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Normalization of inclusion size quartz TL data

A. D. Franklin and W. F. Hornyak

Physics Department, University of Maryland, College Park, Maryland, 20742-4111 USA

A series of experiments is reported on TL observed with quartz inclusion size grains (~100 µm diameter). The relative standard deviation of the peak height of the Slowly Bleaching Peak (SBP) varies inversely as the square root of the sample weight. The rate of this variation indicates that only about 8% of the grains produces the observed TL. Little correlation was found among peak heights for the 70 °C, SBP, and the Rapidly Bleaching Peak (RBP) for first or subsequent glow cycles. These results indicate that normalization of TL data in large quartz grains by reference to any of these three peaks results in at least as large scatter as does weight normalization alone. However, a drastic HF treatment was found to strongly reduce the scatter in the TL data peak heights. Weight normalization, perhaps using some variant of this effect, with as large specimens as possible seems the best course for obtaining reliable data from inclusion size grains.

Introduction

The TL data from 100 µm quartz particles, when normalized by weight, exhibits large run-to-run scatter for aliquots from the same sample. This appears to reflect the fact that only a few of the particles in the aliquot produce most of the TL (Benko, 1983; Huntley and Kirkey, 1985; Spooner et al., 1988). This scatter represents a significant limitation on the precision obtainable in TL dating using the inclusion method (Fleming, 1979), employing 100 µm particles. In this paper we present statistical evidence for this bright/dark model, with only a small percentage of bright particles among a majority of dark ones, and then examine several possible normalization methods for improving the precision of measurements of TL intensity. These include weight normalization combined with chemical reduction of particle size and normalization not by weight but by reference to the TL intensity of various TL peaks generated by standardized beta doses (e.g. zero glow monitoring, Aitken et al., 1979).

Experimental conditions

All material used for the experiments reported here was derived from a Middle Stone Age site in the Northwest Kalahari desert. Unless otherwise noted, all were treated with 3 N HCl to remove carbonates, 30% H₂O₂ to remove organics, and unstirred 48% HF at room temperature for 30 minutes followed by 3N HCl to remove alkali feldspars. The resulting powder was sieved and the 90-150 µm portion was fractionated by density using a sodium polytungstate solution of density 2.70 g/cm³ and centrifuging. The light fraction was retained for these experiments. The natural TL of these materials exhibits two major peaks, one at 275 °C and one at 310-330 °C (depending on filter used) for a ramp rate of 1 °C/s. These peaks can be identified with the 325 °C and 375 °C peaks reported in the literature at heating rates of 20 °C/s. The 275 °C peak is rapidly

removed by bleaching with a solar simulator, and is designated here the Rapidly Bleaching Peak (RBP). The 310-330 °C peak is much more resistant to bleaching and is designated the Slowly Bleaching Peak (SBP). Complete removal of the RBP without affecting the SBP was accomplished by bleaching with the solar simulator with a Schott VG-9 (green) filter for 4 hours. The TL data were measured using a Chance HA-3 filter plus a Corning 7-59, a Schott UG-11, or a Schott VG-9 filter with an EMI-9635Q phototube operated in the single photon counting mode. The data were collected and processed digitally. All data quoted, except as noted, are counts/(°C·mg) at the maxima of various TL peaks, averaged over at least 6 aliquots.

Statistical Effects of Bright/Dark Model

Under the simplifying assumption of just two kinds of otherwise identical particles, bright and dark, the TL per unit weight (weight-normalized) should conform to a binomial distribution. Let n be the total number of grains of density 2.65 g/cm³ in the aliquot, w the weight in mg, and p the probability that a given grain is bright. Provided that $np \geq 5$, the ratio s' of the standard deviation of the mean of the distribution relative to the mean for monodisperse quartz spheres of 100 µm diameter is given by (e.g. Li, 1957):

$$s' = 1/\sqrt{720w(p-1)}$$

Figure 1 displays a plot of s' vs. $1/\sqrt{w}$ for the SBP. A least-squares fit through the origin produced a value of $p \approx 0.08$, confirming the statistical origin of the scatter and suggesting 1 bright grain in about 10 or less. That the scatter is not instrumental was shown by exposing a similar aliquot preheated (to 500 °C) in the TL apparatus without disturbing it to 5 repeated cycles composed of an accurately reproducible Sr-90 beta dose of about 27 Gy and a TL glow run to 500 °C. The peak intensity at

about 285 °C increased about 10% in the course of these runs, approximately linearly with run number. This rise appears to reflect a small predose effect. The relative standard deviation of these data about a least squares fit to the linear increase was about 1.5%, not really different from the counting statistics value of 1.3%.

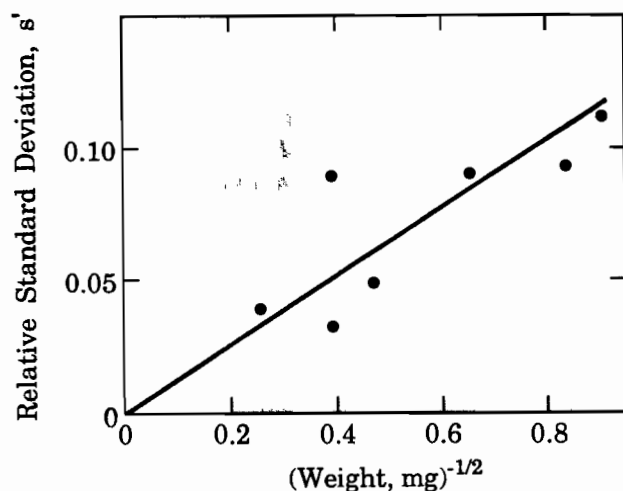


Figure 1. Relative standard deviation (of the mean) of replicate measurements of the height of the weight-normalized Slowly Bleaching TL peak from Kalahari sample KA9-1 vs the inverse square sample weight. No laboratory doses were given, but the specimens had been exposed to the solar simulator for 30 minutes.

Chemical Reduction in Particle Size

The bright/dark model suggests that improvement in precision could be made by increasing the absolute number of bright particles per aliquot. As in the experiment quoted above, this can be done by simply increasing the aliquot weight. The relative standard deviation for a single measurement fell from a high of about 30% for 1 mg aliquots to a value as low as 10% for aliquots of 15 mg. With increasing size of the aliquot, however, goes the risk of particle pile-up.

Rather than simply increasing sample weight, the scatter might also be reduced by maintaining the weight and reducing the size of the particles making up the aliquot. Starting with a sample of the 90-150 µm fraction from "natural" material (ie without added laboratory dose) we experimented with reducing the size with a drastic HF etch, 90 minutes at 70 °C. X-ray diffraction revealed an excellent quartz pattern and nothing else. The powder remaining from the HF treatment, after washing and drying, was again sieved. New fractions were obtained from 90-150 µm (i.e. not much reduction in size) to <45 µm (i.e. drastic reduction in size). There was a small drop in intensity of the SBP

with decreasing new particle size. This drop was reproduced in two separate experiments but was statistically significant only for the new <45 µm fraction.

