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Concerning the normalization of additive dose optically stimulated luminescence data from quartz

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Abstract: *Since 'natural normalization' is not possible for low dose samples, several 'second dose' normalization procedures are compared. The use of light and of heat for bleaching before the second dose is considered. Attention is paid to any indication of second dose sensitivity being dependent on the magnitude of the first dose.*

Introduction

Since the introduction of optically stimulated luminescence dating by Huntley et al. (1985) some aspects of the technique have been developed analogously to established aspects of thermoluminescence dating procedures while others are unique to optical stimulation. The concern here is with one particular aspect of additive dose optically stimulated luminescence dating, namely sample to sample normalization when stimulating quartz of low natural dose with green light. 'Natural normalization', that is the use of the luminescence intensity resulting from a brief exposure of each sample to stimulating light prior to the addition of any laboratory dose, provides a very attractive approach to compensating for sample to sample variations (Stokes, 1992) and indeed natural normalisation has been much used with the infrared stimulation of feldspar (Wintle, 1993; Ollerhead et al., 1994). However the accuracy achievable from natural normalization must become less as the natural dose approaches zero and the method cannot be applied to investigations of the dose response properties of bleached material. In these circumstances normalization must be by an appropriate second measurement on the sample and Stokes (1992), using an argon ion laser for stimulation of young quartz, reduced the luminescence to about 1% of the initial value and then applied a dose of 5 Gy to each sample for normalisation. Variations on second dose normalization are compared below, involving the use of light or heat for bleaching before the second dose and looking for any indication of second dose sensitivity being dependent on the magnitude of the first dose.

The samples and measurement system

The quartz samples were prepared from BDH "acid washed sand" which was treated with concentrated HF for one hour. The purity of the quartz after this treatment was tested by verifying, for some randomly selected samples, that no luminescence was stimulated by exposure to infra red radiation. Any such signal would have indicated the presence of feldspar, the most probable contaminant. Some of the quartz was bleached by exposure to daylight for at least one week and some by heating for 5 minutes at 500°C before further use. Samples for measurement were deposited on silicone grease in the central portion of thin stainless steel discs 12 mm in diameter. Dosing was by exposure to a calibrated 90Sr beta source. A 16 green LED system with peak emission at 565 nm wavelength was used for stimulation (Galloway, 1992, 1993) while a carefully chosen filter combination of HA3, BG39, UG11, 7-59 and 7-60 filters, with a peak transmission at 365 nm wavelength, preceded the photomultiplier (EMI type 9635QA) which counted the luminescence photons. The multi-sample system described by Galloway (1991) was used for the measurements.

Method of measurement

First of all for each sample the background due to scattered light and photomultiplier noise was measured. A beta dose was then given, the sample preheated for 1 minute at 220°C to remove any unstable signal component and the luminescence measured. This was repeated for three samples for each dose and for eight doses ranging from 12.5 Gy to 175 Gy and the samples were then normalized to generate a growth curve showing the dependence of luminescence on dose. A separate set of 24 samples was processed in this way for each of the normalization procedures considered.

Normalization required that the samples be bleached prior to application of the normalizing beta dose and bleaching was either by placing the samples in a Honle SOL-2 "solar simulator" for 1 hour or by heating the samples to 450°C for 1 minute. The sensitivity to the normalizing beta dose might depend on the dose to which the samples had been exposed before bleaching, as can occur in the case of thermoluminescence, Aitken (1985). To allow for this possibility the normalized dose response was compared for samples which had a second beta dose added to bring all to the same total dose before bleaching, with samples which had not; this was applied in both the SOL-2 and the heat bleaching procedures. In addition to these four normalization methods, one further procedure was investigated, based on a "single aliquot" approach developed for infra red stimulation of feldspar (Duller, 1991, 1992) and tested to a limited extent with green stimulation of quartz (Galloway, 1994). In this variant, each sample had a second beta dose added so that the sum of the two beta doses equalled the maximum beta dose applied to any of the set of samples to be normalized. The sample was then preheated, read and compensation applied in the single aliquot manner for loss of signal due to the first preheating and reading.

The normalized dose response measurements

a) Quartz bleached by daylight

Dose response curves for quartz initially bleached by daylight and normalized by four of the procedures outlined above are shown in fig. 1. It is immediately clear that one of the methods of normalization, involving heating of the samples to 450°C before applying the normalizing dose, is not successful in reducing the scatter in the data. The two procedures which involved giving all samples an equal dose before normalization, one with bleaching by the SOL-2 and the other developed from the single aliquot approach, show an essentially identical dose response with little scatter in the normalised data. The procedure in which the samples were not given equal doses before bleaching and normalization gives a response which shows little scatter but falls below the others. This could be due to the sensitivity after bleaching increasing with previous dose. That preheating can cause thermal sensitization analogously to the thermoluminescence pre-dose effect has been observed by Godfrey-Smith (1994). The data in fig. 1 were measured with a 100 s exposure time to the green LEDs. The more encouraging normalization procedures, that is excluding the one involving 450°C heating as a bleach, were investigated further using green light measurement times of 50 s and 12.5 s with the results shown in figs. 2 and 3. These measurements confirm that the two procedures which involved giving all samples an equal dose before normalization show an essentially identical dose response with little scatter in the normalised data, while the procedure in which the samples were not given equal doses before bleaching and normalization gives a response which falls below the others.

However extending the investigation to higher doses at which saturation becomes more significant, fig. 4, shows that normalization based on measurements corrected in the single aliquot manner is no longer satisfactory; the simple correction procedure is strictly applicable only when the luminescence response to dose is linear (Duller, 1994).

b) Quartz bleached by heating at 500°C

The tests on quartz initially bleached by daylight should be relevant to sediment dating so for completeness similar tests were carried out on quartz which had all luminescence removed by heating which should be relevant to the dating of pottery. The response curves for five different normalization procedures are shown in fig. 5 and in terms of scatter in the normalized luminescence values there is nothing to choose between the procedures. Four out of the five procedures give consistent results and only the procedure in which the samples were not given equal doses before bleaching by the SOL-2 and normalization gives a response which differs by falling below the others. This procedure showed the same tendency in the tests on daylight bleached quartz in figs. 1, 2 and 3. The initially heated quartz behaved differently from the initially daylight bleached quartz discussed in section (a) above when heating to 450°C was used as a bleach prior to application of the normalizing beta dose, in that entirely acceptable normalization resulted (compare fig. 5 and fig. 1). Further, when 450°C heating was used as a bleach the normalization was not dependent on whether the samples had received the same dose before normalization, in contrast to the situation when the SOL-2 was used for bleaching.

It is interesting to note in passing that the dose response curves for the daylight bleached quartz show a different relationship between luminescence and dose from those for the 500°C heated quartz. Initially the response of the former is approximately linear, up to about 25 Gy say, whereas the latter is clearly non-linear in this region.

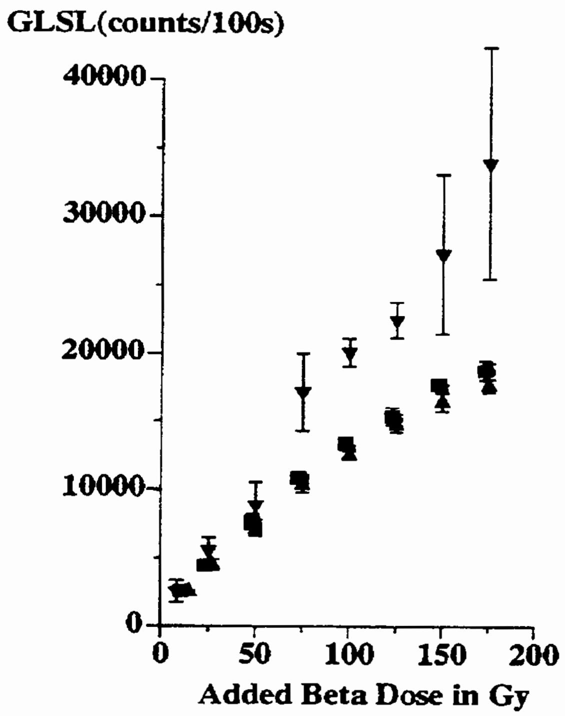

Figure 1
The dependence of the green light stimulated luminescence (GLSL) on beta dose for quartz previously bleached by daylight. The luminescence was measured for 100 s. Each point represents the mean of three measurements while the error bars indicate the spread in the three measurements. Four different normalization procedures are compared.

