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Exponential regressions for TL/ESR using regenerated dose response curves

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Introduction

For some years there has been much effort in improving exponential regression techniques for extrapolating the ESR/TL dose response curves (henceforth DRC) when using the additive technique, e.g., Mejdahl (1985), Debenham (1985), Berger et al. (1987), Poljakov and Hütt, (1990). However, at the same time it was recognized that in many cases the dose response curve were not strictly of the saturating exponential form neither in TL (e.g., comments of McKeever in Bulur and Özer, 1992) nor in ESR (Grün, 1991). As a result all errors evaluated from applying an exponential regression are underestimated (see Grün and Rhodes, 1991). Some specific solutions have been proposed, e.g., in the case where the dose response curve shows an exponential domain at low doses and a linear domain at higher doses (Berger, 1990; Grün, 1990). However, several mathematical functions would be necessary for extrapolating the various types of dose response curves that are experimentally encountered. For some years to come those functions will remain no more than acceptable approximations, because the basic phenomena are still partly unknown, are complex and the associated parameters are widely scattered - even for a given variety of minerals.

This paper deals with the case where the dose response curve of minerals which have been zeroed by heat in the past, such as volcanic materials, seem to be a single saturating exponential function at first glance. This kind of shape is very common, although the studied dose-range must be limited to low doses in many cases because changes in the shape occur when the dose is increased above a certain limit (e.g. a 'second rise').

The present work was intended to test a technique of regression in which account is taken of the regenerated dose response curve: the basic assumption is that the dose response curve obtained with a laboratory reset sample (for brevity referred to here as the 2nd DRC) is often a close approximation to the additive dose response curve (1st DRC), with, in most cases, allowance for a scaling factor. This assumption of proportionality between the two growth curves had been already used by Valladas and Gillot (1978) in the normalization technique. Consequently, the 1st DRC will be fitted with a function in which some parameters derive from the fitting of the 2nd DRC. In the present work, a single saturating exponential function has been used in the fitting procedure, because it is commonly applied and has a realistic physical meaning but the principle can be extended to other, more developed functions. The technique (hereafter denoted *2+1*) is illustrated by 2 simulations on test data from (i) a calculated curve, which seems to be exponential but is not strictly exponential and (ii) an experimental growth curve. An experimental application to dating will be presented in a forthcoming paper.

The test data

The simulated dose response curve.

The calculated test curve was adopted from Li (1991); which consists of two overlying saturating exponential functions,

$$I_1 = 443 \{ 1 - \exp(-0.00268D) \}$$

$$I_2 = 443 \{ 1 - \exp(-0.0134D) \}$$

where, D(Gy) is the radiation dose and I (a.u.) is the TL/ESR intensity.

A curve of this type may be proposed when two trapping processes occur simultaneously at one defect site or when two different minerals are mixed. In order to study various shapes of dose response curves in a variety of situations, three combinations of the two functions were used, namely $0.25I_1+I_2$, I_1+I_2 and $I_1+0.25I_2$, and referred to here as 0.25/1, 1/1 and 1/0.25

Figure 1.
Example of calculated test curves. The test function is a combination of I_1 and I_2 ($I = I_1 + I_2$ in the present case) where,

$$I_1 = 443\,\{1 - \exp(-0.00268D)\}$$
$$I_2 = 443\,\{1 - \exp(-0.0134D)\}$$

The shift of the test curve along the dose axis allows to simulate different preset palaeodoses, i.e., 50, 100, 200 and 300 Gy (as shown).

