Comments on TL Age Underestimates of Stalagmitic Calcite

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Debenham and Aitken (1984) have obtained TL age estimates on twenty seven stalagmitic calcite samples and compared them with the known U-series age estimates. Within the reproducibility/error limits specified for each estimate, these authors have concluded that no case of overestimation of the age by the TL technique is evident and there are about five cases where TL clearly gave underestimates; they have indicated that zoning effects and/or geochemical reactions during burial could be the possible causes for the TL underestimation. However, taking into account the two cases where the U-series ages have been given infinity limits and the six cases where the U-series estimations have been specified as lower limits, one can list in fact eleven cases where the TL technique can be construed to have yielded underestimates of the true ages.

I presented, during the Third Specialist Seminar on TL and ESR Dating at Helsingør (Nambi, 1983), a summary of TL age underestimates on a variety of geological samples (mostly $CaCO_3$), and brought to notice a possible correlation between the underestimation factor (= TL age/true age = t'/t) and the paleo alpha dose received by the sample (=$\int_o^t D_\alpha .dt$). Subsequently, I have observed that data of Reena De et al (1983) on calcitic oozes and of May (1977) on basaltic lava flows, also exhibit the same trend concerning TL age underestimates (Nambi 1984). Very recently TL dating failure on a nine year old laboratory - grown CaF_2 crystal containing 1555 ppm of natural uranium also indicated a proportionality between the extent of underestimation and the magnitude of paleo alpha dose (Nambi et al 1984). It should be stressed that if zoning effects or geochemical actions during burial - diagenesis is a good example in $CaCO_3$ - could be the cause for TL age underestimation, a systematic trend between the extent of underestimation and the paleo alpha dose is not warranted in all cases.

I am happy to find a similar trend in the Oxford data on TL dating of stalagmitic calcites (Fig. 1). In this figure the paleo alpha doses have been calculated assuming the U-series ages and using the dose rate conversion constants with corrections for isotopic disequilibrium as given by Hennig and Grun (1983). Nine cases have been indicated in this figure as having an upper limit for the underestimation factor and a lower limit for the paleo alpha dose to include the possibility of infinite age limit indicated in the U-series dating. There is lot of scatter of the data in the paleo alpha dose region of 200 - 500 Gy where the TL underestimation just sets in; some of the scatter points have been found to belong to samples where U-leaching cannot be ruled out (Debenham, 1984). Nevertheless the dependence of the extent of underestimation with the paleo alpha dose is quite evident especially for the nine cases mentioned above. As the paleo alpha doses covered by the Oxford study differ hardly by one order of magnitude - as against four orders of magnitude in the data presented in Nambi, 1984 - the pattern seen here is less dramatic in its appeal.