(1) samples were brought to the same total beta dose (175 Gy) before bleaching in the SOL-2 and application of the normalizing dose, indicated by squares.

(2) samples were brought to the same total beta dose (175 Gy), preheated and read for normalization with correction in the single aliquot manner, indicated by circles (which are indistinguishable from the squares on which they are superimposed).

(3) samples were bleached in the SOL-2 without bringing to the same total dose and the normalizing dose applied, indicated by upward pointing triangles.

(4) samples were brought to the same total dose (175 Gy) then bleached by heating to 450°C before application of the normalizing dose, indicated by downward pointing triangles and large error bars.

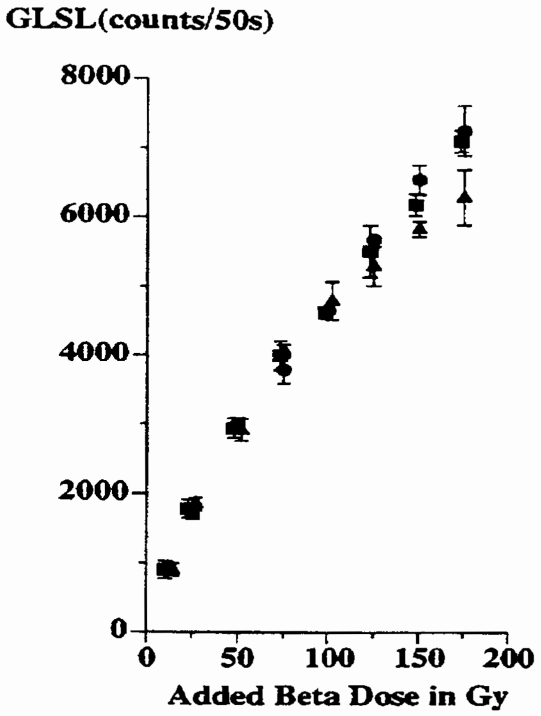

Figure 2
As fig. 1 but for 50 s green light stimulation, omitting the least successful normalization procedure involving heating to 450°C.

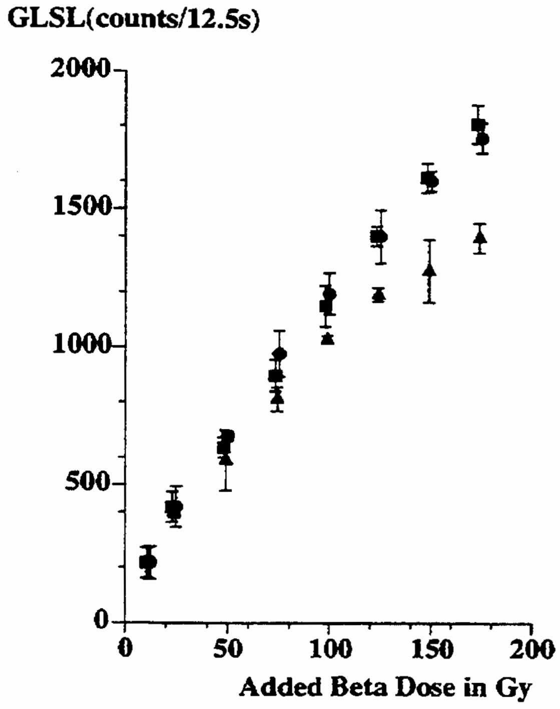

Figure 3
As fig. 2 but for 12.5 s green light stimulation.

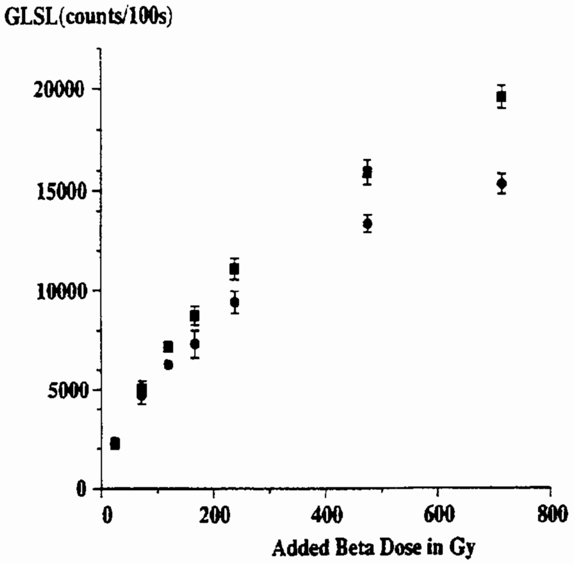

Figure 4
The dependence of the green light stimulated luminescence (GLSL) on beta dose for quartz previously bleached by daylight, as in fig. 1 but extending to higher doses at which saturation becomes more significant. Each point represents the mean of three measurements while the error bars indicate the spread in the three measurements. Two different normalization procedures are compared.

(1) samples were brought to the same total beta dose (700 Gy) before bleaching in the SOL-2 and application of the normalizing dose, indicated by squares.

(2) samples were brought to the same total beta dose (700 Gy), preheated and read for normalization with correction in the single aliquot manner, indicated by circles.

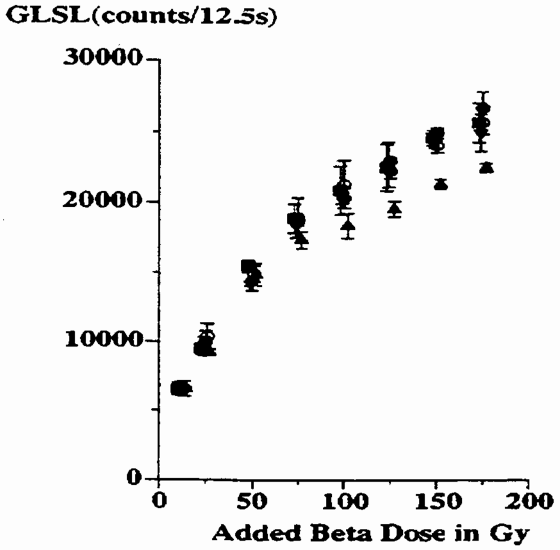

Figure 5
The dependence of the green light stimulated luminescence (GLSL) on beta dose for quartz previously bleached by heating to 500°C. The luminescence was measured for 12.5 s. Each point represents the mean of three measurements while the error bars indicate the spread in the three measurements. Five different normalization procedures are compared.

(1) samples were brought to the same total beta dose before bleaching in the SOL-2 and application of the normalizing dose, indicated by solid squares.

(2) samples were brought to the same total beta dose, preheated and read for normalization with correction in the single aliquot manner, indicated by solid circles.

(3) samples were bleached in the SOL-2 without bringing to the same total dose and the normalizing dose applied, indicated by upward pointing triangles.

(4) samples were brought to the same total dose then bleached by heating to 450°C before application of the normalizing dose, indicated by open circles.

(5) samples were bleached by heating to 450°C without being brought to the same total dose before normalization, indicated by downward pointing triangles.

Due to overlap not all symbols are inividually distinguishable.

