

Test data for exponential fits

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Introduction

To fit exponential models to TL dose-response curves, several TL dating research groups are presently using different fitting routines, some written in-house from basic mathematical principles (eg Berger et al., 1987b; Grün and Macdonald, 1989), some purchased commercially, and some written with the incorporation of "canned" procedures. Shared data sets may indicate any significant differences among these programs. We present here two data sets that can be used to compare the results from exponential fits. These data may help independent workers to determine if their fitting programs actually do what they are intended to do.

As has been amply shown in the more mature geochronological disciplines (eg Brooks et al. 1972), it is extremely important that any such differences be documented early in the use of these algorithms, so that the limitations and assumptions underpinning each computation method can be appreciated. Lack of such an appreciation may lead to undesirable and unnecessary conflicts in interpretation of TL dating results.

The fitting method

We wish to compute the intersection point of two extrapolated exponential curves, as frequently encountered with the partial bleach method of Wintle and Huntley (1980). The first data set presented here was obtained from a glaciolacustrine silt, QNL84-2, described by Berger, Clague and Huntley (1987a). The second data set is from a lake sediment of Berger (unpublished). These data sets have a similar scatter (standard deviations are 4% and 3% respectively, calculated from equation 4 of Berger et al., 1987b), but differ in the percent extrapolation from the applied dose range. The form of the exponential curves applied here is

$$I = I_0 \{ 1 - \exp[- (D + D_x)/D_0] \}$$

where I is the TL intensity in photon counts, I_0 is the saturation value of the TL, D is the laboratory applied dose, $-D_x$ is the extrapolated X axis intercept, and D_0 is a fitting parameter. The desired equivalent-dose value is either $+D_x$ if the additive-dose method is used, or the dose at the intersection of the two curves if the partial-bleach method is used.

The data are listed in Table 1. The best estimates of the curve parameters are calculated by three methods: a) the quasi-likelihood or iterative least-squares method described by Berger et al. (1987b) [the first data set

(QNL84-2) is shown plotted in Figure 3 of that paper, but with the use of equal weighting]; b) a weighted least-squares method programmed for DJH by S. G. Cowan; and c) a weighted least-squares method using a simplex fitting routine programmed by DJH.

The error estimates in the intersection values are calculated in two ways; method (i) uses a fast delta method outlined by Berger et al. (1987b), whereas method (ii) uses a slow interval or likelihood-ratio technique (also see Berger et al. 1987b). Specifically, in (ii) different trial sets of parameters are tested for statistical "reasonableness" using a likelihood-ratio test, and the range of values of accepted parameters is then used to calculate the "error" (or chosen probability interval) in the intersection.

The weighting factors used in all three methods were those appropriate to an error model with a constant percent error in the TL intensity and no error in the dose variable [For justification see Appendix A of Berger et al. (1987b)]. In such a weighting scheme, the variance is proportional to the square of the TL intensity. It should be noted here for comparison that the simplex procedure used (only) for the additive-dose method by Grün and Macdonald (1989) makes no explicit assumption about the error model and consequently employs equal weighting.

The weighting scheme of method (a) uses the best estimate of the TL intensity (ie the intensity calculated from the fit), whereas method (b) uses the measured TL intensity. This is a subtle but important distinction in weighting schemes. This calculated intensity is required for statistical rigour in the derivation of the algorithm because the constant percent error in the TL signal is not yet known independently (but see below). However, we show below that in practice this distinction in weighting schemes produces no significant difference in results, for these data sets.

The fitting model (saturating exponential) is assumed to be a correct representation of the data. A discussion of the possibility of bias introduced by incorrect modeling is beyond the scope of this note.

Results

The data for the lake sediment are shown plotted in figure 1 with the best-fit curves of method (a). The best estimates of the curve parameters and the intersection values, where calculated, are compared in table 2. For all data, each of the estimates derived with method (a) for the parameters I_0 , D_x and D_0 lies