Much more significant was an effect on the sensitivity of the 70 °C peak to a small beta dose applied before the TL run but after the etching. Because the severe HF treatment involves prolonged heating at 70 °C, a large increase in sensitivity observed for the new 90-150 µm fraction over that for the untreated material may simply reflect a pre-dose effect. However there was a drop by a factor of 3 in the sensitivity in the treated material from the new 90-150 µm to the new <45 µm fraction. The material not removed by the HF appears to possess a lower sensitivity than that removed.

The HF treatment has the desired effect of strongly reducing the run-to-run scatter when the new <45 µm fraction is compared to the untreated material. Table I sets forth the percentage standard deviations for a single aliquot from measurements involving 10 aliquots for each condition.

The changes in scatter for the SBP peak are mirrored by somewhat similar changes in the scatter of the 70 °C peak. Surprisingly, the big increase in precision occurs simply with the HF treatment without a substantial reduction in particle size. Since the majority of the treated powder lies in the 90-150 µm range, the practical conclusion is that use of the drastic HF treatment to reduce scatter in the data may be worthwhile for dating with inclusion-size particles, but not because of particle-size reduction.

Zero-Glow Normalization (Normalization with the 70 °C Peak)

An alternative to weight normalization is some variant of the zero-glow technique (Aitken et al., 1979) in which a standard test dose is used to create a TL peak at 70 °C whose intensity, a measure of the amount of sensitive material in the specimen, may be used to normalize the experimental glow curve.

In its original form a low-temperature peak was excited by a small in-situ β dose prior to running the glow curve. In quartz, in our measurements at a ramp speed of 1 °C/s, the peak at 70 °C was used. This peak represents emission at 370 nm and appeared strongly with the Corning 7-59 blue filter or even the Schott UG-11 ultraviolet filter. Several similar experiments were performed, in which the counts/second at the peak maximum (peak height) for up to 15 replicate specimens were measured and the standard deviations (s) as a percentage of the mean peak height determined. Such measurements were made for the 70 °C peak and the SBP and also for their ratio, and are shown in table 2.

Table 1. Percentage Standard Deviation of a Single Aliquot

Material	Yield (%)*	s' (%)		
		SBP	70 °C	SBP/70°C Ratio
Untreated, 90-150 µm		41	23	27
HF-Treated, 90-150 µm	74	13	11	19
HF-Treated, 45-90 µm	25	18	15	13
HF-Treated, <45 µm	1	7	11	6

*"Yield" denotes the weight percent in the various fractions produced by sieving after the HF treatment.

Table 2. Relative Standard Deviations

No. Replicated	70 °C Peak, s'(1)	SBP, s' (2)	Ratio, s'(r)
14	15.1	10.1	15.9
10	11.5	16.7	14.9
15	21.2	11.4	10.8
15	15.4	18.4	12.2

If the two peaks are totally uncorrelated s' of the ratio should be given by

$$s'(r) = \sqrt{[s'(2)]^2 + [s'(1)]^2}$$

where s'(1) and s'(2) are the standard deviations of the two peaks.

If the peaks are completely correlated s'(r) should be small, reflecting only counting statistics. The experimental values of s'(r) lay between these two extremes (col. 5, table 1; col. 4, table 2), indicating some but by no means total correlation. A number of factors might contribute to the partial independence of the scatter in the two peak intensities, which we may classify as either instrumental or aliquot factors.

Instrumental factors include uncertainty in the beta dose arising from the shutter on the beta-source and variation in particle position due to motion during the run, variation in the time of waiting between the end of the beta dose and the start of the glowing out, and scatter due to counting statistics. Aliquot factors can arise because the sources of luminescence (the electron/hole traps and recombination/luminescence centers) are not the same for the two peaks and might be distributed differently among the grains of the aliquot being measured. The same grains would not necessarily be bright for the two peaks. This reasoning extends also to the pre-dose effect, which involves "reservoir" traps that also are probably not distributed uniformly across the

grains. We must assume that changes in sensitivity triggered by heating the material during the TL measurement can also vary from grain to grain. Included with the aliquot factors is the effect of the distribution of the grains of the powder during measurement on the measured TL intensity because of pile-up and on the effective beta dose because of pile-up and changes in back-scattering. The instrumental factors were assessed in several experiments in which the same aliquot was left in place and repeatedly given a small beta dose and then glowed out to some maximum temperature, both the 70 °C and the SBP peaks being measured in separate experiments. There was a steady decrease in the 70 °C peak height, apparently linear in run number, for glow curves carried only to 150 °C. About the least squares line fitted to these numbers there was a scatter yielding a relative standard deviation of only 2.3%, very little different from the counting statistics value of 1.5%. For the SBP data peak, cycled to 500 °C, there was a small increase in intensity with run number, again approximately linear. The scatter about the least-squares line was 1.5%, again essentially all accounted for by counting statistics (1.3%). This experiment shows that of the instrumental errors, only the statistical counting error has much significance, but this makes only a small contribution to the observed scatter in peak intensities.

There are two major contributions to the aliquot error. One is inherent in the material itself, the variation in brightness of the grains of the aliquot, with the

possibility that different grains are bright for different TL peaks. The other contribution may arise from variations in the arrangement of the grains in the bowl. Because of entrained H₂O the arrangement of the grains can be altered, particularly during the first glow curve run. During subsequent runs with an otherwise undisturbed aliquot no further changes in arrangement should occur. Thus examining ratios of peaks from second and, especially, third glow curves should eliminate effects due to motion of the grains which might occur during the first glow curve run. Table 3 below summarizes the results of two such experiments. The same aliquot was left in place and given 3 cycles each consisting of a small beta dose followed by glowing out to the maximum temperature shown, the TL peaks being recorded in the process. A number of aliquots were treated in this way and the aliquot to aliquot scatter in peak heights and ratios examined.

With a maximum temperature of only 125 °C the 70 °C peak heights from successive cycles on the same aliquot are well correlated. The scatter in the ratios, however, is considerably larger than can be accounted for by counting statistics (~1.7% for the ratios). The scatter increases markedly when the maximum temperature in each cycle is raised to 500 °C, but is much more apparent between the first and second cycles. Some source of scatter is introduced by the heating, is greatest in the first heating, and increases with increasing maximum temperature.

The mean peak intensity values for the three cycles (7000, 6720, 6770 counts/(°C-mg) give no evidence at all for a pre-dose effect stimulated by the 125 °C heating cycles. Variation in the predose effect can probably be ruled out as a source of the aliquot to aliquot excess scatter over counting statistics evident for a maximum temperature of 125 °C in table 3.

For the experiment in which the maximum temperature was 500 °C the mean peak intensities averaged over the aliquots for the 3 cycles were 7550, 29622 and 33464

counts/(°C-mg). There is strong evidence here for a predose effect stimulated by the temperature cycling to 500 °C. Most of the predose increase in sensitivity takes place as a result of the first cycle. This pattern, on the assumption that the predose effect varies from grain to grain, can account for the increase in scatter in the ratios for the 500 °C cycles relative to the 125 °C cycles, and accords with the observation that the increase in scatter is markedly less for the ratio I₂/I₃ than for the other two. The same effects are seen in the product-moment correlation coefficients. These results show that the 70 °C peak during the first glow run exhibits considerable scatter, in the range 10-20%, arising from the statistical uncertainty in the number of grains in the aliquot that are bright in this peak. For cycles to 500 °C the intensities of the second and third glow peaks do not correlate well with that of the first glow peak, primarily because of a predose effect varying from grain to grain in a way similar to, but not well correlated with, the brightness for the 70 °C peak. The second and third glow peak intensities correlate with each other somewhat better than either does with the intensity of the first glow peak.

