/\beta)$  against  $1/T^*$  for this material would help to remove this source of concern!