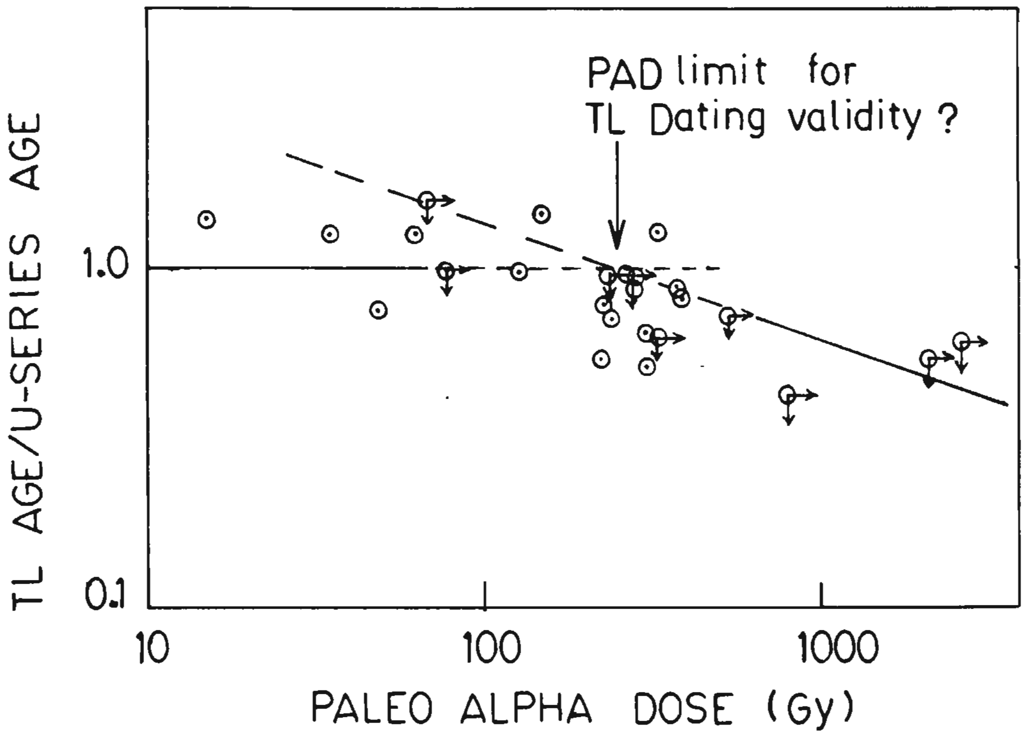

Figure 1

Deviation of TL age estimates from the U-series age estimates with progressive increase of Paleo Alpha Dose (PAD) in stalagmitic calcite samples.
(Derived from Debenham & Aitken (1984); See text for explanation on points marked with arrow-heads; the dotted indicate the dose region of uncertainty for the extensions of true and underestimation lines)

I should also point out that the "underestimation lines" have been found with different slopes and different "paleo alpha dose limits for TL dating validity" in the three groups of data so far analysed (Nambi 1984; Nambi et al 1985 and present work). In other words, in the empirical fit for the underestimation line given by $t'/t = a[\int_0^t \dot{D}_\alpha . dt]^{-b}$, the constants a and b assume different values in different materials which is quite logical to expect if one imagines an alpha-induced progressive damage effect; a dynamic equilibrium level sets in due to alpha damage and the TL saturation level depends upon not the trap life time, but the ratio of effective alpha + beta + gamma dose rate and the alpha dose rate (Nambi 1984).

Observation of the same trend in independently collected data from different sources cannot be set aside as accidental for lack of a good physical explanation at the moment. In the recently concluded Fourth Specialist Seminar on TL and ESR Dating at Worms, many new aspects of the alpha irradiation yields in various materials came to light viz., temperature dependence, dose dependence and impurity dependence (Nambi et al, 1984 and De Canniere et al, 1985). It is hoped that as more and more data encompassing a wide range of paleo alpha doses and extents of TL underestimations of the age are discovered to be mutually related and more and more of the microscopic details of the alpha irradiation effects are unearthed, a clear physical explanation of the TL age underestimation will emerge.