When trying to use the 70 °C peak to normalize the experimental values of intensity in the SBP and RBP (data) peaks, a further complication is possible in that the distribution of brightness among the grains may be different for the 70 °C and data peaks. Table 4 examines the relation between the natural RBP' observed in the first glow cycles and the 70 °C peak generated by a small test dose, for 3 cycles with 500 °C maximum temperature. RBP' is primed to indicate that the observed peak is actually the RBP and SBP superimposed, in about equal proportions, as seen in natural material using a combination of HA-3 and UG-11 filters. This is the same experimental sequence shown under T_{max} = 500 °C in table 3. There is no correlation between the RBP' and the 70 °C peak from any of the cycles.

Table 3. Relative Standard Deviations of 70 °C Peak Heights and Ratios

		Cycle Number			Ratio for Cycles Shown		
T Max		1	2	3	1/2	1/3	2/3
125 °C	s',%	16.4	14.6	17.5	3.9	4.9	5.6
	r				0.972	0.966	0.948
500 °C	s',%	16.6	23.1	18.5	11.3	11.4	6.8
	r				0.854	0.818	0.961

s' = relative standard deviation; r = product-moment correlation coefficient

Second Glow Normalization

By Second Glow Normalization we mean using the data peak generated in a second glow cycle to normalize the same peak observed in the first cycle. Here we made TL measurements using a combination of a Schott VG-9 (green) with a Chance HA-3 filter. There is almost no contribution to these data from the RBP, the measured TL exhibiting essentially only the SBP. A large beta dose was administered at the beginning of the second glow cycle in order to generate a reasonably intense SBP(2). No measurement could be made of the 70 °C(2) peak, which was strong enough to saturate the PM tube. Eight replicates were measured, with the sequence first glow, beta dose, second glow without disturbing the specimen. To eliminate small run to run variations in temperature, the first glow data were shifted along the temperature axis to make each TL peak coincide with one member of the set as standard (Franklin et al., 1987). The second glow data were similarly temperature shifted using the second glow TL peak of the same member of the replicate set as a standard. Averages over all replicates were then obtained for both the first and second glow data. The ratio of the first to the second glow averages produced a reasonable plateau from 230 °C to 300 °C.

With this plateau defining the temperature range we then examined the mean and standard deviation of the

counts/(°C·mg) for the first glow and second glow data and their ratio over the replicates at 10 °C intervals. The results are shown in table 5. The first and second glow peaks exhibit only partial correlation, the relative standard deviation of their ratio amounting to more than half what it would be if they were statistically independent (compare last two columns of table 5).

Indeed, the scatter in the ratios is greater than the scatter in the weight-normalized first glow data (compare columns 3 and 5). Product moment correlation coefficients for these selected temperatures ranged from 0.328 to 0.750.

Conclusions

We conclude that most of the scatter in the 70 °C peak, RBP, and SBP in 100 µm quartz aliquots arises from differential grain brightness and that this distribution in brightness among the grains of an aliquot is not the same for any of the three peaks. Furthermore, when several glow cycles to 500 °C are repeated on the same aliquot the response to the predose effect is distributed among the grains in still a fourth way, so that even the ratios of the 70 °C peaks from two glow cycles exhibit considerable scatter. This is particularly true for ratios involving the 70 °C peak from the first glow curve.

It follows that in general normalization of the data peaks

Table 4. Relative Standard Deviations of RBP' and 70 °C Peak and Their Ratios

Glow Cycle	1		2		3		
Peak	70 °C	RBP'	70 °C	70 °C	RBP'(1)/70(1)	RBP'(1)/70(2)	RBP'(1)/70(3)
s',%	16.6	13.1	23.1	18.5	19.3	21.7	23.7
r					0.098	-0.109	-0.355

Table 5. Relative standard deviations of SBP peaks and their ratios

Temp.	Glow Cycle Peak	1 SBP(1)	2 SBP(2)	Ratio SBP(1)/SBP(2)	$\sqrt{SBP(1)^2 + SBP(2)^2}$
230	s',%	23.0	44.4	25.0	50.1
240	s',%	20.4	40.5	23.9	45.4
250	s',%	18.5	38.3	25.2	42.5
260	s',%	17.6	37.0	27.3	41.0
270	s',%	18.3	36.7	31.0	41.0
280*	s',%	18.0	35.9	31.9	40.2
290	s',%	18.5	35.0	29.7	39.6
300	s',%	19.1	42.0	28.4	46.1

from 100 μm particles using the 70 °C peak from any glow cycle offers no improvements over, and mostly less precision than, weight normalization. There appears to be little correlation between the RBP' and the SBP. Since the RBP' is a superposition of the RBP and the SBP this suggests these two also involve different bright grains and the RBP' cannot be used to normalize the SBP and vice versa. Finally, even the SBP observed during a second glow cycle does not correlate very well with the SBP from the first cycle. The SBP cannot be used to normalize itself with second glow normalization.

It appears that weight normalization, perhaps using large specimens, or some form of the drastic HF treatment, produces the most satisfactory TL data with "inclusion" size particles. These experiments were carried out using quartz from a single source but the characteristics involved seem so general that the conclusions probably apply to any "inclusion" size quartz powders derived from natural sediments.

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PR Reviewer's Comments (John R. Prescott)

Although photoluminescence dating techniques are becoming increasingly used, there is still considerable scope for traditional TL. There is therefore a place for studies aimed at improving the precision of TL and this is such a study. The conclusions are somewhat disappointing, at least to the extent that none of the normalization procedures based on TL itself seem to work. This reviewer can add the result of our experience with "self" - normalization of the RPB peak itself. This is successful: with a standard calibration dose of 7 Gy, administered after the first series of glows, and with due allowance for pre-dose sensitization of this dose, the relative standard deviation is typically improved from 5-7% to 3-5%. It may seem like a trivial matter, but mass normalization is unnecessary if all disks are prepared with the same mass. The extra work involved is not onerous.

A device for centering samples in ESR measurement

R.G. Lyons

Dept of Geography and Oceanography, ADFA, University of New South Wales, Northcott Dr., Campbell, ACT 2600, Australia.

This simple device, designed for use with a Varian ED ESR spectrometer using 2 mm ID quartz tubes, is easy and cheap to construct, efficient to use and improves reproducibility for both mass and density normalized aliquots. Because much of my work has been on alpha irradiated samples, the sample size for ESR cavity is particularly significant for these small samples, which occupy only a few mm of the cavity window, and accurate positioning, both vertically and horizontally, is absolutely critical.