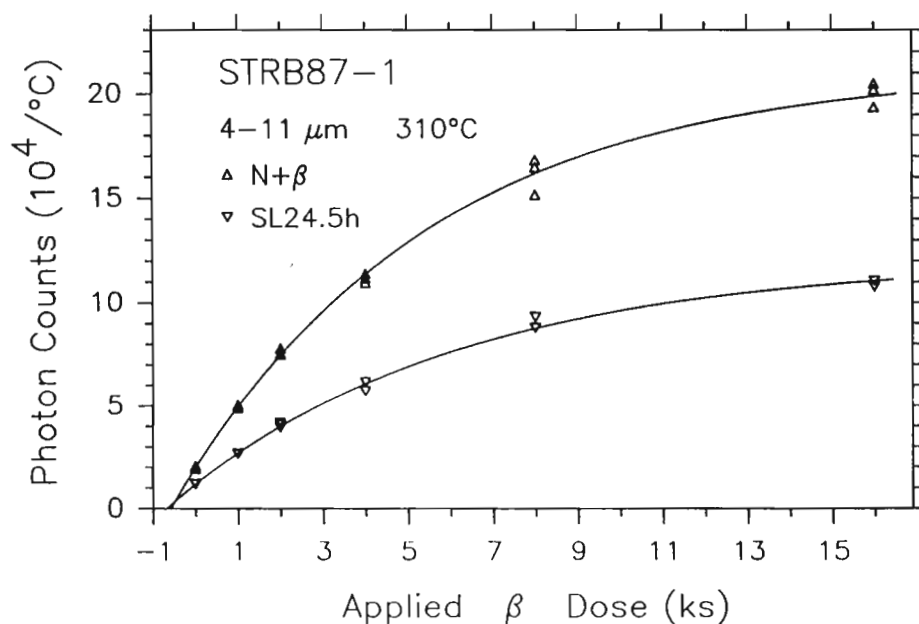


Figure 1.

Partial-bleach data and the best-fit weighted, saturating exponential curves for sample STRB87-1, computed using the method of GWB. With an unequal-weighting scheme such as employed here, high-dose points have less influence than low-dose points in determining the error in the extrapolation. For this reason the data in Figure 1 are not distributed evenly over the applied-dose range, but rather successive applied doses have been doubled.

between the two estimates calculated by the other methods. In all cases the range of these estimates was much less than their respective uncertainties, which were typically ≈ 3 -10%. The intersection values from methods (a) and (b) also agree closely.

Thus, the exact way in which the fitting is made has no significant effect on the outcome for these data. This is reassuring, for Berger et al. (1987b) stated that with good data sets (those having >10 -15 points per curve, and $<5\%$ standard deviation in the intensity values) most fitting methods should yield the same results (within error). The methods in table 2 also do not differ in the computation times for each set of three-curve parameters [3-5 s for method (a) with compiled True Basic; 3-4 s for method (b) with compiled QuickBasic, both using an 80286 CPU @ 8 MHz, without a math coprocessor].

However, the methods do differ dramatically in the computation time (under the above machine conditions) of the error in the two-curve intersection. The delta method ("a" here or "GWB" in table 2) required only ≈ 8 s per intersection value, whereas the interval method (b) required ≈ 420 s (500 trial fittings were used for the computation). A math coprocessor will reduce significantly this computation time (for both methods). For example, with an 8088 CPU @ 4.77 MHz using a coprocessor 8087 chip the error computation time for method (b) was reduced to ≈ 150 s, still far slower than the algorithm for the delta method run without a coprocessor, even allowing for the differences in CPUs. This dramatic difference in time (a factor of 50!) is not surprising because interval

methods for the estimation of intersection errors are "numerically fierce" (Berger et al., 1987b).

Implications for the error model

We have used the scatter in the data about the regression curve to obtain an estimate of the constant percent error. However, ultimately it is desirable to estimate this error from more specific experiments, and then to use the observed data scatter to obtain a chi-squared estimate and thereby to compute a goodness-of-fit parameter (Berger et al., 1987b). A chi-squared estimate would provide an assessment of the probability that the measured scatter of data points is too large (or too small). The null hypothesis is that the scatter is due only to a random variation within a population of possible TL values whose mean is the *best-fit* TL value and whose variance is known independently (the expected error in each data point). A goodness-of-fit parameter would enable us objectively to recognise and to reject spurious data points, as routinely practiced in the more mature isotopic dating methods (Brooks et al., 1972).

In view of the apparently enormous variety of TL responses in nature, even for one mineral type, can a sufficient knowledge of the variance in each intensity value ever be obtained? Could replicate TL measurements from many (100?) discs at a single dose value for each of several "known" mixes of minerals provide characteristic (representative) variances applicable to sediments having similar relative concentrations of minerals (estimated routinely by powder X-ray diffraction, for example)?

Table 1.

Data for samples QNL84-2 and STRB87-1. For the former, 2-4 μm grains, for the latter, 4-11 μm grains were used. TL data are photon counts/°C. Doses are in minutes of ⁶⁰Co gamma radiation at 1.6 Gy/min (QNL84-2), and in kiloseconds of ⁹⁰Sr beta radiation at 90 Gy/ks (STRB87-1).