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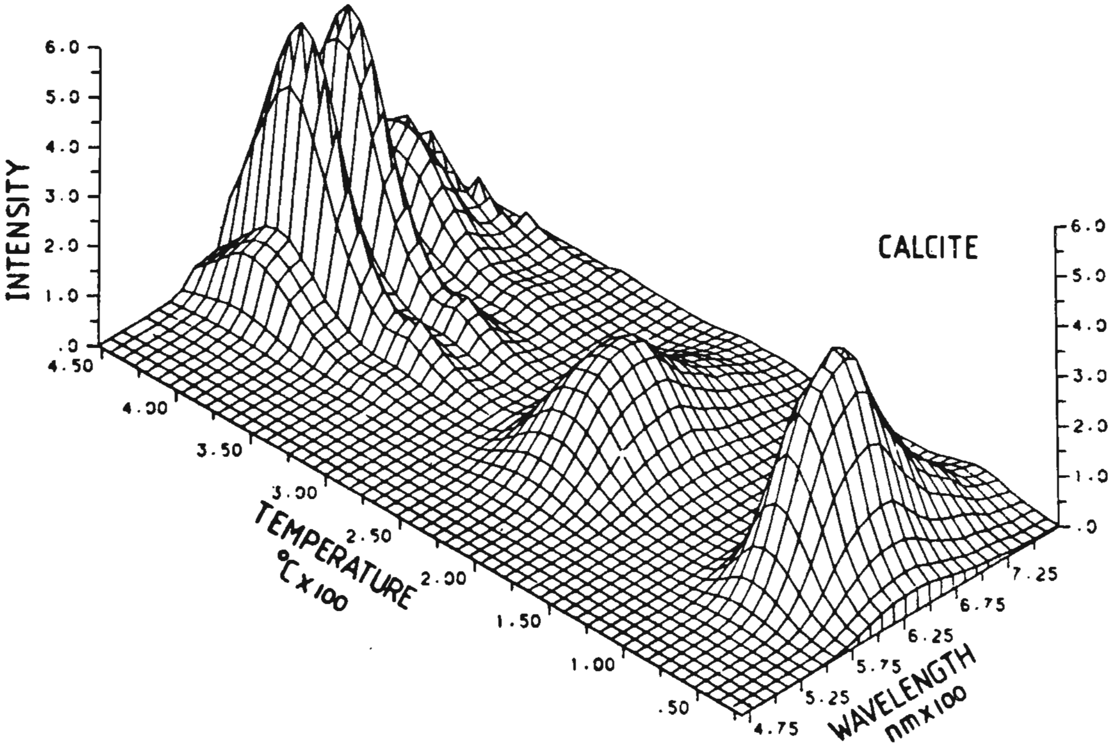
Reviewers' comments (M. J. Aitken and N. C. Debenham)

The author's hypothesis regarding correlation of TL age underestimate with paleo alpha dose is an interesting one but there is a danger that the 'reinforcement syndrome' well known in magnetic reversal stratigraphy will lead to too ready an interpretation of data in this way, with insufficient weight being given to other possible interfering factors. In the case of stalagmitic calcite Dr Nambi takes for granted that uranium-series ages can be equated with true ages; the fallacy in this is particularly evidenced by the eight uranium series determinations made on the four samples from the South Fissure at Pontnewydd which gave a wide scatter of dates even between repeat measurements on the same sample, whereas the TL dates are consistent among themselves.

In the case of the calcium fluoride experiment an explanation readily to hand is that the heavy doping gave rise to substantial anomalous fading.



Thermoluminescence Dating of Loess Deposition in Normandy

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Wintle et al (1984) have reported fourteen dates obtained on a section of loess at Saint Romain, Normandy, by thermoluminescence (TL). These are seriously affected by the conclusions of a dating program I have carried out on sediments from N.W. Europe (Debenham, 1985), which shows the instability of the TL signal limits the age range of the technique. Figure 1 summarises the findings of this work, being a plot of sediment ages as measured by TL against the estimated dates of deposition. While the latter are in many cases questionable, there is little doubt that the measured ages are limited by decay of the TL signal, and that the instability has a mean life close to 100 ka.

The experimental technique practised by Wintle et al significantly differs from my own in three respects:

(i) she selected TL emissions in the wavelength range 330-380 nm (cf. my choice of 280-380 nm);

(ii) her samples were heated at 230°C for 1 min before TL measurement; and

(iii) her method of date calculation neglected the fact that, while the alpha-induced TL intensity grows linearly with dose, the beta- and gamma-induced component shows non-linear growth (see Aitken, 1984 for discussion).

Since the maximum TL ages given in Wintle et al (1984) exceed the limiting value apparent in fig. 1, it was considered whether the different wavelength range and the pre-heat treatment used by Wintle had resulted in the selection of a more stable TL component. In order to test this possibility, four of the sediments from N.W. Europe, all older than 200 ka, were remeasured using the 330-380 nm wavelength selection and a variety of pre-heat treatments (150°C for 16 hrs; 200^{o}C for 1 min; 250^{o}C for 1 min). In all cases, equivalent dose values were not measurably altered. It thus appears that the TL signal measured by Wintle has the same mean life as that used to derive the data shown in fig. 1.