The sample tube holder shown in figure 1 ensures the sample is held vertical: the usual holder for the bottom of the tube produces too much noise and distortion if it is introduced into the cavity. Another device, also shown in figure 1, allows accurate, fast positioning of the sample in the vertical centre of the cavity. A standard sample is centred by trial and error to maximize the ESR intensity, the tube and sample holder transferred to the centering device and the depth of insertion set. The next sample tube can then be inserted to the same depth before being transferred to the spectrometer. This greatly improves reproducibility, as well as reducing the time taken for each changeover and hence the effect of machine drift (a real problem with this particular instrument) to acceptable levels.

Experimentation shows that for small samples, accurate positioning alone is not sufficient for good reproducibility, as the degree of packing can give rise to a variation of up to 15% in the ESR intensity value even for correctly positioned samples. Calculations based on the way the cavity varies in sensitivity and assuming a potential variation in packing density of 30%, confirms these data, and it is important that mass normalized samples, even when correctly positioned, be packed to a uniform height. The same care to ensure uniform packing is needed when more sample is available and the common practice of overfilling the sample tubes to ensure that the cavity window (10 mm height) is completely covered can be adopted. A simple correction based on average density, may still leave significant uncertainties (up to 10%) in the ESR intensity, unless the sample has constant density.

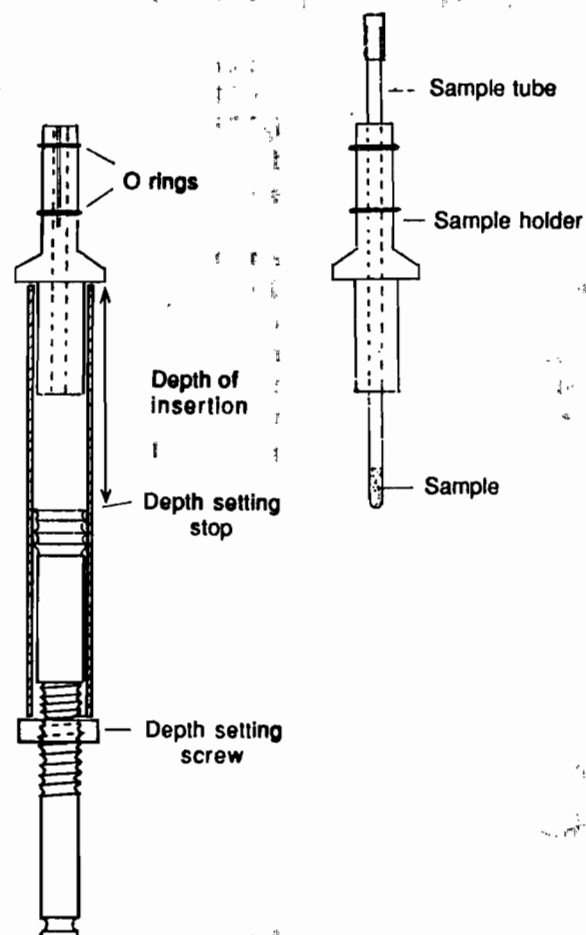


Figure 1.

The sample holder and depth setting device. The sample tube is held vertically and tensioned by 2 rubber rings so that no insert is needed in the cavity for centering. The sample tube in its holder is inserted in the device which is set to a predetermined depth to give maximum sensitivity for the sample height. The device is constructed of teflon with a glass cylinder for visual checking.

PI Henry Schwarcz

This is a practical device for a specific model of spectrometer. Similar devices could be constructed for other spectrometers and would be useful for assuring minimum error in ESR signals. The packing of the powder in the tube is also important, as noted; the effect of variable packing can be controlled by repeatedly tapping each tube on a hard surface, after the sample has been loaded and before insertion into the holder.

The use of a single aliquot method for intercalibration between radioactive sources.

Geoff Duller

Institute of Earth Studies, University College of Wales, Aberystwyth, Dyfed SY23 3DB, Wales, UK.

Intercalibration between radioactive sources is an onerous, yet essential, part of TL and ESR work. A simple method involving infrared stimulated luminescence (IRSL) is described which can be performed using a single aliquot.

The method is a development of the single aliquot technique described in Duller (1991) for the determination of equivalent dose (ED). Using multiple IRSL measurements on a single disc, with bleaching and dosing of the sample between each measurement, the ED could be determined using a regeneration method. However, using this procedure it was found that if the same dose were repeated several times the IRSL signal progressively increased. This effect was most pronounced when using IR to bleach the sample between each dose, though it was seen to a smaller extent when using a SOL 2 solar simulator. This phenomenon was ascribed to competition for electrons during irradiation. As successive doses were added, part of the trapped charge population was not removed by bleaching, and the build up of this remnant charge increased the sensitivity of the IRSL signal to dose.

To exemplify the problems of accuracy, rather than precision, associated with using IR to bleach a sample between each regeneration step, six discs of potassium feldspar were used to calibrate the β -source within the first Risø reader at Aberystwyth against itself. In this way the correct answer was known and so any inaccuracies could be easily seen. Potassium feldspar ($<2.58 \text{ g cm}^{-3}$) from sample GDNZ 17 (a New Zealand dune sand) with a grain size of 180-211 μm was used throughout the experiments.

The six discs were first heated to 450 °C at 5 °C/s. After cooling they were given a β -irradiation for 600s. After preheating at 220 °C for 10 minutes the samples had their IRSL measured for 100 seconds at 50 °C (filters used were a BG-39 and 5-60). They were then exposed to IR for 15 minutes (power approx. 40 mW/cm²) at 50 °C to reduce the IRSL signal to a residual level ($<1\%$ of the 'natural' signal due to 600 s of β -irradiation) before being irradiated by the same Risø β -source, preheated and their IRSL remeasured. Further bleaching and irradiation was repeated to build up a response curve. Irradiations of 0, 200, 400, 600, 800, 1000, 0, 600 and 1000 s were used. Figure 1(a) shows that the growth

curve is supra-linear, and that the ED generated (533 ± 4 s) is significantly lower than the expected 600s (table 1(a)). The precision of the result is excellent ($\pm 1\%$), but there is an overestimation of the true source strength by 13%. The repeat determinations of the I_0 , I_0+600 s, and I_0+1000 s points all show an increase in sensitivity of the sample aliquots.

An alternative approach is to heat the aliquots between each regeneration step. To test this, the same procedure as described above was used, calibrating the first Risø β -source against itself, but the samples were heated to 450 °C between each phase of regeneration. Identical doses were used as before, and a typical growth curve is shown in figure 1(b). The growth curve is linear and the ED generated is 588 ± 13 s (table 1(b)), within 1 sigma of the true figure. Repeat measurements of three of the data points fall on the same growth curve.

To demonstrate the use of this method two intercalibrations between the two Risø reader β -sources were performed, one using IR, and the other heating between each step to define the residual level. Figure 2 shows the two growth curves generated.

The curve in figure 2(a) was generated using the same procedure as that used for figure 1(a). After heating the discs to 450 °C at 5 °C/s a β -irradiation of 600s was given in the first Risø reader. Irradiations for 0, 200, 400, 600, 800, 1000, 0, and 600 s were given with the second Risø reader β -source. The form of the growth curve is supra-linear, and when the I_0 and I_0+600 s measurements were repeated (after all other measurements had been made) they showed a significant increase in IRSL signal. The precision of the results generated using this method is excellent (table 2(a)), but the accuracy poor.