QNL84 - 2				STRB87 - 1			
Unbleached		Bleached		Unbleached		Bleached	
Dose	counts	Dose	counts	Dose	counts	Dose	counts
0	38671	0	20766	0	20522.2	0	11814.6
0	40646	0	21393	0	19373.6	0	11587.8
0	38149	0	22493	0	21040.6	0	11708.6
0	35836	120	31290	0	18899.1	1	26645.2
120	65931	120	33779	1	50382.5	1	26445.2
120	67887	240	43221	1	48570.2	1	26368.6
120	66133	240	43450	1	49529.5	2	41487.1
240	82496	240	41427	2	77706.6	2	39125.1
240	86708	480	51804	2	75291.3	2	40582.5
240	86580	480	59555	2	74563.8	4	61532.1
480	110978	480	54013	4	111547.5	4	57023.6
480	113807	960	75748	4	113899.1	8	93015.8
480	114192	960	76613	4	109461.1	8	87907.7
480	109652			8	164564.9	8	87655.2
960	130373			8	151504.2	16	107618.3
960	137789			8	168042.1	16	110394.2
				16	204726.5		
				16	201964.3		
				16	193457.6		

Table 2 Best-fit parameters for the two data sets

Fit ^a	QNL84-2 chan. 120						STRB87-1 chan.145					
	unbleached (16 pts)			bleached (13 pts)			unbleached (19 pts)			bleached (16 pts)		
	I ₀	D _x	D ₀	I ₀	D _x	D ₀	I ₀	D _x	D ₀	I ₀	D _x	D ₀
GWB	14.280	122.74 ±6.73	392.0	9.64	193.4 ±18.8	762	21.214	0.5832 ±0.0178	5.96	12.043	0.6800 ±0.0226	6.67
	intersection D _e = 86.4 ±10.1						intersection D _e = 0.4846 ±0.0368					
SC	14.246 ± 0.459	121.86 ± 6.74	389.9 ±30.8	9.67 ±1.02	195.2 ±19.5	773 ±152	21.153 ±0.483	0.5825 ±0.0181	5.95 ±0.25	12.029 ±0.320	0.6823 ±0.0226	6.68 ±0.31
	intersection De = 85.0 ^{+22.1} _{-19.9}						intersection De = 0.4814 ^{+0.0782} _{-0.0825}					
S-c	14.297	123.18	393.1	9.63	192.5	757	21.243	0.5835	5.97	12.051	0.6790	6.67
S-m	14.246	121.86	389.9	9.67	195.2	773	21.153	0.5825	5.95	12.029	0.6823	6.68

Values for I₀ are the photon counts divided by 10⁴

Footnote a)

Four different fitting procedures were used:

1) GWB is the method of Berger et al. (1987b); 2) SC is the method of Cowan in which the variance is proportional to the square of the *measured* TL; 3) S-c is a weighted least-squares fit using a simplex routine, in which the variance is proportional to the square of the *calculated* TL value (as in method GWB); and 4) S-m, as for (3) but with the variance as in (2).

Two different methods of error estimation were used: the delta method of Berger et al. (1987b), and the interval method of Cowan. In method SC each fitting parameter was not allowed to vary by more than 2σ. Errors are quoted at the 68% confidence level (1σ) except for the intersection errors of method SC, which are computed at the 95% level (≈2σ). These 95% confidence estimates were obtained by adding in quadrature the limits due to the unbleached and bleached data, these being calculated independently. This addition is an approximate, ad hoc method for finding the intersection error for method SC, but the close agreement with the results of method GWB suggests that this is a valid approximation.

Summary

For good data and relatively small extrapolations, we observed no significant difference in results from the three different fitting routines illustrated here. However, if computation speed is important, then the delta method of Berger et al. (1987b) is much faster than the interval method in calculating the intersection error.

Acknowledgements

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PR. Reviewed by A. Franklin

A convenient method for preparation of fine-grain samples

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In the TL dating of pottery, the fine-grain samples are usually separated by suspending in acetone, and the acetone is eventually lost by evaporation (Zimmerman, 1971, Aitken, 1985). We have developed a separation method for fine-grains using a water flotation process. This method is more convenient and quicker than the acetone method, and it has been used in our laboratory for the past few years.

The operational procedures for the method are as follows:

1. The pottery fragment is crushed by squeezing in a vice; the rubble produced by the "vicing" is then gently crushed further in an agate pestle and mortar, and the grains in the size of less than 60 μm are selected by sieving.
2. Put the selected grains, about 400 mg, into a 150 ml beaker; the grains are then washed in the acetone-alcohol-distilled water sequence. In order to avoid the coagulation of grains after the acetone evaporation, alcohol must be used as a transition material.
3. Pour more distilled water into the beaker until the height is 70 mm. After stirring, the beaker is kept still for 10 mins, so that the grains of size greater than 8 μm deposit at the bottom of the beaker.
4. Slowly pour the suspension into another beaker. This is then kept still for 60 mins; at the end of this period grains having a size of less than 3 μm are still in suspension.
5. Finally, this suspension is tipped into another beaker thereby obtaining grains in the size range of 3 to 8 microns. The settling times of 10 mins, and 60 mins are calculated approximately according to Stokes Formula,

$$S = \frac{9 \eta h}{2(\rho - \rho') g r^2}$$

where S is the settling time, h is the height of water in the beaker, η is the viscosity coefficient of water, ρ and ρ' are the density of the grains and the water respectively, g is gravitation acceleration, and r is the radius of the grain.