The method of calculating the TL ages was next investigated. Wintle has kindly supplied me with the basic data regarding three of her loess samples. These have now been reanalysed allowing for the different growth forms for the TL induced by alphas and that due to betas and gammas. The effect on the TL ages is to reduce them: sample h by 17%, and samples i and n both by 12%. Assuming a mean life of 100 ka for the TL signal, sample n now appears to be of such an age that its TL has effectively reached dynamic equilibrium. The TL ages of the younger loesses from Saint Romain should be corrected for the effects of the instability.

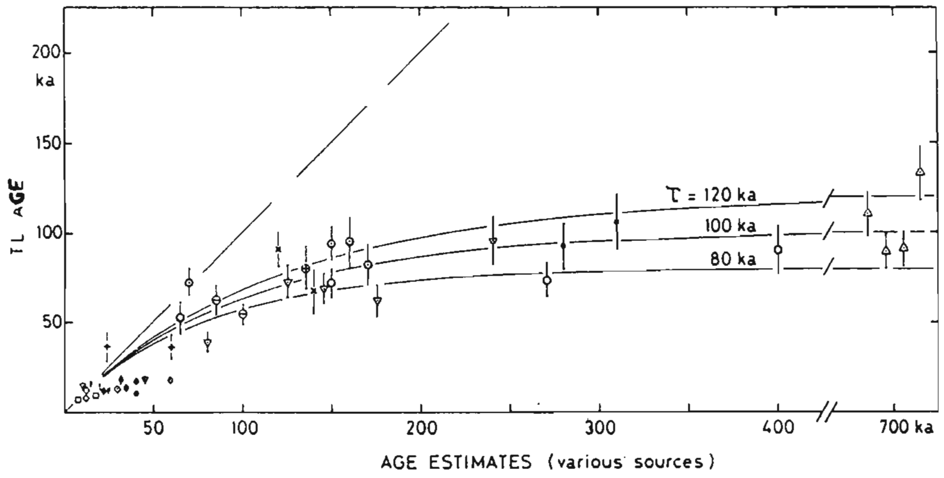

Figure 1

TL ages of sediments from N.W. Europe compared to expected dates based on various geological and archaeometric grounds. Symbols identify samples from different sites. Curves show expected TL ages, T*, assuming a constant accumulation rate to an unstable signal of mean life,* τ*;* $T = \tau\ (1-exp\ (t/\tau))$ *for* τ *= 80ka, 100ka and 120 ka.*

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Thermoluminescence Dating of Loess Deposition in Normandy

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At the Fourth International Seminar on TL and ESR Dating held in September 1984, Debenham (1985a) reported that he had been unable to obtain a TL date of more than 140 ka for any sediment, although his samples of loess from N.W. Europe included about ten for which there were geological age estimates in excess of this up to 700 ka. His plot of TL age versus geologically estimated age reproduced above (1985b) shows a deviation for samples as young as 50 ka and he suggested that this might be caused by a time-dependent loss of luminescence centres such that the TL age, T, is given by

$$T = \tau \left(1 - e^{-t/\tau} \right) \qquad \ldots\ldots\ldots\ldots\ldots(1)$$

where τ is the lifetime of the decay process and t is the real age of the sample. At the same meeting Strickertsson (1985) demonstrated that the electron trap in potassium feldspar responsible for the TL signal observed by Debenham and Wintle et al. (1984) has sufficient thermal stability that it should in principle be useful for dating samples with ages up to about 1 Ma. However, no laboratory TL experiment can test a sample for a time-dependent loss of luminescence centres such as has been proposed by Debenham.