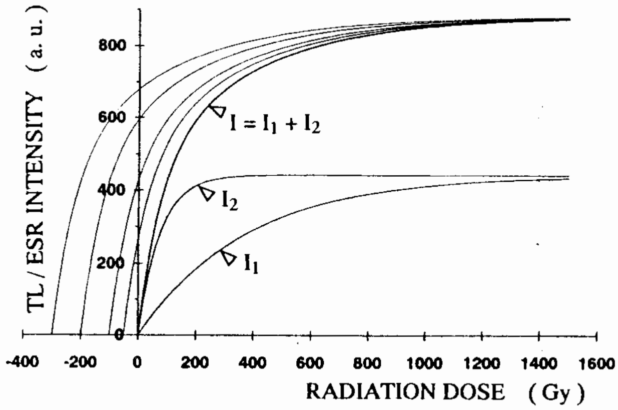


Figure 2.
Test curve $I_1+0.25I_2$; preset palaeodose (PP): 200Gy. Black boxes: 2nd DRC test points; open boxes: 1st DRC test points; no errors on the data; P_{S1}: examples of extrapolations on 1st DRC only; P_{2+1}: examples of extrapolations with the 2+1 technique. Solid lines represents the theoretical growth-curve.

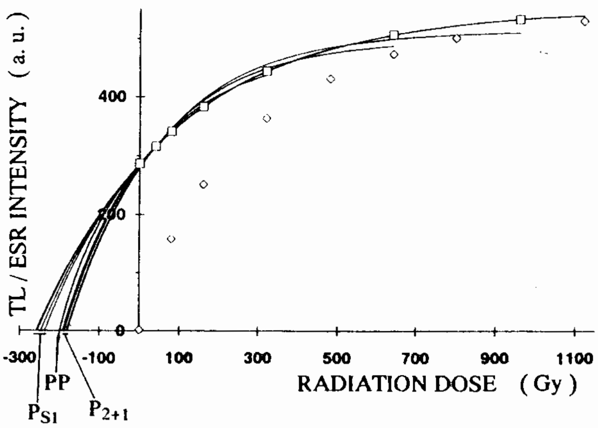
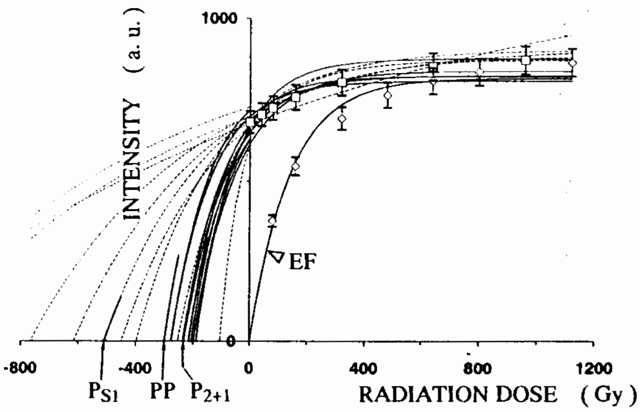


Figure 3.
Regression on a simulated test curve 1/1, with 5% standard deviations on intensities; preset palaeodose (PP): 300 Gy; maximal dose taken into account on 1st DRC: 960 Gy. P_{S1}, P_{2+1}: as in fig.2, the most probable value (the value obtained without errors). EF: exponential fit on 2nd DRC, using mean points. Dashed lines: ten first curves calculated with S1; solid lines: ten first calculated curves with 2+1.



(table 1); the preset palaeodose was varied from 50 to 300 Gy (fig. 1). Simulated experimental points were fixed at doses of 0, 40, 80, 160, 320, 640, 800, 960 and 1120 Gy. Several ranges of doses were used for curve fitting (see bottom of table 1). For every case, the range of doses for the second growth was taken in accordance with the usual choice, i.e., roughly equal to the total dose range of the first dose response curve (i.e. estimated palaeodose + maximum added dose).

The experimental dose response curve
The experimental set of data points was taken from the measured red TL peak (~620 nm, at 380 °C) of quartz grains annealed (at 400 °C for 15h) and afterwards irradiated in the laboratory. The standard deviation of the intensities was evaluated from repeated measurements (8-10 for each dose). The curve of peak height vs dose was used as a 2nd DRC (as in fact it is) and also as a 1st DRC by a shift along the dose axis of 145 or 290 Gy (see table 2). Thus, the palaeodose obtained by regression of the 1st DRC should be equal to the amount of dose shift, i.e. 145 or 290 Gy (fig.5).