6. A flat-bottomed funnel of 80 mm diameter, with a stopcock at its exit, is used for the preparation of the disc samples. A copper wire ring with a handle is placed within the funnel at the bottom; a large piece of glass is placed on this ring and this glass carries about 30 aluminium discs (10 mm in diameter and 0.5 mm thick).

7. The separated fine-grains in the beaker are resuspended in distilled water and then the suspension is poured into the funnel. During pouring, a spiral wire with a handle is used to hold down the discs and keep them still. This is removed when pouring has finished.
8. After all the fine-grains have been deposited onto the discs (about 30 min) the valve is opened and the water is drained off. The draining is initially rapid but gradually reduced until it is until drop by drop. When the distance between the water plane and sample is about 2-3 mm, the dripping must be very slow, about 15-20 second per drop, until the water is drained away. This process takes about 60 min. The drip rate is controlled by the stopcock.
9. The glass piece is then taken out of the funnel by means of the handle on the copper ring and the disc samples are dried at a low-temperature (50 °C).

The whole process takes about 3.5 hours. If a larger diameter funnel is used and the quantity of grains increased, a greater number of discs can be obtained in the same time.

Usually 30 such disc samples are prepared at a time; each disc carries 0.8-1.0 mg of sample, and the disc-to-disc scatter in TL or weight reproducibility should be not more than $\pm 5\%$. Usually the reproducibility of weight is slightly worse than that of TL, because the sensitivity of the balance is not as good as the TL instrument. It may be noted that 1 mg per disc corresponds to a thickness of 1.3 mg cm^{-2} and this is suitable for measurement of alpha-particle effectiveness, being comparable with the ultrathin TLD of 1 mg cm^{-2} used in the measurement of alpha dose-rate (Wang, 1989).

In conclusion, this method not only is convenient and quick but has good reproducibility.

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Infrared stimulated photoluminescence dating of sediments

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Following the original idea by Huntley et al (1985) to use the photoluminescence (PL) of quartz for dating, and the subsequent investigations by Smith et al (1986), we were encouraged to study the physical basis of the phenomenon. The first stimulation spectra for alkali feldspars were reported at Tallinn in April, 1987 at the Conference "Isotope methods for Baltic regional geological problems", and then in Cambridge (Hütt et al, 1988). It was shown that within a spectral region from green to the infrared, the alkali feldspar PL stimulation is connected with quite stable traps and may be used for dating. On the basis of (preliminary) experiments, a PL energy diagram was proposed with a corresponding mechanism, which explained the unexpected stability of "infrared" traps. As a result of these studies, infrared stimulation of alkali feldspars was proposed for optical dating. This permits much simplified equipment compared with that required when using green stimulation, and solves problems of interference of the stimulating light with the measured luminescence. The first "infrared" dates obtained seem to be promising.

Physical basis

The final stimulation spectra of natural alkali feldspar was obtained (fig. 1) using a pulsed excimer dye laser as a light source. This is an improvement over the previous Xe lamp system because it avoids the cutting effects of filters, and it confirms that the signal is relatively weak when using green stimulation. (see fig. 1). More detailed experiments were performed with the aim of calculating the energy parameters of the corresponding traps. As was confirmed in our previous paper (Hütt et al, 1988), the alkali feldspar PL response is a result of a complicated thermo-optical mechanism; green light transports electrons from the ground state directly to the conduction band, but infrared light ensures that electrons are only transported to excited states where additional energy (thermal activation) is needed for transition to the conduction band. The last process is exponentially dependent on temperature:

$$I = I_0 \exp \frac{E_T^*}{kT} \quad (1)$$

where, I and I_0 are the PL intensities after infrared stimulation at a temperature of T and at room temperature, respectively; k is the Boltzmann constant, and E_T^* is the thermal activation energy.

The slope of $\ln I/I_0$ vs $1/T$ yields $E_T^* = 0.2 \pm 0.1$ eV which is the thermal activation energy for electrons from excited states to the conduction band (this value was previously measured indirectly as 0.8 eV). Within error limits this value was the same for infrared stimulation with both $E_{\lambda 1} = 1.33$ eV and $E_{\lambda 2} = 1.43$ eV.

The thermo-optical bleaching (TOB) curve (after sample preheating to 250 °C) can give an estimate of the ground state thermal activation energy (fig. 2, curve 1) using the method described by Luschik (1955). Since the order of the kinetic process has not been reliably established, the results for both first and second order are considered.

$$E_T = \frac{kT_m^2}{\delta_K \ln 2} = 1.55 \pm 0.2 \text{ eV (I order)} \quad (2)$$

$$E_T = \frac{2kT_m^2}{\delta_K \ln 2} = 2.15 \pm 0.2 \text{ eV (II order)} \quad (3)$$

where, T_m is the temperature at which the PL is at half the intensity of the initial value, and where δ_K is the temperature interval over which this fall has occurred.