Conclusions

Normalization involving sample bleaching by SOL-2 prior to application of the normalizing dose was successful provided all samples had been given the same total dose before bleaching.

Normalization involving bleaching the sample by heating prior to application of the normalizing dose was only successful for quartz which had been zeroed originally by heating.

A variant of the single aliquot method also provided a successful procedure for normalization within the dose range for which the luminescence response is approximately linearly related to dose.

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Reviewer

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Non linear approach of TL response to dose: polynomial approximation.

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Abstract: *In the field of luminescence or electron spin resonance dating, exploitation of data techniques are under investigation in order to make the dating results more accurate. A non linear approach of natural dose determination is presented, using a polynomial fitting of thermoluminescence growth-curves (intensity of signals versus irradiation dose). This is based on the usual implicit assumption that the zeroing procedures performed to remove the signals due to natural irradiation do not affect the shape of the growth-curves. The basis and way of use of this polynomial approximation are detailed.*

A review of the additive dose technique and growth curve fitting

a) Experimental process for natural dose determination

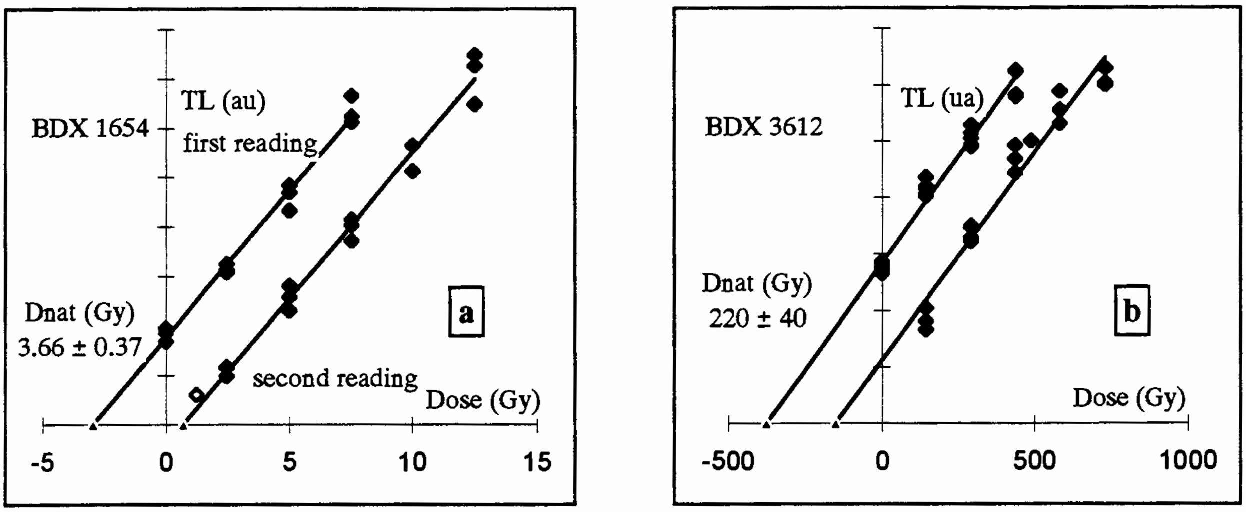
The usual procedure for natural dose measurement consists in reading a first series of additive dosed identical aliquots of the material to be dated. This first series of experiments is followed, after zeroing the natural signals of new aliquots, by a second series of laboratory irradiations and readings. These second reading experiments, second glow-curves of thermoluminescence, are used to regenerate the signal and to build the growth curve of the intensity with the laboratory dose administered to the sample. This curve is then fitted through the experimental points of the first series of experiments (natural plus artificial dosed material) assuming that the zeroing procedure does not affect the growth characteristics. The intercept of the curve with the dose-axis at null intensity allows the natural dose evaluation (Zimmerman, 1971; Fleming, 1979; Aitken, 1985).

b) Linear fitting of growth curves

The simplest and well known exploitation of data consists of a linear fit of the first reading points with a regression line whose intercept with the x-axis (dose-axis) gives a first "uncorrected" value of the natural dose, D. To take into account the non linearity behaviour of the TL-response to dose, the same treatment is carried out with the second reading experiments . The extrapolation of the best fit line with the x-axis gives a corrective term, d, which is algebrically added to D in order to obtain the natural dose [1]:

$$D_{nat} = D + d \qquad [1]$$

This kind of treatment is convenient when the experimental behaviour is "not so far" from linearity. Practically, when the absolute value of the corrective term d does not exceed 10 to 15 % of the total natural dose, one may consider that the linear approximation is a satisfying tool for dose determination. However, if the growth curve of the material exhibits a significant non linearity _ a supralinearity which corresponds to an enhancement of sensitivity with the increasing irradiation dose, or a saturation of signal, which demonstrates a limited ability for charge trapping _ the utilization of a linear fit can produce large errors or uncertainties in dose determination. Several examples are given at figure 1: fig. 1a shows a typical supralinear feature of fine grains (mainly quartz) extracted from a medieval brick sampled at the Saragosse cathedral (Spain), fig. 1b a saturating growth curve of heated fine grains, quartz and feldspars, extracted from a mousterian fire area of Grotte XVI, Dordogne (France) and fig. 1c an intermediate, nearly linear, growth curve of fine grains, (quartz and a small amount of potash feldspar), from a piece of early neolithic pottery sampled at Matera-Trasano, Basilicate (Italy). In most cases, the question is the reliability of dose determination using linear regressions of growth curves.



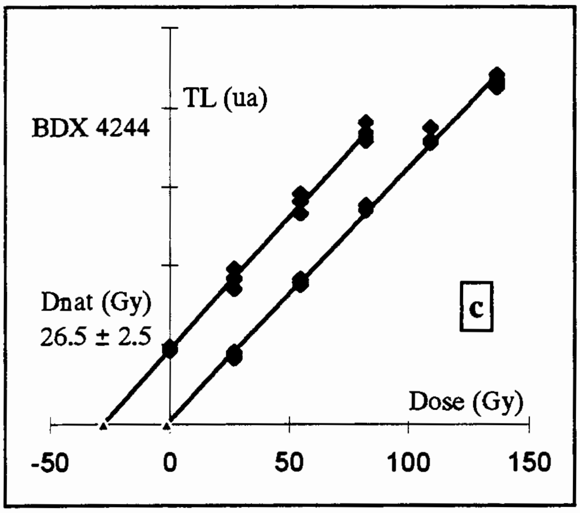


Figure 1
Linear fit of growth curves; D_{nat} values are given by the distance in unit of dose between the intercepts of the best fit regression line with dose axis for both first and second reading experimental points, marked by triangles. Fig. 1a shows a supralinear behaviour of fine grains, mainly quartz, extracted from a medieval brick, BDX 1654, at the Saragosse cathedral (Spain); the white diamond shaped point was not taken into account for the regression calculations. Fig. 1b: a saturating growth of heated fine quartz grains collected in a mousterian fire area at Grotte XVI, Cénac et St Julien, Dordogne (France), BDX 3612. Fig. 1c: an intermediate growth of TL with dose exhibited by fine grains of quartz extracted from a piece of early neolithic pottery collected at Matera Trasano, Basilicate (Italy), BDX 4244. The second reading intensities of TL have been corrected from sensitivity changes.

function	remarks and references
[2] $I = A.(1 - e^{-ax})$	single exponential, deduced from one trap kinetics model. (Mejdahl, 1985; Poljakov and Hütt, 1990; Grün and Brumby, 1994; Lamothe *et al.*, 1994).
[3] $I = A.(1 - e^{-ax}) + B.x$	same as [2] including a linear term, B.x, related to the creation of new traps during irradiation. (Schwarcz, 1994; Grün and Mc Donald,1989).
[4] $I = A.(1 - e^{-ax^{\gamma}})$	the γ exponent to the dose x allows to reproduce non-linearity effects at low doses. (Grün and Mc Donald, 1989; Barabas *et al.*, 1992;...).
[5] $I = A.(1 - e^{-ax})^{\alpha}$	The α exponent, if greater than unity, simulates the supralinearity behaviour. (Valladas and Gillot, 1978).
[6] $I = A.(1 - e^{-ax}) - B.(1 - e^{-bx})$	This linear combination of 2 exponential functions is deduced from a kinetics model involving two linked traps. (Ninagawa *et al.*, 1992).