The problem observed above is related to the remnant trapped charge. Heating the sample to 450 °C removes any residual TL or IRSL signal. Figure 2(b) shows a growth curve generated using identical analytical conditions to those for figure 2(a), except that the sample was heated to 450 °C at 5 °C/s between each regeneration phase instead of being exposed to IR. The growth curve is now linear, and when measurements are repeated (after irradiations for 0, 600 and 1000 s) they lie exactly on the growth curve.

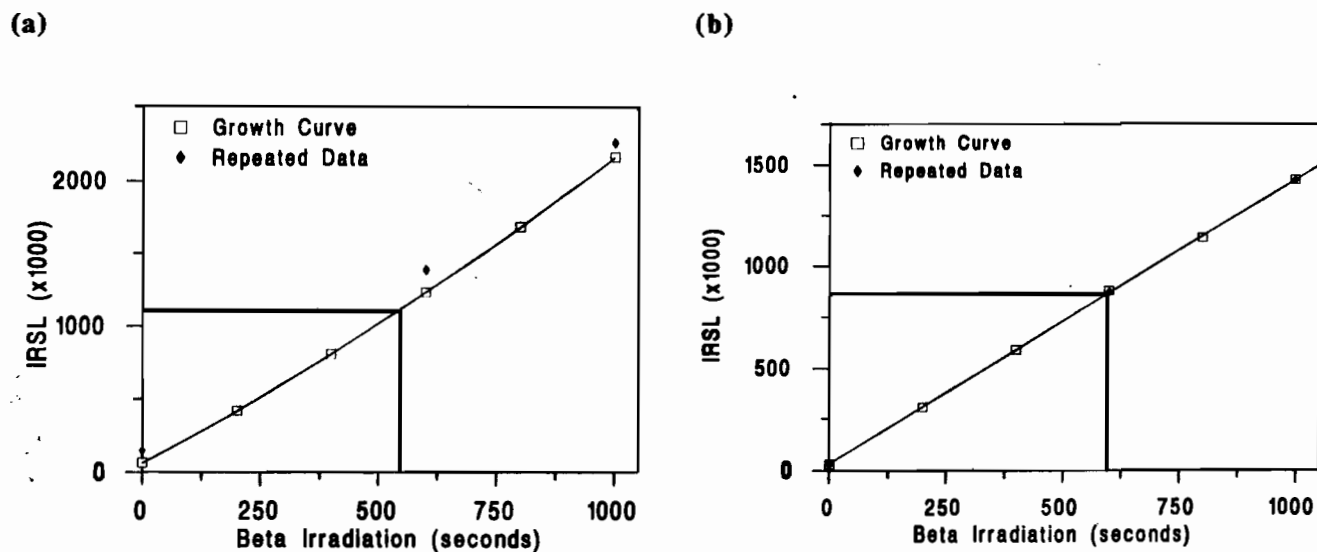


Figure 1.

Intercalibration of the Risø I β -source against itself (a) exposing the samples to 15 minutes of IR and (b) heating the samples to 450 °C at 5 °C/s, between each phase of regeneration. The IRSL signal has been integrated over the 100 s of measurement. The data points at I_0 , I_0+600 s and I_0+1000 s were repeated after all other measurements were made.

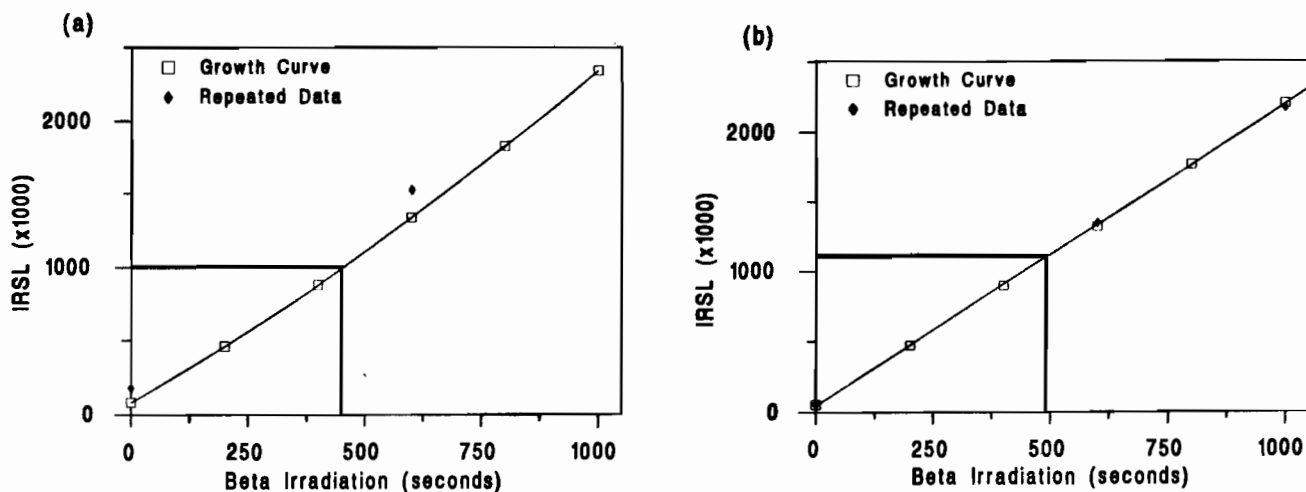


Figure 2.

Intercalibration between the Risø I and Risø II beta sources (a) exposing the samples to 15 minutes of IR and (b) heating the samples to 450 °C at 5 °C/s, between each phase of regeneration. The IRSL signal has been integrated over the 100s of measurement. The data points at I_0 and I_0+600 s (also I_0+1000 s for fig. 2(b)) were repeated after all other measurements were made.

Table 1(a).

Intercalibration of the first Risø β -source against itself using IR to reduce the IRSL signal to a residual level.

Disc No.	ED (s) ^a	Source ratio ^b
1	535	1.12
5	533	1.13
9	527	1.14
13	535	1.12
17	539	1.11
21	529	1.13

Average = 1.13 ± 0.01

Table 1(b).

Intercalibration of the first Risø β -source against itself heating samples to 450 °C to reduce the IRSL signal to a residual level.

Disc No.	ED (s) ^a	Source ratio ^b
4	604	0.99
8	565	1.06
12	596	1.01
16	587	1.02
20	583	1.03
24	592	1.01

Average = 1.02 ± 0.02

Notes

(a) 'ED' is the irradiation time interpolated from the growth curve to be necessary to match the signal produced by the initial 600 s of irradiation with the Risø β -source. A linear fit was used.

(b) By definition this ratio should be 1.00 since the β -source is being calibrated against itself.

Table 2(a).

Intercalibration between the two Risø β -sources using IR to reduce the IRSL signal to a residual level.

Disc No.	ED (s) ^a	Source ratio ^b
14	440	1.36
16	442	1.36
18	428	1.40
20	429	1.40
22	430	1.40
24	429	1.40

Average = 1.39 ± 0.02

Table 2(b).