Using the apparent TL ages for geologically older samples and the assumption that $T \rightarrow \tau$, Debenham estimated τ as 100±10 ka. It is possible to invert equation 1 so as to estimate the real age of the sample. I show these estimates in column 3 of Table 1 after I have taken account of the effect of different growth forms for the TL due to alpha particles and to beta and gamma radiation. Until the effect discovered by Debenham is better understood, I do not recommend that the values in column 3 be regarded as definitive. A clear understanding of the parameter τ in equation (1) and an estimate of its value with well-constrained confidence limits is an absolute requirement before this stage can be reached. The fact that the measured TL in the samples listed in Table 1 increases measurably with geological age, strongly suggests that they are, in principal, datable by the TL method; the very large confidence limits given for the older samples in column 3 reflect uncertainty in the parameter τ in equation (1), and not our uncertainty in the differences between the samples.

Table 1. Fine-grain feldspar age estimates (ka) for samples from Saint Romain

Sample No.	from ref. 4	col. 1 recalc. by Debenham	col. 2 corrected for fading τ=100±10ka
QTL20 h	80 ± 7	74 ± 12	$135\,^{+160}_{-45}$
QTL20 i	88 ± 8	77 ± 10	$150\,^{+150}_{-45}$
QTL20 n	114 ± 10	101 ± 12	>180
QTL20 a	12.6 ± 1.3		13.5 ± 1.6
QTL20 b	14.4 ± 1.3		$15.5\,^{+1.7}_{-1.6}$
QTL20 c	13.7 ± 1.2		$14.7\,^{+1.6}_{-1.4}$
QTL20 d	14.2 ± 1.3		$15.3\,^{+1.7}_{-1.6}$
QTL20 e	11.1 ± 1.0		11.8 ± 1.2
QTL20 f	16.4 ± 1.5		$17.9\,^{+2.0}_{-1.9}$

It will be crucial to the further development of the method to obtain TL results for more samples that can be unequivocably correlated with the 120 ka-old interglacial that is confidently dated in the deep-sea, in Britain, and in raised beach deposits around the world. If the soil that we believed we had dated at this stage at Saint Romain is in fact significantly older, then the same may be true for Debenham's samples which have been assigned similar ages. In this case the value of τ becomes less well constrained than is shown in Debenham's figure 1.

In the meantime we can conclude, with respect to the results given in Wintle et al. (1984) that the section encompassing samples a-f was deposited very rapidly about 15 ka ago, that a hiatus of at least 50 ka intervened between samples g and f, and that the sequence from g-o probably represents an orderly record whose real age range should be determinable once there is a better understanding of the effect discovered by Debenham.

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Thermoluminescence Dating of Loess Deposition in Normandy

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Debenham concluded from a recent TL dating study (Debenham, 1985) of loess deposits in N.W. Europe that the time period accessible by the TL method used was limited by the decay of luminescence centres in the material. The decay was found to have an associated mean life close to 100 ka. Debenham suggests that also the results obtained by Wintle et al (1984) in a study of loess deposits from Normandy would be affected by decay.

In view of the growing recognition of the great potential of the TL technique for dating Quaternary deposits I feel it is important to point out that a decay phenomenon of that nature is not a general observation in TL dating of sediments. I suggest that it may be related to the particular mineral fraction (polymineral, 4-11 μm grains) used by Debenham.

In figures 1 and 2 I present some results of a TL dating study (Funder et al, 1985) of marine sediments from Kap København, Peary Land, Greenland, estimated to date from Late Tertiary/Early Quaternary. The dating was based on potassium feldspar grains, 0.1-0.3 mm, extracted from the sediment. Linear extrapolation from the points in the TL growth curve in figure 1 yields an equivalent dose of 4160 Gy, while polynomial regression based on all four points yields 3060 Gy. The latter, combined with a dose-rate of 2.86 Gy/ka, gives a TL age of about 1.07 Ma (assuming the residual dose at deposition to be zero). It is obvious that the TL properties of the mineral used here are not affected by the decay phenomenon described by Debenham.

The upper limit of the time period that can be assessed by the TL dating method has not yet been established with certainty. The limiting factors can be expected to be either saturation of the TL level, when all trapping centres in the material are occupied, or decay (untrapping of charges at ambient temperatures) that will result in a dynamic equilibrium where the rate of untrapping equals the rate of trapping. In potassium feldspar saturation appears to occur at a dose level of about 4500 Gy corresponding to ages of 1-5 Ma, depending on the dose rate.