Table 1: exponential based functions used in natural dose determination: short comments and some references.

Note that a strictly correct measurement of the natural dose by linear fitting requires irradiation doses used for second reading experiments to be equal to those which were used during the first series of experiments, i.e. the sum of the natural dose and the artificial doses, which implies the previous knowledge of the natural dose... i.e. that which is being determined. This problem gives rise to the most common limitations in linear fit. To solve it, more convenient functions are used.

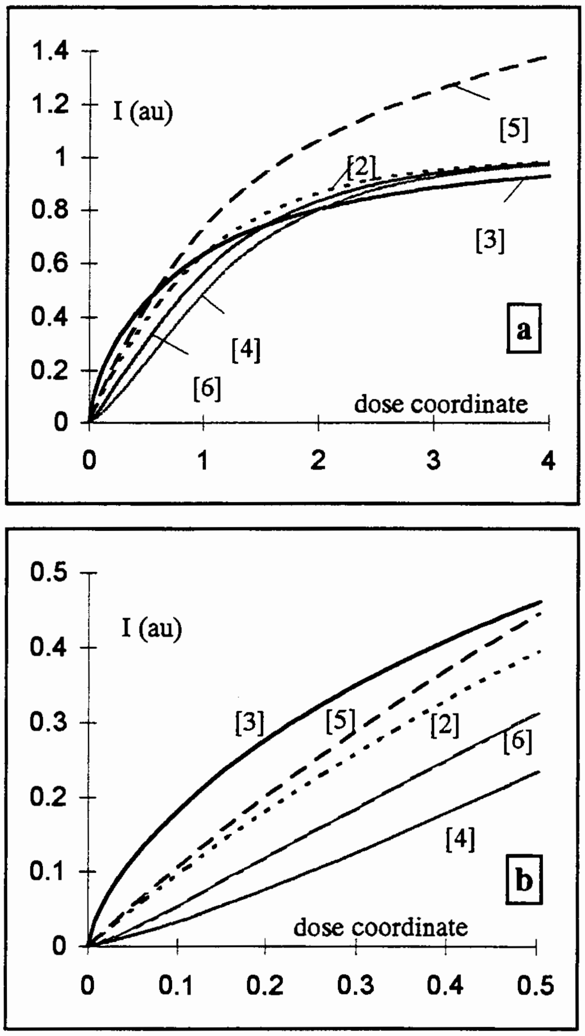


Figure 2

Graphic display of functions reported in table 1: the curve numbers recall those of functions in table 1. Figure 2a shows the general shape of the curves until 'high' dose saturation; figure 2b details the low dose region. Curves [5] and [6] reproduce the supralinearity behaviour of TL that many samples exhibit after β irradiation. (* list of parameters used for this representation: function [2]: $A=1$, $a=1$; [3]: $A=1$, $a=1$, $B=0{,}1$; [4]: $A=1$, $a=1$, $\gamma=0.7$; [5]: $A=1$, $a=1$, $\alpha=1.25$; [6]: $A=1.5$, $a=1$, $B=0.5$, $b=2.5$).*

c) Non linear fitting of growth curves

One of the most common approaches of the variation of the signal intensity, I, with the dose x, is a saturating exponential [2] as reported in table 1. This model is deduced from kinetics studies involving one single trap. Since it cannot be of general use, other functions including saturating exponentials have already been suggested in the field of luminescence or electron dating and are displayed in table 1. All of these representations are based on both theoretical models and empirical observations. An additive exponent to the dose or to the saturating function itself, relations [4] and [5], reproduces the supralinearity or sublinearity curvature of growth curves in the low dose region. Figure 2 provides a comparison of these exponential based functions. Note that this kind of representation of the growth curves has been successfully carried out with highly irradiated materials in the field of geological dating (see table 1 for references).

Since the main application of TL at the Bordeaux dating laboratory concerns archaeological materials (Schvoerer *et al.*, 1974; Bechtel, 1983; Guibert *et al.*, 1994), mostly ceramics, which are usually younger and less irradiated than geological ones, other types of functions were preferred in order to describe a supralinear behaviour as well as a saturating growth. On the other hand, exponential functions based on trap filling models involving one or two traps of a particular type of mineral did not seem appropriate to describe the complex properties of the TL signals originating from polymineral fine grains, according to the technique usually performed in Bordeaux. Investigations had been carried out to check the reliability of polynomial approximation in natural dose determination during the early 1990's in Bordeaux. Polynomial fitting technique is now of general use by our group (Guibert *et al*, 1994). Other authors have also reported recent dating results using a polynomial approach for growth curve fitting and natural dose determination (Mercier *et al.*, 1991; Chawla *et al.*, 1992; Loyer *et al.*, 1995).

We will present in the following sections the basic principle and the use of polynomials for natural dose determination .

Natural dose determination principle by a least square method

As presented above, the natural dose is determined by the intercept of the "natural plus dose" growth curve with the dose-axis. The first step of the measurement procedure is to choose the more appropriate function. This operation is generally done with the second reading experiments: let this function be $f_2(x)$ where x is the laboratory dose and the index 2 recalls this is related to the experimental data of the second reading series of records. If the material behaves ideally, the signal to dose response, either natural or artificial, does not change with the annealing process by either heating, or bleaching or a combination of both. Changes in sensitivity are usually observed; however, in most cases, the zeroing procedure does not affect the relative variations of intensity of signals with dose. Hence, supported by experimental evidence, we can write that, if f_1 describes the growth curve for the first reading experiments, f_1 and f_2 are assumed to be linked by the following relationship [7]:

$$f_1(x) = A.f_2(x+D_{nat}) \qquad [7]$$

where x is the laboratory dose, A a multiplicative factor which characterizes the differences in sensitivity observed between first and second reading and D_{nat} the natural dose which is being determined. First and second growth curves have the same characteristics; a translation of dose D_{nat} - changing $f_2(x)$ into $f_2(x+D_{nat})$ - and an application of a scale factor equal to the ratio of sensitivities, A, are the only differences between the first and second growth curves.

To determine D_{nat}, the best fit of $f_1(x)$ through the experimental points (Intensity / dose) corresponding to the first reading will minimize the quadratic sum E of the differences between the experimental points and the $f_1(x)$ function:

$$E = \sum_{i=1}^{n} (y_i - f_1(x_i))^2 \qquad [8]$$

with y_i and x_i respectively being the signal intensity and the dose absorbed by the i^{th} aliquot. According to the relation [7], [8] becomes:

$$E = \sum_{i=1}^{n} (y_i - A.f_2(x_i+D_{nat}))^2 \qquad [9]$$

where n is the number of experimental data obtained during the first reading experiments. This least square procedure means that the best adjusted values of A and D_{nat} verify the following expressions [10] and [11], at the same time:

$$\frac{\delta E}{\delta A} = 0 \Rightarrow$$

$$\sum_{i=1}^{n} f_2(x_i+D_{nat}).(y_i - A.f_2(x_i+D_{nat})) = 0 \qquad [10]$$

$$\frac{\delta E}{\delta D_{nat}} = 0 \Rightarrow$$

$$\sum_{i=1}^{n} f_2'(x_i+D_{nat}).(y_i - A.f_2(x_i+D_{nat})) = 0 \qquad [11]$$

where f_2' is the first derivative function of f_2 respect to D_{nat}.