On the basis of more precise experiments than reported in Hütt et al (1988), a new energy diagram is suggested (see fig. 3). The existence of an infrared stimulated PL signal even at room temperature is understandable considering the rather small activation energy needed for electrons to be transported from the excited state to the conduction band (only 0.2eV). There are several possible explanations for the relatively easy PL signal bleachability by natural light (compared with TL). Huntley et al (1985) suggested optical selection of particularly light sensitive grains; existence of especially light sensitive traps may be an alternative explanation. On the basis of the experiments discussed above (see fig. 2) it is possible to suggest that the same trap is responsible for both the TL and PL observed. There is good correlation between the high temperature maximum of the TL and the temperature at which the PL has fallen to half of its maximum (~310 °C). Furthermore, assuming first order kinetics, the activation energy of the corresponding TL trap was estimated by Strickertsson (1985) to be 1.62 ± 0.05 eV, and independently by us

Figure 1. Alkali feldspars PL-response stimulation spectra.
I = intensity of PL response in arbitrary units;
E = energy of stimulating light (eV).

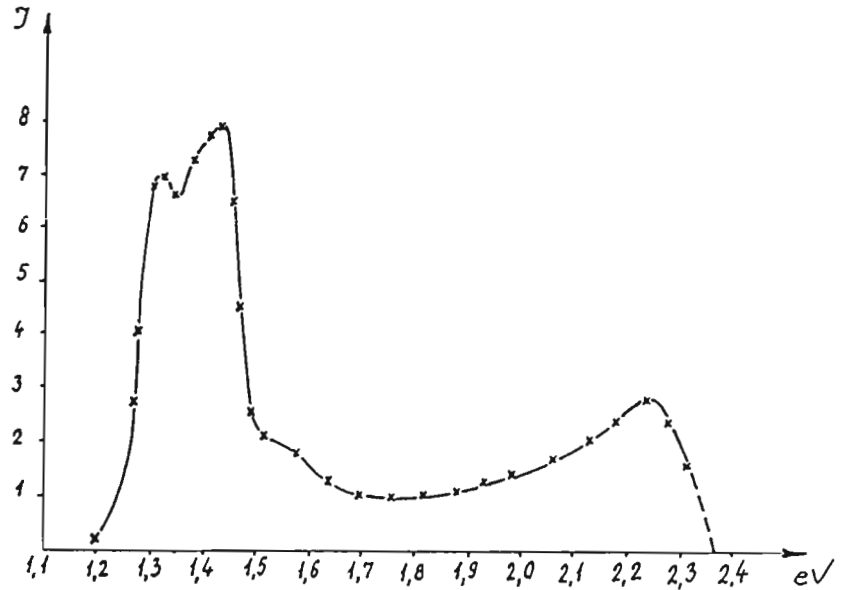


Figure 2. Thermo-optical bleaching curve (1) and TL glow curve (2). *I* = intensity of PL response for stimulation with light $E_\lambda = 1.43\text{eV}$ (1.33eV) (1) or TL-intensity (2). For (1) the sample was preheated to 250 °C.

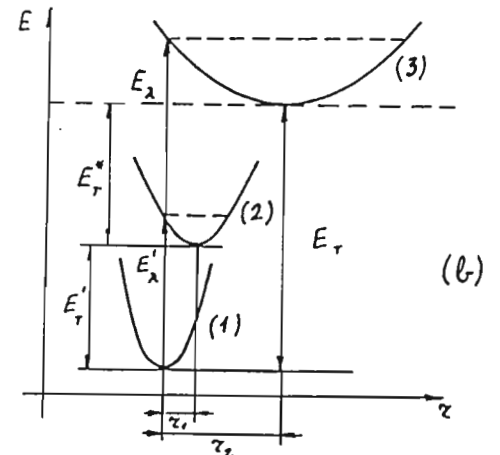
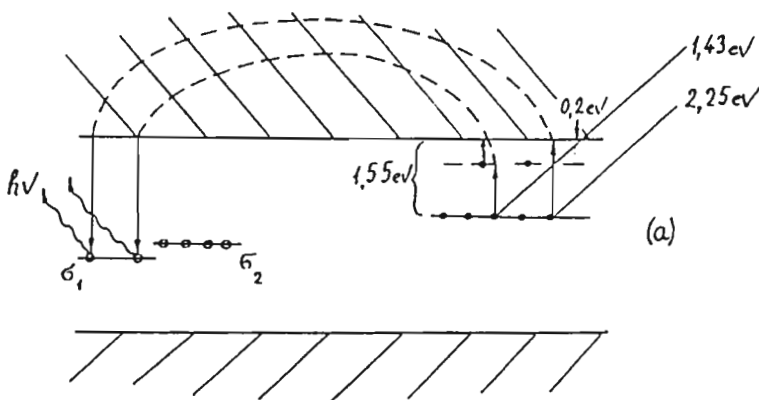
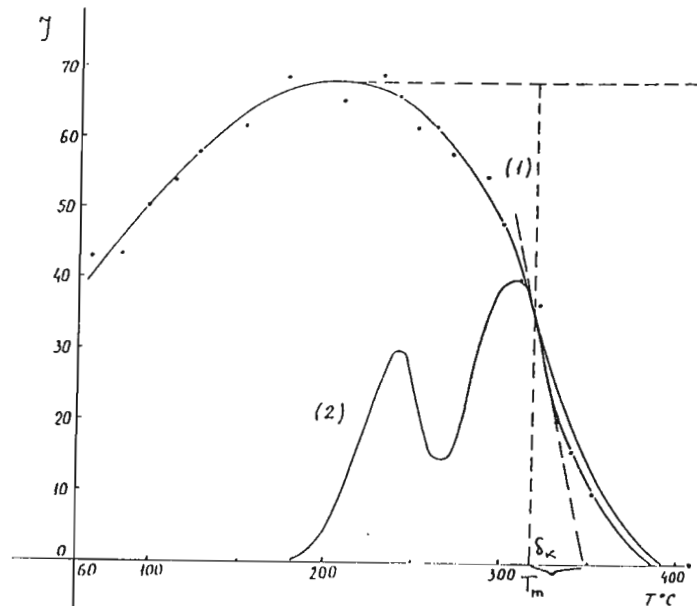