In both cases, the two curves (1st and 2nd DRC) were adjusted to the same scale, so that the curves could be superimposed on each other by a shift equivalent to the palaeodose along the dose axis, i.e., no sensitization was simulated. The results given here would not be altered by a change in sensitivity because the regression technique presented below is able to take it into account.

The *2+1* regression technique

As a first step, the 2nd DRC is fitted using the function

$$I = I_{1max} [1 - \exp(-D/D_o)] \qquad (1)$$

where,
I_{1max} is the maximum intensity, D the dose and D_o the characteristic saturation dose. In the second step, the 1st growth is fitted using the function

$$I = I_{2max} [1 - \exp\text{-}\{(D - D_e)/D_o\}] \qquad (2)$$

where, D_e is the palaeodose to be evaluated; I_{2max} is usually different from I_{1max} because of sensitization after annealing.

The coefficients I_{1max}, I_{2max} and D_o are obtained by minimization - by a simple and rapid method of successive approximations - of the sum of the squares of the relative differences between the experimental points and the fitting curve. The use of relative differences is justified when the relative uncertainty $\Delta I/I$ (I = TL or ESR intensity) is constant, combined with a significant variation of amplitude of I in the given dose range (Nougier, 1985). Minimization of the sum of square absolute differences would give too much importance to the largest intensities. That is, the fitting curve would lie closer (relatively) to the high intensity points than to the lower ones; this effect is avoided by the use of the relative differences.

In our laboratory, the standard deviation of the calculated palaeodose (s.d.) is obtained by use of a Monte-Carlo technique (Pilleyre, 1991). Assuming that the error on the dose is negligible, the uncertainty of the intensity is estimated. The process of curve fitting is then repeated many times (e.g., 100 times) by randomly generating data sets that show a Gaussian distribution around the mean intensities of the experimental data set. The s.d. of the palaeodose is then derived from the distribution of the calculated palaeodoses. The palaeodose itself is calculated with the set of points corresponding to the mean intensities. This most probable palaeodose is generally not equal to the mean of the palaeodoses calculated with the Monte-Carlo technique. A Monte Carlo technique was also adopted by Grün and Rhodes (1991).

In the present work, no uncertainties were taken into account in the first (theoretical) simulation (but in one case specified below), the points belonging to the calculated test curve; standard deviations (one sigma) were used in the second (experimental) simulation.

Results

Figure 2 illustrates the regressions and table 1 lists the results. As already specified, different regressions were calculated by varying the maximum dose (see table 1). It can be observed that very few extrapolations yield the correct palaeodose but, in nearly all cases, the exponential regressions on 1st DRC only (henceforth *S1*) gave worse results than the *2+1* technique . On the other hand, the lower the maximum dose taken into account, the better the results. This effect is more marked on *S1* extrapolations, where increasing the maximum dose has a strong influence on the calculated palaeodose. At the same time, the effect of the palaeodose value can be observed (table 1): when the test palaeodose for a given test curve is higher, the 1st DRC