Intercalibration between the two Risø β -sources heating samples to 450 °C to reduce the IRSL signal to a residual level.

Disc No.	ED (s) ^a	Source ratio ^b
1	505	1.19
5	515	1.17
9	494	1.22
13	493	1.22
17	483	1.24
21	488	1.23

Average = 1.21 ± 0.03

Notes

(a) 'ED' is the irradiation time using the second Risø source necessary to match the signal produced by 600 s of irradiation with the first Risø source. A linear fit was used.

(b) The source ratio is the ratio of Risø II/Risø I source strengths.

All samples gave a flat plot of 'ED' against IRSL exposure time for the 100 s period analyzed. The signal from the irradiation with the first Risø β -source could be compared to the growth curve generated by the second β -source using the regeneration analysis program written by R.Grün. The precision here is also excellent (table 2(b)), but the result is significantly different from that in table 2(a). This second method is considered to give a reliable ratio between the two sources.

Any sensitivity changes that may result from heating the sample occur before the 'test' dose is given to the sample. Subsequent heating of the sample causes no further changes in sensitivity but removes the residual trapped charge population within the crystal. This method has two major advantages over other methods of intercalibration, first that it is very quick and simple to perform, and hence one may reasonably perform many determinations to get a precise value. Secondly, the calibration is determined for the actual material being dated, not for calcium fluoride or some other phosphor. Hence there is no need for further adjustment of the data. This approach is appropriate for the basic calibration using a gamma source.

Reference

Duller G.A.T., (1991) Equivalent dose determination using single aliquots. *Nuclear Tracks and Radiation Measurements* 18, 371-378.

Acknowledgements

The author would like to thank Dr Ann Wintle and Dr Vagn Mejdahl for very helpful suggestions which have greatly improved this paper.

PI Vagn Mejdahl

I was glad to see this note on intercalibration of sources and hope it will mark the beginning of a thorough discussion on this important problem.

The extreme precision that can be achieved with the single aliquot-regeneration technique introduced by Duller (1991) makes it a very attractive method. However, source calibration using IR stimulation appears to be even more complex than with TL.

The paper shows convincingly that part of the problem is the occurrence of a sensitivity change when only IR irradiation is used as "anneal" between each measurement step. The fact that the change can be eliminated by heating the samples to 450 °C. corroborates the suggestion made earlier by Duller (1991) that the sensitivity change may be caused by the change in distribution of trapped charges resulting from the gradual build up of charges in traps not affected by the IR irradiation.

I agree that the procedure described should ensure a correct intercomparison of two beta sources. The next,

closely related question is then how should one proceed in actual dating using the single aliquot-regeneration method. Because there is the same problem with sensitivity change it seems necessary to apply the same heating anneal between each measurement step. However, because no initial heating is possible, one is faced with the problem of a sensitivity change as a result of the first heating, especially for sediments.

Perhaps even more important, it seems to me that the main problem, namely the intercomparison of beta and gamma sources remains unsolved. At the 1991 UK meeting in Aberystwyth I reported that when we used the single aliquot-regeneration technique with IR stimulation for intercomparing our beta and gamma sources, we obtained an apparent beta dose rate of 3.60 Gy/min as compared with 2.40 Gy/min when using TL. This has turned out to be a very consistent feature. However, when using IR stimulation with multiple samples as with TL, I obtain precisely the same figure as with TL. The results of the experiments described by Duller suggest that perhaps 13% of this 50% difference may be ascribed to sensitivity changes, but this still leaves a puzzling 37% unexplained.

Applying IR-OSL and TL for dating some Late-Glacial samples of well-known ages we obtained correct results when using the respective calibration values. Perhaps the lesson to be learned is that one should use the same procedure for calibration and dating.

Author's Reply

I attempted in this short note to restrict myself to a discussion of the use of a single aliquot regeneration method for intercalibration between radioactive sources. I deliberately avoided the far more complex issues concerning the use of single aliquots for equivalent dose determination of sedimentary materials since I do not see the approach used here being applicable to these materials (unless it can be shown that heating a sample to 450 °C causes no change in its sensitivity to irradiation!). Furthermore I would suggest that using the same, inaccurate, procedure for calibration and dating, as suggested by Dr Mejdahl, may fortuitously produce a correct result, but should not be relied upon for routine dating.

I agree entirely with Dr Mejdahl that it is now vital to show whether this method provides results consistent with those from TL measurements for the calibration between a gamma source and a beta source.

Comment on: A cautionary note: apparent sensitivity change resulting from curve fitting†

Enver Bulur & Ay Melek Özer

Department of Physics, Middle East Technical University, Ankara, Turkey.

In both TL and ESR dating studies the artificial growth curves are described commonly by a saturation function of the form $Y(D) = Y_{MAX}[1 - \exp(-\gamma D)]$, where Y_{MAX} is the saturation value of the TL (or ESR) intensity, γ is the dose sensitivity and D is the irradiation dose. As Li (1991) has noted that trying to describe a growth curve composed of more than one exponential saturation functions with a simple one exponential model, leads to an ED value which differs from the true ED value. He has tried to show this by applying a curve fitting method employing a simple exponential function to a computer generated data set composed of two exponential saturation functions. This can easily be shown analytically with less effort by using only elementary calculus. In this note the analytical verification of existence of the above mentioned difference is done by obtaining the analytical expression of calculated ED as a function of true ED.

Calculations

The growth curve composed of two exponential terms is expressed as

$$I = I_{MAX1} [1 - e^{-\alpha(ED+D_i)}] + I_{MAX2} [1 - e^{-\beta(ED+D_i)}] \quad (1)$$

where I_{MAX1} and I_{MAX2} are saturation values, α and β ($\alpha \neq \beta$) are dose sensitivities of the two components, respectively. D_i are the dose values for irradiation steps and ED is the equivalent dose. In the curve fitting procedure equation 1 is forced to be equal to a simple exponential saturation function for each irradiation step D_i .

$$I_{MAX1} [1 - e^{-\alpha(ED+D_i)}] + I_{MAX2} [1 - e^{-\beta(ED+D_i)}] = Y_{MAX} [1 - e^{-\gamma(ED+D_i)}] \quad (2)$$

where Y_{MAX} is the saturation value ($Y_{MAX} = I_{MAX1} + I_{MAX2}$), γ is the dose sensitivity and ED' is the equivalent dose obtained from curve fitting. The result of this can be investigated for three special values of $(ED+D_i)$:

i) For small values of $(ED+D_i)$ equation 2 reduces to

$$Y_{MAX} [1 - \gamma(ED+D_i)] \approx [I_{MAX1} \alpha + I_{MAX2} \beta] (ED+D_i) \quad (3)$$

and then $(ED'+D_i)$ is expressed as

$$(ED'+D_i) \approx -\frac{1}{\gamma} \ln \left[1 - \frac{(I_{MAX1} \alpha + I_{MAX2} \beta) (ED+D_i)}{Y_{MAX}} \right] \quad (4)$$