Figure 3. Energy diagram of PL-processes (a) and the same process in potential configuration coordinates (b): 1 = ground state, 2 = excited state, 3 = conduction band
 $E_T = 1.55(2.15)\text{ eV}$ $E_\lambda = 2.25\text{eV}$; $E_T^* = 0.2\text{eV}$; $E_\lambda' = 1.43(1.33)\text{ eV}$; $E_T' = 1.35(1.95)\text{ eV}$.

to be 1.7 ± 0.05 eV. This is in good agreement with the ground state activation energy of 1.55 ± 0.2 eV from TOB analysis. The differences in light sensitivity of the PL and TL signals may be connected with the distribution of charge to the recombination centres involved in TL and PL processes. Obviously only the quick component of TL-signal bleaching (Wintle and Huntley 1982; Hütt, 1988) is seen as PL. We propose that this is a result of electron recombination at centres with greater effective cross section (σ_1 in fig. 3) than those responsible for much of the TL emission (σ_2). To check this hypothesis it will be necessary to study the spectral composition of the PL response. The experimental results can be used to give an indication of the order of kinetics of the PL process. The coefficient $a = E_{\lambda}/E_T$ is not constant - the value is characteristic of particular electron transitions, but in any case it has to be greater than 1. If the kinetics of the process are second order then the "infrared" electron transition from the ground state to the excited state (λ_1) gives:

$$a = \frac{E_{\lambda_1}}{E_T} = \frac{1.33}{1.95} \text{ eV} < 1,$$

which is clearly incorrect (same for λ_2).

E_T is the energy of activation from the ground state to the excited state, calculated by $E_T = E_T - E_T^* = 2.15 - 0.2 = 1.95$ eV (see fig.3). Hence we suspect that the PL is closer to a first order kinetic process.

Infrared PL dating

Using the theory discussed above as a basis, new equipment was developed which is very small and simple in operation. As a source of stimulation, an infrared laser (860 nm; 40 mW) is used which gives a light intensity at the sample of ~ 7 mW cm⁻². The laser can work in either continuous or pulse mode. The time of the pulse can be changed automatically (we use a 3 sec. pulse). A special chamber is used with a mirror to maximize the PL signal collection. The PL response (~ 400 nm) is detected by a photon counting system in parallel with a multichannel analyser. We have detected only alkali feldspar PL signals - infrared stimulation in this region was not effective for quartz. To investigate the PL signal sensitivity to natural light, bleaching studies were conducted by exposing bulk samples of alkali feldspars to sunlight (fig. 4). We have to be very quick and careful during sampling; more than 50% of the PL signal may be lost during 5 minutes of sun exposure. Our preliminary results from dating samples with different genesis from different localities (~ 70 samples) were successful with dunes and marine sediments. Lake sediments were almost as successful, but the results from fluvio-glacial samples were problematic. Following from our paleodose laboratory reconstruction model (ibid, Hütt and Poljacov) we tentatively suggest that "infrared" PL dating is

applicable to samples within the age range $\sim 10^3$ to $3 \cdot 10^5$ a. Examples of preliminary dating results are given in fig. 5. We used the additive dose method with exponential fitting. For TL and PL dating, the same samples were used. "Zero" reconstruction for TL was performed using the R- Γ procedure and total bleach methods. ESR dates on shell were obtained by Molodkov (Tallinn). The good correlation between the three paleodosimetric dating methods testifies to the accuracy of the results.