Finally, the determination of the natural dose is equivalent to solve the general equation system just mentionned. Since the function f_2 has no particular form, these relations are of very general interest and can be applied to any type of derivable function, exponential as well as polynomial.

Polynomial approximation.

a) Polynomial fitting of second reading growth curve. The starting point of the polynomial approach is that the intensity equals zero when no irradiation was delivered to the sample to be dated: $f2(0) = 0$. Consequently, particular kinds of function are looked for whose general formulae are given by relation [12]:

$$f_2(x) = x.S_2(x) \qquad [12]$$

where $S_2(x)$ is the mean sensitivity of the material being studied, varying with the dose x in the case of non linear growth. With such a formula, the growth curve which characterizes the second reading experiments is forced to start at zero, for no irradiation. Practically, $S_2(x)$ is approximated by polynomial functions, deduced from a vectorial basis of orthogonal polynomials (Draper and Smith, 1966). A set of N polynomials is defined whose degree ranges from 0 to N-1, where N is the number of strictly different doses administered to the sample during the second reading experiments.

Figures 3a to 3c illustrate a polynomial fit of the experimental mean sensitivity, evaluated by the ratio of signal intensity to dose, in different examples of growth from fine grains of archaeological materials previously presented.

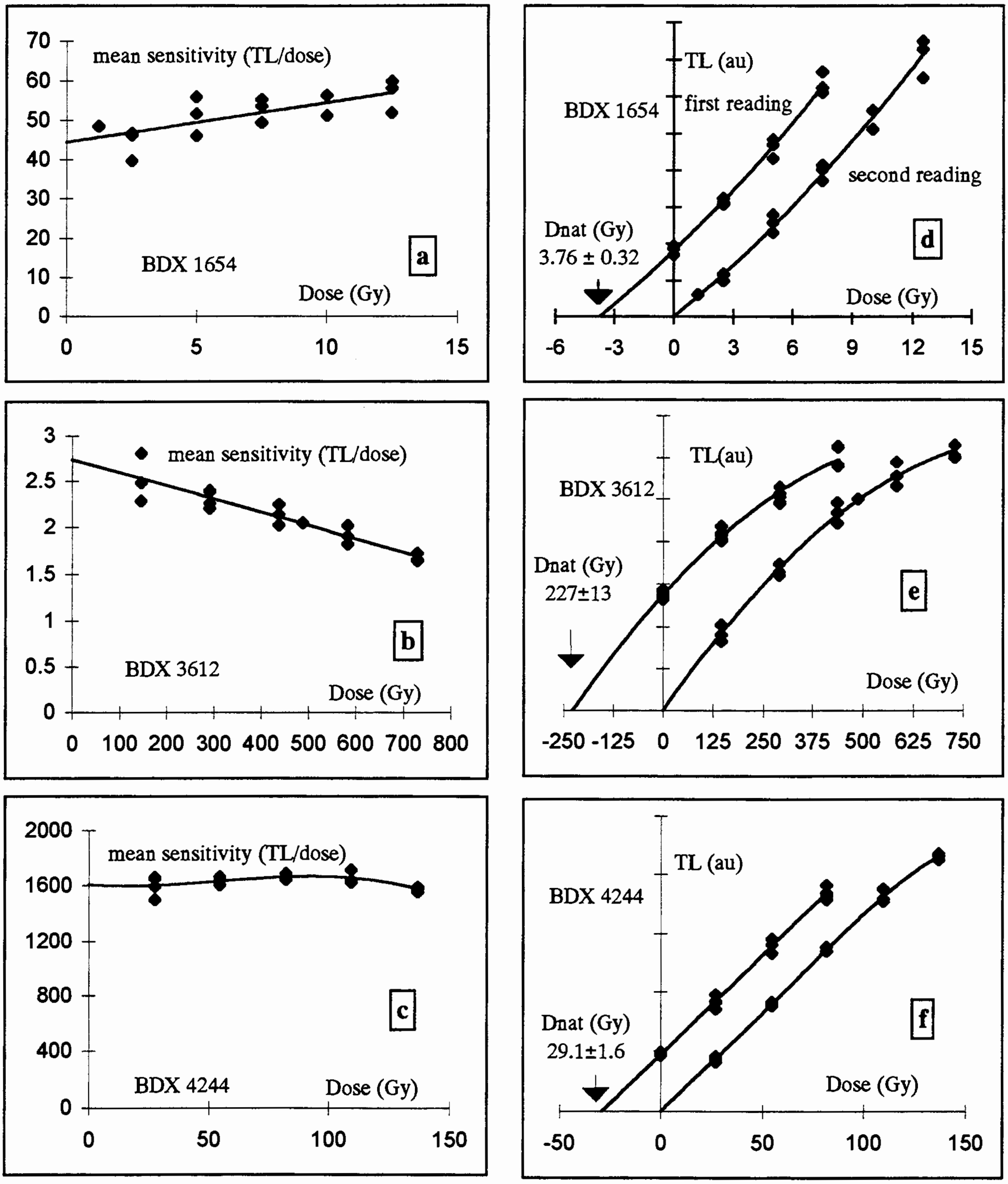


Figure 3: *Mean TL sensitivity (ratio of intensity to dose) and TL intensity as a function of laboratory dose: experimental points and polynomial fitting (same examples as in fig.1). Fig 3a and 3d, medieval sample BDX 1654 dated 1259±49 AD: a first order polynomial fit of the mean TL sensitivity has been choosen, TL growth curves are thus approached by a parabola. Fig 3b and 3e, heated fine grains of quartz from a mousterian fire area, BDX 3612, dated 65.6±4.6 ky: first order polynomial for mean sensitivity and second order for growth curves. Fig 3c and 3f: fine grain from the early neolithic potery, BDX 4244, dated 5910±440 BC: 3rd order polynomial for sensitivity, 4th order for TL growth curves. Corrections for differences in sensitivity between first growth and regenerated growth have been taken into account in order to make the curves parallel and easy to compare.*

b) Natural dose evaluation.

The second reading growth curve is at this stage approached by N different polynomial functions $f_{2,k}(x) = x.P_{2,k}(x)$ with x being the dose and $P_{2,k}(x)$ the kth degree polynomial.

The least square fit of polynomials $f_{2,k}(x)$ through the experimental points of the first series of measurements (natural plus laboratory irradiated aliquots) is done using the method previously described. N different values of D_{nat} are then obtained according to the existence of the N different polynomial functions $f_{2,k}(x)$ which satisfy the equations [10] and [11].

At this stage of data exploitation, the chronologist has to choose the best representation among the N possibilities. For that purpose, the N sums of squares E_k which characterize the quality of the polynomial fitting, deduced from the expression [9] are evaluated and displayed (see appendix 1 for a desciption of a computerised procedure):

$$E_k = \sum_{i=1}^{n} (y_i - A_k.f_{2,k}(x_i + D_{nat,k}))^2 \qquad [13]$$

with k being the degree of the polynomial function $f_{2,k}$, n the number of the first series experiments, y_i the signal intensity of the ith experiment, x_i the corresponding laboratory dose; A_k and $D_{nat,k}$ are the best adjusted values of A and D_{nat} obtained with the kth degree polynomial. An estimate of the quality of the calculation is also given by a graphic representation of mean sensitivity and growth curves as displayed in figures 3a to 3f and commented in corresponding caption.

The polynomial function which gives the least sum of squares, E_k, is selected and if several polynomials seem to be available, the one of lower degree is usually preferred in order to avoid some meaningless pseudo oscillations of the growth curves which may occur when the statistical dispersion of the experimental data is high. By this way, the natural dose is determined.