For small values of x , $\ln(1-x)$ reduces to $-x$, so

$$(ED' + D_i) \approx \frac{1}{\gamma} \frac{[I_{MAX1} \alpha + I_{MAX2} \beta]}{Y_{MAX}} (ED+D_i) \quad (5)$$

In order to have $(ED'+D_i)$ to be equal to $(ED+D_i)$ it is apparent that

$$\gamma Y_{MAX} \approx I_{MAX1} \alpha + I_{MAX2} \beta \quad (6)$$

must be satisfied. This can be obtained for any α , β and γ for fixed Y_{MAX} , I_{MAX1} and I_{MAX2} ; so it is always possible to find ED' values very close to true ED values.

ii) If $(ED+D_i)$ is not so small then one should take into account higher order terms in the expansion of exponential terms in equation 2, ie

$$\sum_n Y_{MAX} \frac{(-\gamma)^n}{n!} (ED'+D_i)^n = \sum_n \left[I_{MAX1} \frac{(-\alpha)^n}{n!} + I_{MAX2} \frac{(-\beta)^n}{n!} \right] (ED+D_i)^n \quad (7)$$

According to the uniqueness theorem for power series to have $(ED'+D_i)$ to be equal to $(ED+D_i)$ the coefficients in equation 7 must be equal to each other for all n , ie

$$\sum_n Y_{MAX} \frac{(-\gamma)^n}{n!} = \sum_n \left[I_{MAX1} \frac{(-\alpha)^n}{n!} + I_{MAX2} \frac{(-\beta)^n}{n!} \right] \quad (8)$$

Now it can easily be deduced that the above set of equations can only be satisfied if and only if $\alpha = \beta$. But

† Li, Sheng-Hua (1991) A Cautionary note: apparent sensitivity change resulting from curve fitting. *Ancient TL* 9(1), 12-13.

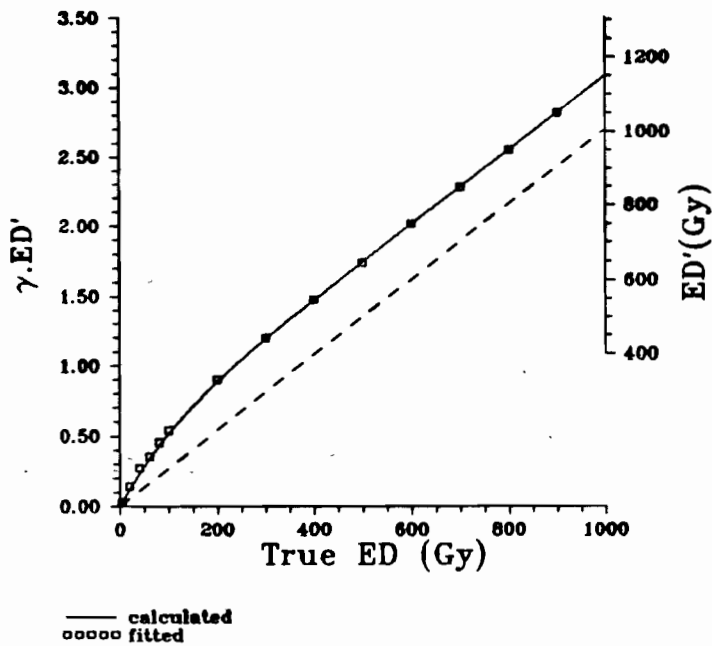


Figure 1.

The graph of $\gamma ED'$ vs. true ED. The solid line refers to the calculated value, dashed line refers to the true ED value and boxes are the data points obtained by curve fitting.

it was stated earlier that $\alpha \neq \beta$ so it is impossible to have $ED' = ED$.

iii) For very large ED values one of the terms in left hand side of Eqn.2 saturates (say $\beta > \alpha$), ie

$$I_{MAX1} [1 - e^{-\alpha(ED+D_1)}] + I_{MAX2} = Y_{MAX} [1 - e^{-\gamma(ED+D_1)}] \quad (9)$$

In this case the growth curve is described by a single exponential saturation function plus a constant. By using a curve fitting technique one can express this curve with a simple exponential saturation function. In the fitting procedure the value for γ is obtained equal to α and ED' is expressed as

$$ED' = ED + \frac{1}{\alpha} \ln \left(\frac{Y_{MAX}}{I_{MAX1}} \right) \quad (10)$$

by setting $D_1 = 0$ for clarity. As it is seen the ED' value can be found in the experiment with a constant shift, after the point at which one of the exponential terms is saturated.

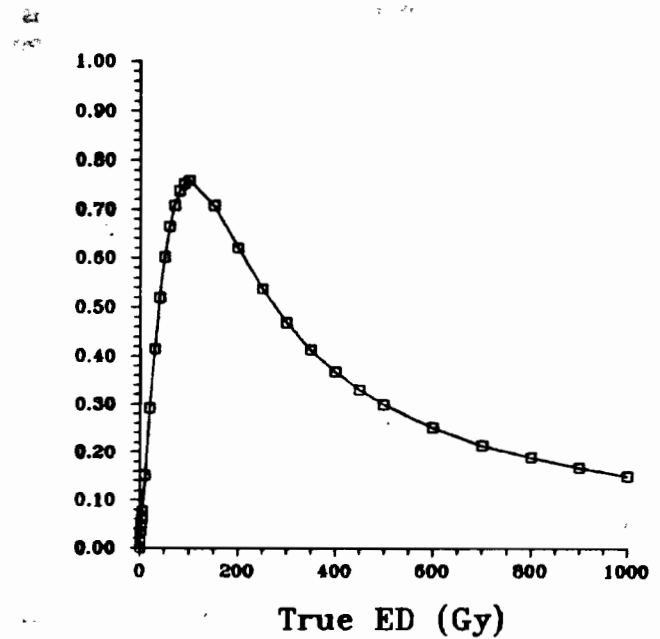


Figure 2.

Fractional error in ED as a function of true ED.

Three special cases mentioned above result in different errors in obtaining the ED value. In order to show this, the hypothetical data given by Li (1991) was used. For each case a data set generated by computer is obtained and fitted to a single exponential model by fixing the saturation value Y_{MAX} . Results are summarized in figure 1. In the figure calculated ($\gamma ED'$) values are plotted against ED due to dose dependent behaviour of γ for small and medium doses. The expression for ($\gamma ED'$) is obtained from equation 2,

$$(\gamma ED') = - \ln \left\{ 1 - \frac{(I_{MAX1} [1 - e^{-\alpha(ED+D_1)}] + I_{MAX2} [1 - e^{-\beta(ED+D_1)}])}{Y_{MAX}} \right\} \quad (11)$$

In the second y axis (right hand side of the graph) ED' values are used since after a certain dose ED dependence of γ disappears so that ED' can be obtained directly from the ED value with a constant shift. As can be seen from the graph for small values of ED, the discrepancy of calculated values from the true value (dashed line) is small and increases logarithmically as the ED value increases. After a certain point the discrepancy remains

constant, implying that one of the exponential terms is saturated. For the data given in Li (1991) this difference is 150 Gy after the ED reaches about 500 Gy.