Conclusions

1. A more precise energy diagram describing the observed PL from alkali feldspars is proposed.
2. It is suggested that the TL and PL dating traps are connected with the same type of crystal defects. The particularly high light sensitivity in the case of PL is most likely due to recombination at a different centre.
3. The infrared dating of some marine sediments has given results which are in good agreement with TL and ESR dating.
4. The expected lower and upper age limits for the infrared PL dating method are 10^3 and 10^5 a respectively.

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This paper was first presented at the Oxford Workshop *Long and short range limits in luminescence dating* in April 1989. In view of the desirability to have wider circulation of the research results contained in this paper, we invited the authors to submit their paper for publication in *Ancient TL*.

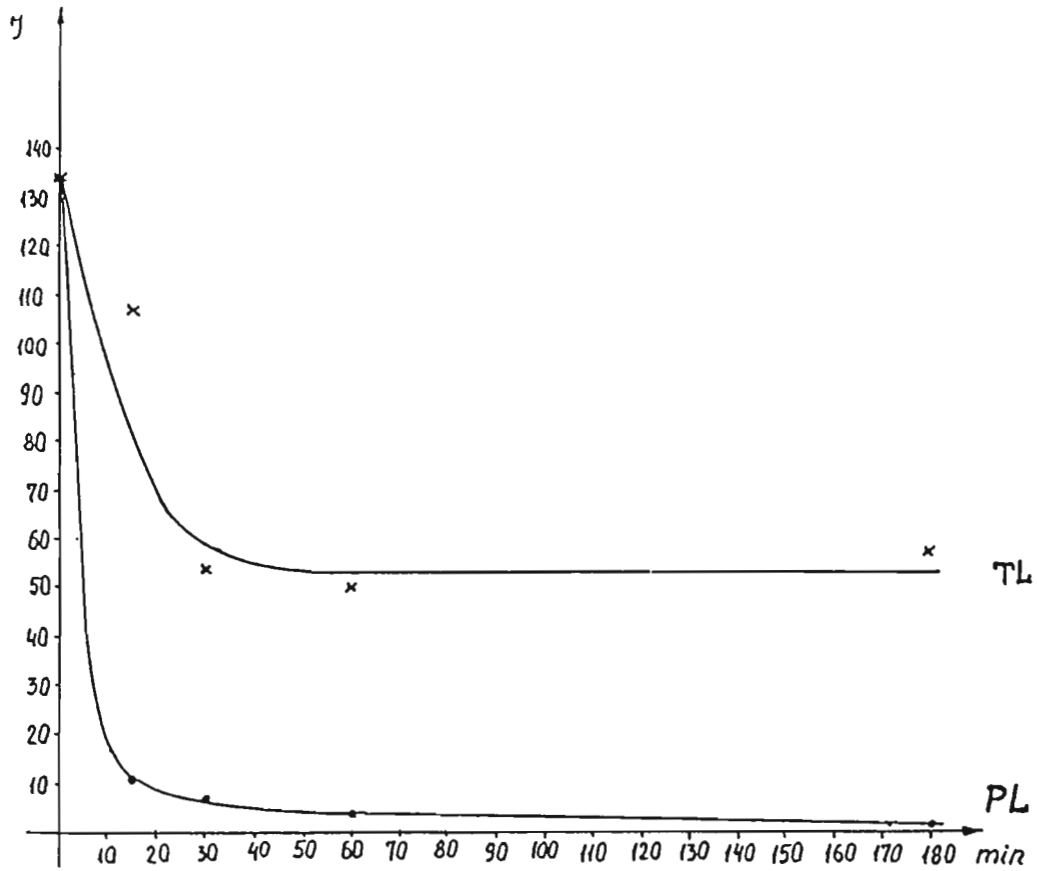


Figure 4. Bleaching of signals by sunlight for a bulk sample (following alkali feldspar extraction).