In order to avoid some possible divergences caused by the extrapolation of the polynomial function out of the range of dose within which it had been defined, a simple rule of thumb is to be respected: the range of laboratory irradiation doses related to the second reading experiments must include the total range of doses, natural dose plus laboratory doses, related to the first series of experiments.

c) Uncertainty evaluation.

Here, we will consider the uncertainty brought on natural dose by the dispersion of signal intensities although other sources of error may exist as, for example, the error on dose rate of calibrated radioactive sources.

The signal intensity originating from different aliquots is assumed to randomly fluctuate. For each experimental point indexed i, an estimate of the uncertainty, the standard-deviation σ_i, is associated to the signal intensity, y_i (appendix 2: evaluation of σ_i). The evaluation of the standard deviation of D_{nat} results from the usual formula [14] giving the standard-deviation of a function of several parameters affected by independent random fluctuations (CEA, 1978):

$$\sigma D_{nat} = \left(\sum_{i=1}^{m} \left(\frac{\delta D_{nat}}{\delta y_i} . \sigma_i \right)^2 \right)^{1/2} \qquad [14]$$

with m being the total number of measurements including the first series of records, "natural plus dosed" aliquots, and the second series, "regenerated" aliquots. However, since the analytical expression of D_{nat} as a function of signal intensities y_i, is *a priori* unknown, a computerised calculation of σD_{nat} is operated following the expression [15] which derives from the previous one:

$$\sigma D_{nat} = \left(\sum_{i=1}^{m} (\Delta D_{nat,i})^2 \right)^{1/2} \qquad [15]$$

where $\Delta D_{nat,i}$ represents the deviation between D_{nat} and a new and transient value of the natural dose calculated by changing the ith intensity y_i into $y_i + \sigma_i$, keeping all other parameters constant.

Conclusion

Many samples to be dated exhibit a non-linear growth of TL with irradiation dose. In order to make the natural dose measurement more accurate and reliable, some researchers have attempted to model the experimental behaviour, signal response to dose, with more appropriate functions than simple lines. Exponential functions or a linear combination of exponential functions had already been presented; they are deduced from kinetics models involving one or two trap-centers, assuming, as usual, a linear relationship between signal intensity and trapped charge population.

Without questionning the previous work, we suggest a more empirical approach for natural dose evaluation, because we think that trap-filling and emptying mechanisms are generally more complicated than those described by only one or two traps. Obviously, this is true with polymineral fine grains (poly-kinetics...) from archaeological samples. A polynomial approximation of growth curves, based on the experimental behaviour of the material being dated, provides a satisfying versatility and can be successfully used in many situations in order to determine the archaeological or geological natural dose.

Acknowledgements

This work was supported by the following organisms: Université de Bordeaux III, CNRS, Région Aquitaine. Special thanks to Frédéric Gérard, research student, for his participation in writing the data exploitation software, to Françoise Roland, CNRS, and Omar Bouchta, Maison des Sciences de l'Homme d'Aquitaine, for their experienced advice in regression analysis.

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Appendix 1

The natural dose is evaluated using a 'home-made' software package, called "polytl" which is a part of an application, written in C language and devoted to the control of an automatised TL apparatus built at the CRPAA laboratory and to the exploitation of data (determination of plateaux region, activation energies, natural doses,...).

The calculation of D_{nat} using polynomials is carried out as follows: in the expression [11], the parameter A can be written:

$$A = \frac{<fy>}{<ff>} \qquad [16]$$

according to a concise representation with:

$$<fy> = \frac{1}{n}\sum_{i=1}^{n} y_i.f_2'(x_i+D_{nat})$$

and $$<ff> = \frac{1}{n}\sum_{i=1}^{n} f_2'(x_i+D_{nat}).f_2(x_i+D_{nat}) .$$

The new expression of A, [16], replaces the old one in [10] which becomes:

$$R = <fy> - \frac{<fy>}{<ff>} <f^2> = 0 \qquad [17]$$

with $$<fy> = \frac{1}{n}\sum_{i=1}^{n} y_i.f_2(x_i+D_{nat})$$

and $$<f^2> = \frac{1}{n}\sum_{i=1}^{n} (f_2(x_i+D_{nat}))^2$$

The equation [17], R=0, is true for the final value of the natural dose D_{nat}. In practice, solutions of the equation [17] are numerically found using an iterative process. From the user's point of view, the parameters sent to "polytl" are the addresses of TL intensities, laboratory doses and TL curve identificator tables. The natural dose calculation is then carried out by the computer. A set of N polynomial functions is displayed. To facilitate the choice of a particular function, the quadratic sums, E_k, expression [13], are shown with the D_{nat} values and the required number of iterations. Mean sensitivity and growth curves (figure 3) are also displayed and can be used for estimating the validity of the fit. The results can be output to a printer or into an ASCII file readable by many commercial software packages for data treatment.

Appendix 2

The standard deviation of TL intensities are evaluated by two different ways:

i. the first one is deduced from the assumption that the standard deviation is constant whatever the intensities;

ii. the second one is based upon the assumption that dispersion of signal intensity leads to dispersion in weight of the aliquots; the standard deviation is thus proportionnal to signal intensity and, in other words, the relative standard deviation is assumed to be constant.

According to the user's choice, the absolute and relative standard deviations are the quadratic mean value of respectively absolute difference and relative difference between the observed signal intensity and the one expected with the best fit polynomial. As the natural dose measurement needs two series of experiments, two independent values of the absolute or relative standard deviations are then determined and utilized for the uncertainty evaluation.

Reviewer
Ann Wintle

Comments

This paper provides a useful summary of the general types of curves used for fitting growth curves. The reader can find a similar criticism of the use of inappropriate fitting procedures in a recent paper by Grün (1996) where he suggests that the use of linear fitting to the "apparently linear" part of an exponential growth curve can produce large systematic errors. In his paper, Grün draws attention to the use of combined regeneration and additive dose data sets, as used in the "Australian slide method" (Prescott et al., 1993) and by Sanzelle et al. (1993). This approach is generalized in equation (7) of Guibert et al (above), where allowance for a sensitivity change between the data sets is made.

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Improvements in sample preparation for the fine grain technique

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Fine grain sample preparation was originally developed by Zimmerman (1971). A more convenient method using water flotation was subsequently proposed by Weida & Junding (1989).

The standard preparation technique we have followed in our laboratory is based on Zimmerman (1971) with modifications as proposed by Frechen (1991). We prefer the 4-11m µm grain size range, because the far-travelled material (in case of loess) is mostly finer than 11 µm; by rejecting grains less than 4 µm, the clay content is effectively eliminated.

As part of an empirical testing of the validity and reliability of luminescence dating for loess/paleosol sequences of the last glacial/interglacial cycle, about 950 fine grain samples have been prepared and investigated during the last 5 years. In order to survive such an "impact", the fine grain preparation technique had to be turned into a more convenient and more rapid procedure. Labour intensive steps, like the enrichment of the right grain size and the pipetting procedure, had to be improved. Especially the need to wash the deposition tubes had to be avoided. We also substitute demineralized water for acetone for most of the working steps as suggested by Wintle & Huntley (1980).

Preparation technique

For sediments like loess or fine grained lake deposits crushing is not necessary, unless the minerals hold together by carbonates or other cements. Cemented sediments or volcanics have to be gently crushed with mortar and pestle.

Coarse grained particles (>2 mm diameter) are removed by dry sieving and the remaining material is treated with 0.1 N hydrochloric acid, 0.01 N sodium oxalate and hydrogen peroxide (30%) in order to remove carbonates and organics. After every step the material is washed 3-4 times with demineralized water and is centrifuged.