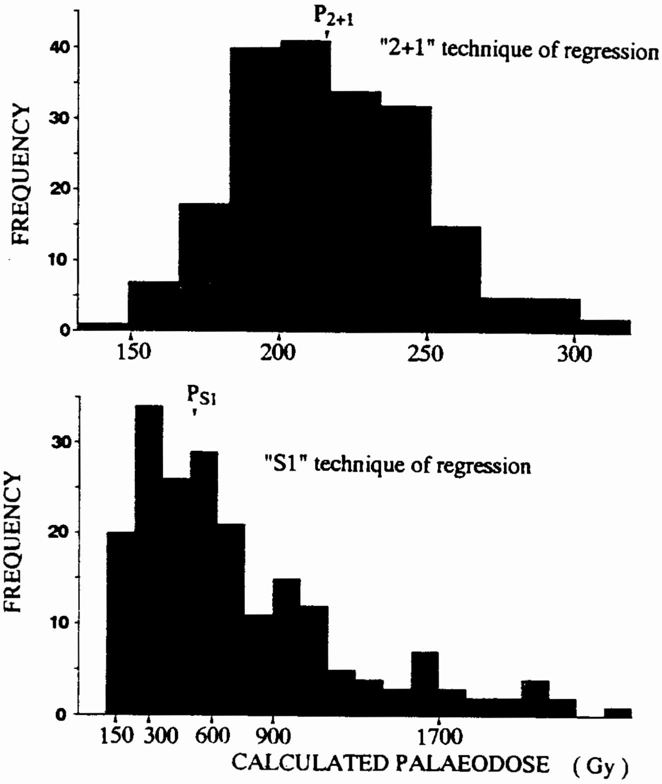


Figure 4.
Distribution of the palaeodoses calculated with the Monte-Carlo technique (200 calculations); maximal dose taken into account: 960 Gy; error limits of 5% on intensities (table 1). Preset palaeodose (underlined): 300 Gy; P_{S1}, P_{2+1}: most probable palaeodoses (= palaeodose obtained without errors on the data), with the two techniques of regression, i.e. 2+1 (upper figure) and S1 (lower figure).

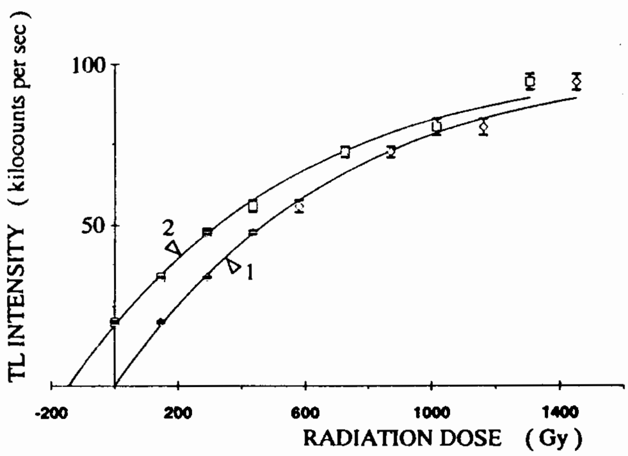


Figure 5.
Experimental test data: red TL of quartz grains annealed in the laboratory and irradiated with a ^{137}Cs gamma source; red filter RG 610 Schott, 5 °C/s heating rate. Errors corresponding to one s.d. derive from several measurements for every dose. 1: 1st DRC, test palaeodose: 145 Gy; 2: 2nd DRC. Full lines: exponential fits (derived from 1st DRC).

lies closer to the saturating domain and the single fit *S1* is less relevant, although it might lie very near to the test points and seem, in this part of the curve, very good from a mathematical point of view.

It was verified that, when the test curve is purely exponential (I_1 or I_2 alone), both techniques give correct results.

The drawback with the above calculations is the lack of consideration of experimental errors, leading to situations far from actuality, especially for the highest palaeodoses. An attempt was made to simulate such situations by use of Monte Carlo calculations, after an arbitrary standard deviation of 5% had been applied to the intensities of the test points. A particularly critical case was selected by taking the highest palaeodose (300Gy) and the 1/1 combination (fig.3).

Afterwards, the histograms of calculated palaeodoses with 200 draws were plotted (fig.4). It can be seen that the *S1* procedure gives an erroneous palaeodose with a very broad distribution, ranging from 90 to 2790 Gy. Moreover, the distribution is clearly non-Gaussian and asymmetric - as already mentioned by Grün and Rhodes (1991) - making the mean value and the s.d. given at the bottom of table 1 almost meaningless. On the contrary, an approximately correct palaeodose value can be obtained with the *2+1* technique, in spite of the very unfavourable conditions. The corresponding probability distribution being not far from normal, the s.d. can be considered as realistic. Comparison of the mean results from the Monte Carlo calculations (in square brackets in the tables) and the most probable value (without brackets) gives a rough indication of the asymmetry of the palaeodose distribution.