Conclusion

In this note analytical verification of the difference between true ED and calculated ED values, when a double exponential growth curve is fitted to a single exponential model, has been performed. As a result it was found that the absolute error in obtaining the ED value for such a system, having two exponential components with different dose sensitivities, does not increase as the ED increases but remains constant after the point at which one of the exponentials is saturated. For ED values near to saturation of the growth curve the relative error becomes smaller, as is the case for small ED values (figure 2).

Reference

Li, Sheng-Hua (1991) A Cautionary note: apparent sensitivity change resulting from curve fitting. *Ancient TL* 9, 12-13.

PI S.W.S. McKeever

The major point of the original paper by Li (1991) was to draw attention to the fact that if one has overlapping TL or ESR signals, then errors in ED will result by proceeding to analyze the growth curve using the assumption that only one component is present. The simulation presented by Li to illustrate this was itself a simplification - perhaps an oversimplification - of a real situation, ie only 2 components were assumed, and each signal was assumed to follow perfect saturating exponentials as functions of dose. In reality, aside from the fact that there may well be more than two components contributing to the measured signal, none of the components being monitored may actually follow saturating exponentials. Indeed, I believe that a saturating exponential is such a special case that it is unlikely to be observed in most cases. (The latter statement is especially true for TL signals for which, in many if not most real samples, the final measured signal is the result of charge exchange interactions between several different defects. The resulting growth curve - signal versus dose - can be quite complex.) In view of this I believe that in some respects an analytical treatment of the situation discussed by Li, as presented in the present paper, is not necessary. It is true that some insights are gained from the analysis - eg the behaviour of the relative error as a function of actual ED - but readers should be cautioned not to assume that such behaviour will be typical of all situations in which overlapping signals are suspected. It is the opinion of this reviewer that the case presented is as unlikely as any other situation.

Symbols in TL & optical dating: provisional list

Martin Aitken

Research Laboratory for Archaeology, 6 Keble Road, Oxford OX1 3QJ, UK.

At a recent meeting of the Scientific Committee of the TL & ESR Dating Seminars it was agreed that papers submitted for the proceedings of the Vienna 1993 seminar would be required to use standardized symbols, terminologies and forms of date citation. The purpose of this note is to set the ball rolling[†] in respect of symbols so that there is adequate time for discussion within the community and amendment if appropriate. It is also desirable that there should be compatibility with the system developed for ESR.

In respect of terminology and citation it seems to me that most of the hard work has been done by the Editor of *Ancient TL* and published as a preamble to the Date List supplements. I suggest therefore that we use this preamble as a basis for discussion of those two aspects. Here I will do no more than reiterate, and to some extent extend, the symbols.

This basic list of symbols is primarily relevant to dating reports; in a research paper it is inevitably necessary to define additional symbols *ad hoc*.

General

α , β , γ , c subscripts to be used to indicate type of radiation (but only when distinction is necessary; often β or γ can be assumed)

Luminescence measurement

L observed level of luminescence, preferably as 'kilocounts per second (kc/s)'. Alternative units: 'Mc/s', 'c/s'; also 'Mc', 'kc', or 'c' per stated time interval (or per stated temperature interval). 'Normalized L ' may also be appropriate and, if unavoidable, ' L (a.u.)'

R (Gy) applied laboratory dose [D should not be used here because of conflict with dose-rate]

L_i and R_s for use in the case when growth with dose has the form of a saturating exponential, i.e. $L=L_i [1-\exp(-R/R_s)]$, where L_i is the value of L for infinite R

E (Gy) dose intercept in the case of linear fitting to a plot of growth with laboratory dose of a laboratory-reset sample, i.e. the supralinearity correction shown in Fig.2.2 of Aitken (1985). [use of I is undesirable on account of that letter's use for 'intensity' in ESR]

Q (Gy) ditto for a sample not reset in the laboratory, but in this case Q should be taken as positive when the intercept is negative; see Fig.2.1 of Aitken (1985) where Q is called 'Equivalent Dose'. The commonly-used abbreviation ED is undesirable for use in mathematical expressions

P (Gy) paleodose, the best estimate of the effective dose received since resetting in antiquity

χ incremental growth of L with R , i.e. incremental sensitivity

k -, a -, b -values indication of α particle effectiveness; for b use (pGy.m²) by preference

S_0 , S_N , and S_B as in pre-dose dating

Dose-rate measurement

D'_α	alpha dose-rate after allowance for effectiveness
D	(Gy/ka; mGy/a; or with 'yr' replacing 'a') total effective dose-rate with suffix 'dry' if no allowance has been made for water content. Use subscripts for components of D; thus $D = D'_\alpha + D_\beta + D_\gamma + D_e$
f	fraction of effective dose-rate due to type of radiation as indicated by subscript
c	(Bq/kg) activity, with appropriate suffix; alternatively radioactivity can be specified in terms of element concentration: ppm or %
α_0	unsealed alpha count-rate, either $\text{Ms}^{-1}\text{mm}^{-2}$ or ks^{-1} with area specified; electronic threshold fraction also to be stated
$\alpha_1, \alpha_2, \alpha_3 \dots$	sealed ditto, duration of sealing to be stated
W	saturation water content as (wt.water/dry wt.)
F	fractional uptake

Citation

A (ka or a) age, with \pm error limits at 68% level of confidence that include influence of all quantifiable sources of error, random plus systematic

NOTE the terminology BP should be avoided since it is used in C-14 with special implications; the plain statement that the age is 'so many years' is usually adequate but if emphasis of absoluteness is desirable then 'so many calendar years' can be used

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PI Ann Wintle

There are about 100 papers per year which present the results of luminescence dating programmes on material ranging from pottery and burnt flint, to volcanic ash, and to unheated sediments. These are published in a range of journals, but are more commonly being accepted in journals specializing in Quaternary Geology. Over the years different notations have developed, some of which relate to the use of different types of measurement procedures for either luminescence or radioactivity measurements. However, it is time that the luminescence community attempted to present a united front and this is being initiated by Martin Aitken.

† Ed. note Comments, or proposals in the form of short papers, should be sent to the Editor.

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Notices

Conferences

° Preliminary announcement

TED - 93
7th International Specialist Seminar
on
Thermoluminescence and Electron Spin Resonance Dating
Krems/Donau, Austria
Monday 5th July - Friday 9th July 1993

Further information:
Prof. Dr. Norbert Vana, Atominstitut
Schüttelstraße 115
A-1020 Vienna
Austria

° Preliminary announcement

The 1992 UK Luminescence and ESR meeting will take place in Durham, UK during the last week of September; further details will be circulated to those laboratories attending the 1991 meeting and to any others who wish to be placed on the mailing list. Enquiries to Ian Bailiff.

° Errata

J. R. Prescott and R.A. Purvinskis: Zero thermoluminescence for zero age (*Ancient TL* 9(2), 1991)

The following was unfortunately omitted from the review of this paper by Prof. D. J. Huntley:

'The transmission curve of the Corning (now Kopp) 7-54 filter suggests it would be a useful alternative to the UG-11 filter. We have found for both old and new 7-54's that the transmission band extends to the blue and green, with peak transmission of 0.5% at 520 nm, that is not shown in the catalogues. Use of this filter is therefore to be discouraged.'