Then the grain sizes are separated in order to obtain polymineral grains in the grain size range of 4-11 µm. The separation of the fraction < 11 µm is done under the gravitation field as described by Weida and Junding (1989). The settling time of the grains takes about 11 minutes for a water column of 7 cm. The grain size fraction >11 µm falls to the bottom of the beaker. This step is repeated several times to enrich the fraction <11 µm in the solution above the bottom.

The enrichment of the grains > 4 µm is carried out in a centrifuge using a method developed for the separation of the clay fraction for x-ray diffraction analysis of soils (Schweitzer 1992).

The physical background and the formula for calculating the settling of the grains and the centrifugation time for other grain sizes or other room temperatures than 20°C have been determined following Tributh and Lagaly (1986) and Schweitzer (in prep.).

Physical background of grain size separation

Stokes` law describes the behaviour of an idealized sphere in a liquid subject to the force of gravity.

The velocity of descent (v) increases until the frictional resistance is equal to the difference between the gravitation force and the force of lift; hence:

$$3\,\pi\,\eta_0\,d\,v = V\,g\rho - V\,g\,\rho_0 \qquad (1)$$

V = Volume of sphere, ρ = density of sphere, ρ_0 = density of liquid, g = acceleration by gravity, η_0 = viscosity of liquid (see table 1), d = diameter of sphere.

temp[°C]	$10^3\,\eta_0$ [kg m^{-1} s^{-1}]	temp [°C]	$10^3\,\eta_0$ [kg m^{-1} s^{-1}]
16	1.109	21	0.9779
17	1.081	22	0.9548
18	1.053	23	0.9325
19	1.027	24	0.9111
20	1.002	25	0.8904

Table 1: Viscosity coefficients of water in relation to different temperatures

In a centrifuge the acceleration by gravity (g) is unimportant and is replaced by the centrifugal acceleration (a_c):

$$3\,\pi\,\eta_0\,d\,v = V\,a_c\,\rho - V\,a_c\,\rho_0 \qquad (2)$$

$$3\,\pi\,\eta_0\,d\,v = V\,4\,\pi^2\left(\frac{U}{60}\right)^2 r\left(\rho-\rho_0\right)$$

for U = revolution per minute, r = distance between rotation axis and sphere.

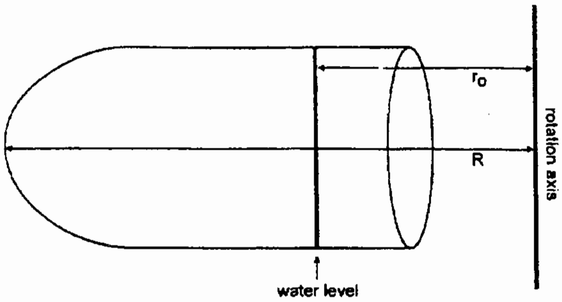

Figure 1

Standard glass tube used for centrifugation (r_0: distance between rotation axis and sphere; R: distance between rotation axis and bottom of the glass tube).

The velocity of each single grain depends on its distance from the rotation axis. Thus equation (2) is only valid for a moment, a very short time/distance interval. For our purpose the formula has to be integrated within the range through which the sphere of defined size might fall (from water surface to bottom of the glass tube (R - r_0), see figure 1), hence we get the centrifugation time (t):

$$t = \frac{18\eta_0 60^2}{4\pi^2\left(\rho-\rho_0\right)d^2U^2}\int_{r_0}^{R}\frac{dr}{r} \qquad (3a)$$

$$t = \frac{\eta_0}{\pi^2\left(\rho-\rho_0\right)d^2\,U^2}\,ln\left(\frac{R}{r_0}\right)\,1.62\;10^4 \qquad (3b)$$

There is a small error due to the acceleration and the deceleration of the centrifuge. The error is minimized, when the time of acceleration and the time of deceleration is equal.

Practical grain size separation

It is important to use the grain size <63 µm. Therefore wet sieving is suggested before using the centrifuge. Larger grains and a too large volume of grains would press the finer material to the bottom during the settling process. It is necessary to repeat the centrifugation of the sample several times.

In order to enrich the grain size fraction <11 µm settling is carried out in demineralized water under the gravitation field. The sediment is filled into a beaker (with a max. content of at least 600 ml).

Equation (1), formula of Stokes, results in the settling time (t):

$$t = \frac{18\;\eta_0\,h}{\left(\rho-\rho_0\right)g\,d^2} \qquad (4)$$

h = height of water in the beaker, ρ and ρ_0 = density of sphere and water, g = acceleration by gravity, η_0 = viscosity coefficient of water, d = diameter of the grain.

We work under the following conditions:

$h = 0.06$ m, $g = 9.81$ m/s^2, $d = 11 * 10^{-6}$ m and a water and room temperature of 20°C, resulting in a viscosity coefficient of $\eta_0 = 1.002 * 10^{-3}$ kg m^{-1} s^{-1}.

For sediments ($\rho = 2.65 * 10^3$ kg/m^3) a settling time of 9 min 13 s is necessary to optimize the grain size separation; for volcanic glass ($\rho = 2.45 * 10^3$ kg/m^3) a settling time of 10 min 29 s is needed due to the lower density of volcanic glass. Afterwards the suspension containing the fraction <11 µm is tipped into a beaker. This operation is repeated about five times. It is important to keep about 2 cm of the liquid in the centrifuge (beaker), because coarser material can be stirred up from the bottom. Coagulation of single grains is avoided by a large water volume.

The next operation is necessary to remove the grain size <4 µm by centrifugation (Fig. 1).

According to the conditions of our laboratory and by use of equation (3b) with R = 0.154 m, h = R-r_0 = 0.07 m, $\eta_0 = 1.002 * 10^{-3}$ kg m^{-1} s^{-1} (the demineralized water has to be tempered at 20 °C), $\rho = 2.65 * 10^3$ kg/m^3, U = 793 rpm (for volcanic glass with $\rho = 2.45 * 10^3$ kg/m^3: 846 rpm), $\rho_0 = 1.0 * 10^3$ kg/m^3, $d = 11 * 10^{-6}$ m we have determined the centrifugation time to $t \sim 60$ sec.

This results in the grain size fraction 4-11 µm being enriched at the bottom of the tube and the supernatant liquid is poured away. The operation of stirring and centrifugation is repeated until the solution is clear. The calculation of grain size behaviour is based on idealized round shaped spheres. The shape of the grains will determine whether the grain size range is near to the expected value or not. Empirical verification of whether the suitable grain size based on the centrifugation time and velocity was enriched, has to be done under the microscope. This is even more important for the separation of glass shards, because of the varying shapes of volcanic glass fragments.

The results obtained by this technique are certainly more precise than those achieved by the use of acetone as described by Frechen (1991) and Zimmerman (1971), and the most striking goal, the separation by the centrifuge takes essentially less time than the standard procedure. A suitable centrifuge is the *Heraeus Sepatech* (Omnifuge 2.0 RS), which is used in our laboratory.

Preparation of fine grain discs

After having prepared the right grain size, 1 ml suspension of the 4-11 µm polymineral fraction (200-250 mg/120 ml acetone) is pipetted onto each aluminium disc and allowed to evaporate in a dry box at 40° C. A theoretical monolayer of grains of 4-11 µm diameter is achieved by 100 mg per 100 ml of acetone. We use about the double concentration because a part of the material settles under the discs. Batches of 90-100 discs are prepared for each sample since additive dose and regeneration methods are used for each sample. Fading experiments are carried out for an extra batch of 50 discs in order to allow five different irradiation steps.

To avoid the labour intensive cleaning of glass tubes we constructed and built an acetal (=polyoxymethylen) block containing 14 * 14 holes. The block is screwed together with a base plastic sheet. In order to make the acetal block water tight and acetone tight, different seals have been tested (see Fig. 2). The best results have been obtained by using different seals of foam rubber and india rubber which are placed between the upper and lower part of the acetal block. Aluminium foil is used to avoid acetone attacking the india rubber. The upper part consisting of acetal has not been corroded by the acetone even after 12 months.