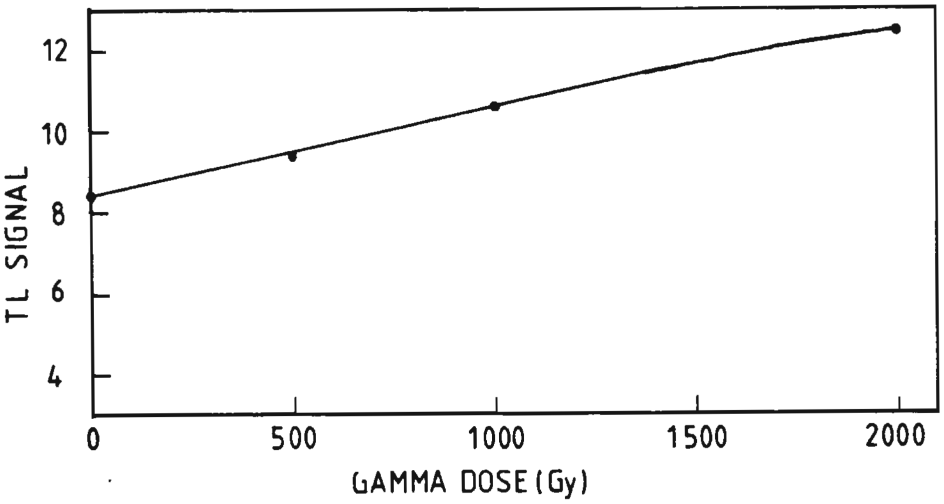

Figure 1. TL growth curve for potassium feldspar, grain size 0.1-0.3 mm (lab. no. 841008), extracted from a marine sediment at Kap København, Peary Land, Greenland. Glow curve region 325-380°C. Blue filter Corning 5-58.

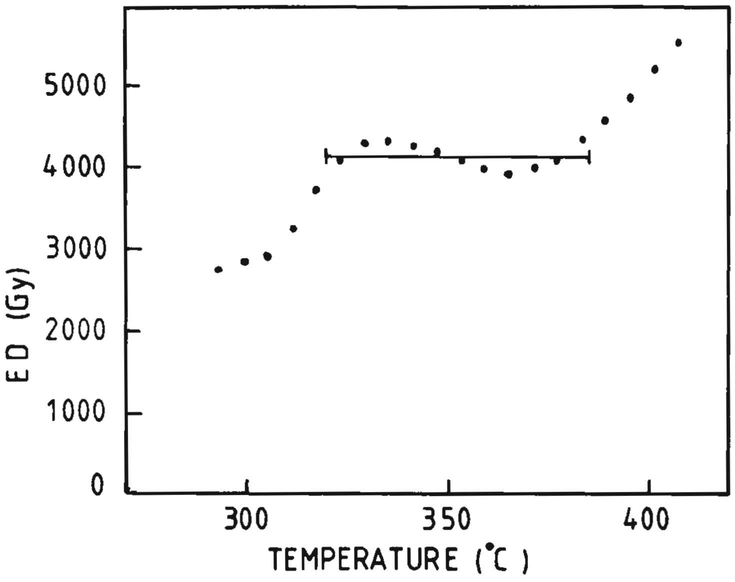

Figure 2. Equivalent dose (linear regression) vs. glow curve temperature for sample no. 841008. A plateau is seen over the region 320-380°C. With a heating rate of 8°C/s the glow peak occurs at 370°C. At temperatures beyond 400°C saturation of the TL level precludes the determination of an equivalent dose. Polynomial regression gave a plateau of only 25°C centered at 370°C.

While it is clear from the TL growth cruve in figure 1 that the natural TL level is not in saturation, one cannot exclude the possibilities of dynamic equilibrium and fading, which would make the TL age an underestimate. The stability of the trapped charges and the upper limit of the TL method can be studied by measuring the TL level in very old sediments to see whether it is in saturation or has reached a dynamic equilibrium. Studies of Tertiary deposits, 20-30 Ma old, are in progress in our laboratory and preliminary results for one of these indicate that the TL level may not be in saturation.