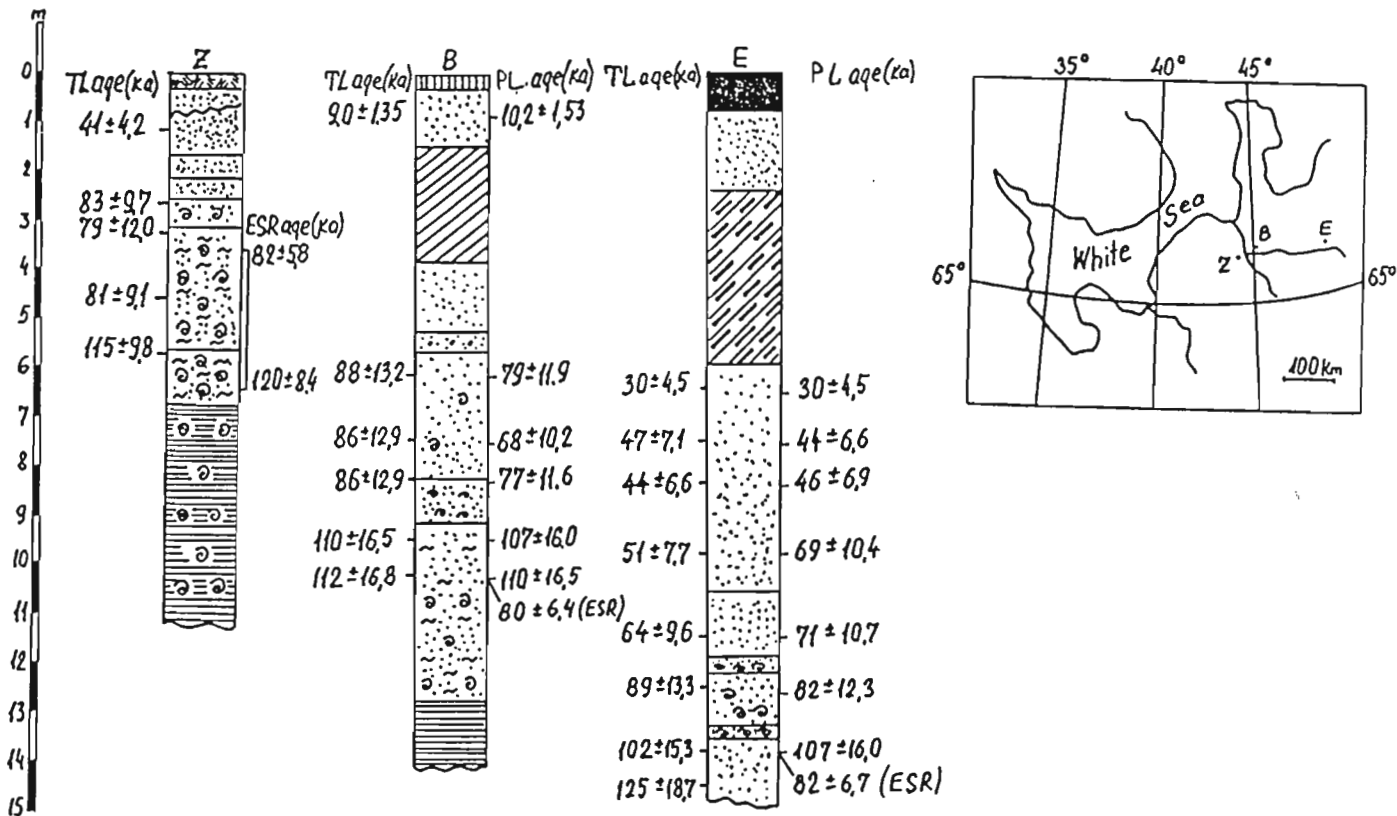


Figure 5. Palaeodosimetrical dating results for marine sediments.

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- Compiled by Ann Wintle and Ian Bailiff

Notices

6th International Specialist Seminar on Thermoluminescence and Electron Spin Resonance Dating

The 6th Specialist Seminar on TL and ESR Dating will be held in Clermont-Ferrand during 2-6 July 1990. Clermont-Ferrand is located in the Massif Central, 380 kms south of Paris. The aim of the Seminar is to bring together active research workers in the fields of thermoluminescence and electron spin resonance dating.

The Seminar will take place under the patronage of the French Committee for the International Quaternary Union (INQUA) and the International Union for Pre- and Protohistoric Sciences (UISPP).

Oral sessions will be preferentially devoted to techniques and problems, dating applications being given on posters.

To make allowance for review papers, the number of oral communications is limited to 60 (no more than one per participant). The total number of publications (including posters and oral presentations) will be limited to 140, according to the maximum capacity of the proceedings.

All sessions will be held in the Congress House (La Maison des Congrès) situated in the centre of Clermont-Ferrand. Accommodation will be provided at Hotel Coubertin, close to the Congress House.

Those interested in attending the Seminar and who have not yet received the first circular and registration form (mailed early March 1989) should apply to the organizers.

Jean Fain and Didier Miallier

Laboratoire de Physique Corpusculaire, F-63177 Aubiere Cedex, France.

Research samples: requested by Yeter Göksu who would like to obtain heated flint samples for experiments to determine temperatures reached during previous heating events.

Yeter Göksu, Inst. für Strahlenschutz, Gesellschaft für Strahlen- und Umweltforschung München, D-8042 Neuherberg, Federal Republic of Germany.

Positions available for one or two graduate students to pursue MSc or PhD degrees in optical and thermoluminescence dating. Preferred background is an honours degree in physics; others with a strong physical science background will be considered. Full time support is available.

For more information and application procedure please write to D.J.Huntley, Physics Dept., Simon Fraser University, Burnaby, B.C., V5A 1S6, Canada.