After all the components are assembled, an aluminium disc is placed in each hole. As the discs are 10 mm in diameter, a drill width of 11 mm was chosen for each hole. Altogether 196 discs can be prepared at the same time. After unscrewing the acetal block the aluminium discs can be picked up from the aluminium foil. Later on the acetal block is cleaned by demineralized water and the aluminium foil has to be replaced. The time needed for cleaning the glass tubes is avoided and the results of the sedimentation process is excellent. For the reproducibility from disc to disc a typical range of values is 5-10% for eolian loess samples.

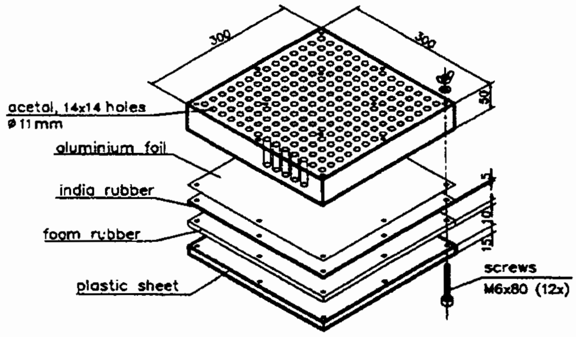

Figure 2
*Acetal (= polyoxymethylen) block containing 14*14 holes for peparation of fine grain discs.*

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Reviewer
Martin Aitken

Thesis Abstract

Thesis title : Thermoluminescence of feldspars.Tunnel afterglow and thermoluminescence emission in the infrared spectral range - Effect on the feldspars dating
Author : Antoine ZINK
date : 7 mars 1996
degree : Docteur de l'Université Bordeaux III - Michel de Montaigne
speciality : Physic applied to Archaeology

Abstract:

Too many feldspars cannot be dated by thermoluminescence, as they display an anomalous fading of their natural thermoluminescence. To understand the presence of this anomalous fading, tunnel afterglow was investigated in some forty feldspars from various origins.

Feldspars were observed to display at lower temperature a tunnel afterglow with quite specific features: its emission is entirely in the near infrared (IR) around 710 nm, it is remarkably intense due to an effect of thermal quenching. At high temperature, in the TL proper, the known emissions between 200 and 600 nm, so-called 'blue' spectral range, could be observed. But we could observe as well in TL a very strong IR component, with the same emission spectrum as in the tunnel emission. This IR emission is attributed to Fe3+.

Systematic investigations were made for an evaluation of respective stabilities of 'blue' and 'IR' TL emissions during storage. The 'IR' TL was observed to be much more stable, and not to fade during the storage, while the 'blue' did.

The two emission bands are bleached by the same wavelengths. Activation energies are similar for the 'blue' and 'IR' emissions. The two bands are strongly coupled. We related the tunnel afterglow with the microscopic structure of feldspars. Disordered feldspars, such as the sanidines display an intense tunnel afterglow. 'Ordered' feldspars, such as the microclines display a weak tunnel afterglow.

Natural TL emission may be observed in the infrared band, even when the 'blue' has faded. The variation of the intensity of the TL with the dose is similar for the two emission bands. If sanidines cannot be dated using the faded 'blue' emissions, measurements made with the 'IR' emission on several samples give a calculated natural dose of the same order as the dose estimated.

The main conclusions are the followings:

- The anomalous fading in feldspars may be attributed to the tunnel effect.
- Microclines are hardly affected by tunnel effect.
- For the sanidines, the tunnel effect prevents all dating using the 'blue' emission. But they might be dated using the 'IR' TL emission. It is of interest, especially, for volcanism dating.

Notices

VICTORIA UNIVERSITY OF WELLINGTON, NEW ZEALAND

RESEARCH SCHOOL OF EARTH SCIENCES
DEPARTMENT OF GEOLOGY

POST-DOCTORAL POSITION (LUMINESCENCE DATING)

In conjunction with a Late Quaternary lake drilling programme (Long Core Paleoclimate Records from New Zealand Lakes), a luminescence (TL/OSL) dating laboratory is being established at Victoria University Wellington, New Zealand. We seek to appoint a Post-Doctoral Fellow to establish the laboratory and to participate in the lake drilling project. This position will suit a recent PhD graduate from a recognised luminescence Programme/Laboratory seeking to establish a research profile. The appointee will be expected to participate fully in the general academic life of the Department of Geology and the Research School of Earth Sciences. The appointee will be encouraged to maintain research interests independent of the lake drilling project.

The 'Long Core Paleoclimate Records from New Zealand Lakes' project is a constituent project within the IGBP-PAGES PEP-II transect. The project aims to recover a series of long cores (100m +) from a variety of lacustrine, bog and estuarine environments, to reconstruct the climate history of New Zealand, over the last several hundred thousand years. Luminescence dating, supported by radiocarbon and tephrochronology where appropriate, will provide the primary chronology. There will be a challenging variety of dateable materials and potential for both basic research in the luminescent properties of the various sediments and in geological histories from luminescence chronologies.

The position is available from January 1997 and we hope to fill it by 1st March 1997 at the latest. It is initially tenable until June 30th 1998. Subject to satisfactory progress and continued funding, the Post-Doc will be extended for a further two years until June 30th 2000.

The base salary is NZ$41,000 but appointments may be made up NZ$44,000 depending on qualifications and experience. NZ$1.00 = US$0.68 approx.

Applicants should send a CV and the names, addresses and fax or email addresses for THREE referees.

The deadline for applications is October 10th 1996, but applications will continue to be accepted after the deadline until the post is filled. If submitting an application after October 10th, please apply by email or fax and arrange for your referees to send their letters of recommendation directly to us by email or fax.

Further information about the position, the laboratory and the project may be obtained from :

Dr Jamie Shulmeister,
Department of Geology,
Victoria University
(James.Shulmeister@vuw.ac.nz)

or

Dr Paul Froggatt,
Office for Research,
Victoria University
(Paul.Froggatt@vuw.ac.nz).

Flint Study Group formed at Canberra LED

The importance of flint as a dating tool in archaeometry has led to strong interest in the material science of flint among some members of the luminescence and electron spin resonance dating community. Informal conversations among LED attendees led to the formation of an electronic-mail based study group conceived initially with a view toward luminescence phenomena in flint. Subsequently, electron spin resonance in flint has already become an added aspect. Discussions led us to the idea that a study group might promote the development of collaborative research projects among study group members in both fundamental and applied dating studies of flint. The primary aims of the group are to promote an exchange of ideas, and to establish an open forum in which questions can be addressed.

Some of the topics of interest discussed at Canberra by current group members were:

Optically stimulated luminescence in quartz phases vs. other silicon dioxide phases in flint Stimulation wavelength dependence of luminescence in flint Emission wavelength studies of thermoluminescence and optically stimulated luminescence in flint Geochemical aspects of electron spin resonance in flint Sensitivity of electron spin resonance and luminescence in flint to different forms of radiation dose

The founding members who signed on at Canberra, and some new members are listed below. Those who wish to join in are welcome to contact by e-mail either Jack Rink (rinkwj@mcmaster.ca) or Julie Rees-Jones (reesj@mcmaster.ca) at McMaster Geology. You will be added to the list and sent an e-mail acknowledgement which contains all of the members' e-mail addresses. Please encourage any of your interested colleages to join in, particularly if they are not already within the dating-specialist circle.

Founding Members:

Richard Bailey
Ian Bailiff
Michelle Clark
Julie Rees-Jones
Tetsuo Hashimoto
Matthias Krbetschek
Andreas Lang
Norbert Mercier
Naomi Porat
Helen Rendell
Jack Rink
Ed Rhodes
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