Table 2 lists the palaeodose results obtained with the experimental test data, with the Monte Carlo calculation (100 draws). As above, the results obtained with the *2+1* technique are, in most cases, closer to the test palaeodose than with a single fit *S1*. The s.d. is larger with the latter fit but often not large enough to allow compatibility between the calculated and the preset palaeodoses. In this simulation, the mean calculated palaeodose is, in all cases, not significantly different from the most probable one.

Table 1.
Summary of the palaeodoses calculated with two different regression techniques; the ratio of the calculated and the preset palaeodose is given. *S1*: exponential fitting on 1st growth only, with minimization of the sum of the square relative differences between the fitting curve and the test points. *2+1*: exponential fitting of 1st growth with use of the 2nd growth parameter (see text). 3 different combinations of the functions I_1 and I_2 were used for the calculated test curve. The dose points taken into account are listed at the top of the table; for the 1st growth, those are the natural signal and the natural +6, +5, +4 or +3 added doses; for the 2nd growth, the zero point and 7, 6, 5 or 4 different doses.

The mean calculated palaeodoses obtained with the Monte-Carlo technique (100 draws) are given in square parentheses in the lower part of the table, where a s.d. of 5% has been taken into account for the intensities. The palaeodoses calculated with the mean points are given in the preceding row(s).

Test curve combination	Preset Palaeodose	Calculated Palaeodose / Preset Palaeodose							
I_1/I_2	(Gy)	*S.1*	*2+1*	*S.1*	*2+1*	*S.1*	*2+1*	*S.1*	*2+1*
0.25/1		1.17	1.06	1.13	1.06	1.07	1.06	1.03	1.05
1/1	50	1.36	1.13	1.29	1.13	1.16	1.14	1.08	1.12
1/0.25		1.28	1.13	1.23	1.13	1.15	1.14	1.08	1.12
0.25/1		1.35	1.00	1.27	1.01	1.15	1.03	1.07	1.03
1/1	100	1.57	1.02	1.47	1.03	1.27	1.07	1.14	1.07
1/0.25		1.32	1.03	1.28	1.03	1.20	1.06	1.13	1.07
0.25/1		1.88	0.87	1.71	0.89	1.42	0.93	1.23	0.96
1/1	200	1.74	0.84	1.67	0.86	1.48	0.90	1.32	0.94
1/0.25		1.30	0.90	1.28	0.91	1.23	0.94	1.19	0.96
0.25/1		2.16	0.75	2.03	0.77	1.70	0.81	1.39	0.84
1/1	300	1.70	0.72	1.66	0.74	1.60	0.78	1.54	0.81
1/0.25		1.22	0.84	1.26	0.83	1.24	0.86	1.25	0.88
1/1	300	1.70	0.72			1.60	0.78		
		±1.86	±0.11			±2.40	±0.22		
with 5% s.d		[2.55]	[0.74]			[2.40]	[0.81]		

Radiation dose points (Gy)					
	1st growth	0, 40, 80, 160, 320, 640, 960	0, 40, 80, 160, 320, 640	0, 40, 80, 160, 320	0, 40, 80, 160
	2nd growth	0, 80, 160, 320, 480, 640, 800, 1120	0, 80, 160, 320, 480, 640, 800	0, 80, 160, 320, 480, 640	0, 80, 160, 320, 480

Another semi-experimental simulation was made identical to the second one but with another quartz sample and a larger dose range and the results were qualitatively very similar to the preceeding ones, with more pronounced tendencies to give completely erroneous palaeodoses by fitting on the 1st DRC only. The shape of the growth curve appeared bi-exponential.

In the two simulations reported here, slightly better results were obtained with the simple regression (*S1*) in the special case of low palaeodoses and narrow dose range of extrapolation (bottom, right of the tables) but in these cases, the two palaeodoses calculated with the two regression techniques were close to each other.