Concerning the loess deposits in question, the result presented above suggest that it would be important to extract larger grains from the loess and separate out specific minerals for TL dating rather than using a mixture of fine-grain minerals with different properties. Only in this way can one hope to clarify the cause of the unexpected decay phenomenon encountered for these loess deposits.

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Reviewer's comments (by M.J.A.) on the three contributions by Debenham, Wintle and Mejdahl

It is good news that coarse-grain potassium feldspar, from Greenland at any rate, can break the 100 ka barrier that is indicated by Debenham's polymineral fine-grain results from N.W. Europe. Although Wintle presumes that the fine-grain signal, in her case and that of Debenham too, comes from potassium feldspar I think this is only on the basis that this mineral is likely to dominate over the plagioclase because of its brightness. Hence extraction of the potassium feldspar from the loess of N.W. Europe might yield a more stable signal. It should also be noted that Strickertsson's lifetime may not be applicable to the fine-grain signal. However, the limit noted by Debenham is unlikely to be due simply to thermal fading because good plateaux are obtained and it is not a sharp peak; indeed for this and other reasons Debenham hypothesises that it is due to decay of luminescence centres. Whatever the cause of the decay, if correction is to be made for it, one important question is the degree of dependence on burial temperature.

Comment on 'Rapid Thick Source Alpha Counting' by Readhead (Ancient TL,2.2)

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1. Reproducibility

In 1976 the Oxford TL laboratory changed from individually-made alpha-counting screens to commercially available ones cut from sheets of silver-activated ZnS spread on a Mylar substrate (available directly from Wm. B. Johnson and Associates, Inc., Research Park, Montville, NJ 07045, USA). The main reason for the change was the lack of reproducibility of the 13.85 cm^2 screens when made up by different people and checked with a solid alpha emitting source (Bowman, 1976). Uniformity of the screen will be more difficult to achieve for a screen about 20 times larger. (Although not given in Readhead's paper in Ancient TL, the area given in his thesis was 326.85 cm^2).

2. Background count-rate

Readhead states that the background of commercial screen material is higher than for made-up screens. His value of 7.5 counts/hr for the large screen gives about the same count rate/cm^2 ks as I routinely obtain for commercial screens (6.65×10^{-3}) with a Vycor (low uranium glass from Corning, USA) disc placed on the ZnS surface to protect it from radon daughters in the air and as reported for the same commercial screens (since 1971) by Fisenne and Keller (1981) who used a Whatman filter for the background measurements. Slightly lower backgrounds (1.39×10^{-3} cts/cm^2 ks) have been achieved by Masters (1980) with a high purity unprocessed silicon wafer on the screen. From a detailed study Masters concludes that electronic noise does not contribute to this and that 3/5 of the background originates from cosmic ray interactions which could only be reduced by massive extended shielding with the remaining 2/5 from radioactive contamination.

Readhead's pair background rate (3×10^{-5} cts/cm^2 ks) is compatible with Huntley and Wintle's observation (1981) of 1 pair count in 2 Ms of background counting for a screen of area 13.8 cm^2.

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Reviewer's Comment (M.J.A.)

Perhaps it is useful to add that the scatter in count-rate reported by Bowman amounted to an overall spread of 13% in the screens made by six different workers. A convenient source for testing screen-to-screen reproducibility can be made by mixing pitchblende into 'Plasticraft' resin. In this way a disc is obtained which rests on the ring at the periphery of the screen so that testing does not contaminate it. By using about one part of pitchblende (uranium content 50%) to one part of resin a count-rate of the order of 300 per second is obtained for a 42 mm diameter screen (area (13.85 cm^2), allowing high precision in 100 seconds of alpha counting.

Although the reproducibility of Johnson screens is excellent within a batch (overall spread less than 3%) we have found substantial batch-to-batch variations. Incidentally, referring to Readhead's Figure 2 it should be noted that some aluminium, although high purity, nevertheless exhibits a significant level of alpha activity and stainless steel is safer for the peripheral ring.

A Note on the Temperature Dependence of Anomalous Fading

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Anomalous fading is generally attributed to the tunneling of electrons from traps to neighbouring sites. In the standard derivation of the probability of quantum-mechanical tunnelling there is no temperature dependence, yet the measurements of Wintle (1977) showed a significant temperature dependence. The purpose of this note is to suggest a simple explanation.

The barrier through which the electron tunnels may be itself temperature dependent due to the vibrations of the neighbouring atoms. This vibrating barrier model leads to the behaviour

tunneling probability $\propto$ exp (C.T.)

where T is the temperature and C a constant.

This relation has been independently derived by numerous authors in various disciplines but is not widely known or applied. Possibly the first derivation was by Tredgold (1962); a summary is given by McKinnon and Hurd (1983).

Testing the applicability of the relation to anomalous fading will not be easy since the time dependence is not a simple exponential, presumably the result of a distribution of tunneling barriers. On the other hand acceleration of anomalous fading by increasing the storage temperature should make its detection more convenient due to the use of shorter storage times. Similar considerations apply to the use of delayed TL measurements to reduce the effect of fading on TL dates as suggested by Huxtable et al (1972), Fleming (1976, p. 126), Wintle (1978), Berger (1984) and Lamothe (1984). Templer (1985) has shown the use of increased storage temperature to be highly promising for dating using zircon grains.

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Reviewer's Comments (M. J. Aitken)

It is timely to be reminded that in her early work on anomalous fading Wintle noted a weak temperature dependence. Besides the mechanism now proposed there is also the possibility that thermally-assisted tunnelling arises because of increased occupancy, as the temperature is raised, of above-ground-state energy levels (see Figure 5 of Visocekas et al., 1976 for illustration). As regards dependence on time most reported data can be interpreted (Visocekas, 1979; Visocekas et al., 1983) as indicating that the amount of TL lost is proportional to log (time).

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Letters

TL & ESR TERMINOLOGY: k FACTORS

K. S. V. Nambi has written to Ancient TL regarding possible confusion in the interpretation of the use of the k factor in TL and ESR that he felt may have arisen at the Worms TL Seminar. I have chosen to precis his letter (I also include a comment (*) by one of the Reviewers of the original submission) and I hope that Dr. Nambi will consider that the basis of the point he raised has been faithfully transposed below.

The k factors for the two techniques may be defined as follows:

k_{TL} = ($\alpha:\gamma$ relative trapping efficiency factor) x ($\alpha:\gamma$ relative recombination efficiency factor)

k_{ESR} = $\alpha:\gamma$ relative trapping efficiency factor giving rise to ESR signal

The TL and ESR k factors will be equivalent where the relative recombination efficiency factor is equal to unity (i.e. no qualitative difference between parameters for α- and γ- TL glow curves. However, where this is not the case, and alpha irradiation gives rise to a new TL peak and/or emission band, the $\alpha:\gamma$ relative recombination factor will be greater than unity and an inequality in the two k factors arises

i.e. $k_{TL} > k_{ESR}$

Dr. Nambi therefore suggests that k_{TL} remains as presently designated, but that a new annotation be chosen for k_{ESR} to discourage the assumption that the k factors are always equivalent.

* Henry Schwarcz has suggested that an inequality of TL and ESR k factors may alternatively be connected with the opacity of samples, which would affect the evaluation of k_{TL} but not k_{ESR}, despite meeting the conditions discussed above.

We welcome further views on this subject.

PLATEAUX AND PREHEATING

G. Valladas (18th International Symposium on Archaeometry and Archaeological Prospection, Bonn, March 1978) showed that a preheat treatment was necessary to separate the 370^{o}C peak and obtain a plateau from quartz. I am not aware of anyone before or since finding the heating necessary or using it. Does one normally get a good plateau without it (do not yield a plateau), or do people normally preheat?

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