Discussion

The first example studied here can only give an indication of what can happen in reality because only a specific type of growth curve and fit was studied. However, it shows that, if the function chosen does not correspond exactly to the physical phenomenon to be fitted, large systematic errors can affect the calculated palaeodose. These errors can be reduced by using the shape of the 2nd DRC. Recognizing from the 1st DRC the appropriate function to be used, e.g., in the present case, a bi-exponential one, would evidently improve the results by application of an adapted treatment like the one proposed by Li (1991) or by Bulur and Özer (1992). In practice a major difficulty comes from experimental uncertainties, which, mostly at the onset of saturation, can accommodate a wide variety of fitting functions.

The use of the 2nd growth data will significantly reduce the freedom of movement, mainly in the dose range of interest, i.e., below the dose equivalent to the palaeodose and far from saturation. But a frequent drawback in the application of the *2+1* technique to experimental results is the effect of sensitization, which not only results in enhancement (generally) of the height of the signal for a given dose after annealing, but in frequent modifications of the shape of the dose response curve. Other reasons such as a differential behaviour under alpha and gamma irradiation can also modify the shape of the 2nd growth (see Aitken, 1985, p.139). In practice, the *2+1* technique can be used provided the whole fitting curve derived from the 2nd DRC lies within error limits of the experimental points of the 1st DRC. If this is not the case, no reliable palaeodose can be calculated by this technique and the situation reverts to that which obtains when only the 1st DRC is available.

Table 2.

Evaluation of the error associated with the determination of the palaeodose is not a simple task and, as outlined by Grün (1991) in the field of ESR dating, "little attention has been paid to the correct estimate of this parameter". It could be added, that most of the literature deals with standard deviations deriving from the regressions, but not with systematic errors generated by the use of improper functions. The present simulation suggests that those systematic errors can be substantially greater than the s.d.; it then appears illusory to make great efforts in improving the rapidity and performances of regression algorithms unless they are more relevant from a physical point of view. For the same reasons, it is not worthwhile to search for a perfect definition in the distribution of the palaeodoses calculated with the Monte Carlo technique by increasing the number of draws. A realistic estimation of the s.d. will not require more than 100 draws, and this is available with a very acceptable duration of calculation with a PC computer (see note at end of paper).

An additional improvement of the technique would be in taking into account the different precisions of the experimental points by weighting them by inverse variance (expressed in percentage of the intensity in this case), such as tested by Grün and Rhodes (1992). As concerns TL, no important modifications of the results can be expected because, as mentioned above, the relative uncertainty of the intensity is nearly constant for a given sample.

As illustrated on fig. 4 and in table 1, the use of the 1st DRC only results in very large uncertainties in unfavourable cases - where the maximum irradiation dose is small and the growth-curve is at the onset of saturation; because this situation is sometimes seen in the literature, this caution is emphasized.

The fact that reducing the dose range of the two growth-curves towards low doses in the regression improved the results can be qualitatively explained; as the dose range is reduced, the fit to the curvature improves, also leading to an improvement in the estimation of the palaeodose. However, as shown by Grün and Rhodes (1991), this is not always the case and reality is more complex. But we want to emphasize this principle for many cases and particularly when it seems more or less evident; e.g., *Idem* table 1, for the experimental test data (see fig.5), with a Monte Carlo technique (100 draws). Errors are quoted at one s.d..

Preset Palaeodose (Gy)	Calculated Palaeodose / Preset Palaeodose							
	S.1	*2+1*	*S.1*	*2+1*	*S.1*	*2+1*	*S.1*	*2+1*
145	1.18±0.06 [1.18]	1.05±0.04 [1.04]	1.10±0.07 [1.12]	1.03±0.04 [1.03]	1.15±0.07 [1.16]	1.03±0.04 [1.03]	1.11±0.09 [1.11]	1.01±0.05 [1.02]
*1st Growth	0, 145, 290, 435, 725, 1015, 1305		0, 145, 290,435, 725, 1015		0, 145, 290, 435, 725		0, 145, 290, 435	
*2nd Growth	0, 145, 290, 435, 580, 870, 1160, 1450		0, 145, 290, 435, 580, 870, 1160		0, 145, 290, 435, 580, 870		0, 145, 290, 435, 580	

* *Radiation dose points (Gy)*

Preset Palaeodose (Gy)	Calculated Palaeodose / Preset Palaeodose							
	S.1	*2+1*	*S.1*	*2+1*	*S.1*	*2+1*	*S.1*	*2+1*
290	-	-	1.10±0.11 [1.11]	0.91±0.04 [0.91]	0.95±0.12 [0.97]	0.95±0.05 [0.96]	0.98±0.14 [1.00]	0.89±0.06 [0.90]
*1st Growth			0, 145, 290, 580, 870, 1160		0, 145, 290, 580, 870		0, 145, 290, 580	
*2nd Growth			0, 145, 290, 435, 580, 870, 1160		0, 145, 290, 435, 580, 870		0, 145, 290, 435, 580	

when a 'second rise' occurs. Above a certain dose, the corresponding dose range can be discarded from regression by considering that the physical phenomenon which becomes dominant at high doses is unimportant in the low dose range. When high dose points have a negative influence on the extrapolation, this effect will be enhanced when the absolute differences between the points and the fitting curve (and not the relative ones) are used in the regression.

The second (experimental) simulation illustrated two aspects of this discussion. Firstly, although the dose response curve seemed to be nearly exponential, it was sufficiently different from an exponential to induce a significant error when the 1st DRC only was considered in the extrapolation and, secondly, the calculated error did not take into account this systematic error.

In order to get a crude estimation of the systematic error, we computed the mean value r of the ratios (calculated palaeodose/preset palaeodose) for the two techniques and for the two examples (i.e. table 1 and table 2). The results were:

Table 1: $r_{S1} = 1.36$ (s = 0.26); $r_{2+1} = 0.96$ (s= 0.12)

Table 2: $r_{S1} = 1.08$ (s = 0.08); $r_{2+1} = 0.98$ (s= 0.06)

It appears that an important systematic overestimation of the true palaeodose can be expected with an extrapolation of the 1st DRC only (*S1*), whereas this systematic error remains within acceptable limits for the *2+1* technique. For the time being, it is necessary to assess a systematic error to be quadratically added to the random error in the quotation of the overall error using the *2+1* technique; the intermediate value of ± 6% can be used provisionally.

Conclusion

It has been demonstrated, with two examples, that extrapolation of an additive growth curve can result in a completely erroneous palaeodose when the fitting function is taken to be exponential but does not in fact accurately represent the dose response of the sample. Generally, results are significantly improved when the shape of the fitting curve is derived from the shape of the second dose response curve, as in the *2+1* technique. Growth curves obtained with experimental data are rarely strictly saturating exponential but the width of experimental errors often hinders an appreciation of the exact mathematical description of the dose response curve. However, a necessary further step of improving the *2+1* technique will consist in developing more suitable fitting functions. We recommend a pragmatic approach such as the *2+1* technique of regression since it is more realistic than some sophisticated techniques applied to the 1st DRC only, even if the curves derived from those techniques seem to provide better mathematical fits to the available data. At the same time, the simulations presented here should encourage one to be very generous with the size of the errors quoted on TL/ESR ages!

Note on computing specification. Calculations were made using a program written in *Turbo Pascal* and running on a PC with a 80386SX CPU and 80387 (16MHz) coprocessor The overall duration for 100 draws ranged from a few seconds and a few tenth of minutes, depending on the shape of the curve.

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PR Rainer Grün

The paper of Sanzelle et al. shows clearly the danger of estimating incorrect D_E values if an incorrect mathematical model is applied for fitting the data of the dose response curve using the additive dose method. The suggestion of applying curve parameters derived from a second dose response curve after the sample was reset is one way to overcome some of the problems to some extent. A similar technique was presented by Prescott et al. (1992) who combined the additive data set with a regenerated dose response curve ("Australian slide method"). This procedure has the general potential to become nearly independent of mathematical assumptions for curve fitting and, hence, the problems that have been outlined in this paper may be overcome.

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