Ancient TL

Using 600-650 nm light for IRSL sample preparation

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Introduction

Recently, Lamothe *et al.* (1994) have shown the feasibility of dating single feldspar grains using Infrared Stimulated Luminescence (IRSL) and how this methodological development could lead to the dating of poorly bleached sediments. In this study, it was shown that only some of the grains seemed to have been adequately bleached before burial. Despite the fact that those grains should have yielded the age of deposition, the optical ages obtained were underestimated by at least 30% when compared to the radiocarbon dates yielded by contemporaneous mollusc shells. In their paper, Lamothe *et al.* (1994) suggested that this underestimation might result from factors such as fading and change in luminescence sensitivity during preheating. An annoying factor could also be inadvertent illumination during the preparation of sample (Aitken, 1994), and particularly while mounting the single grain on the aluminum disk. This brief note presents the spectral transmittance characteristics of a new glass filter which, combined with a cinemoid Lee 106, allows agreeable light illumination of feldspar while mounting the sample with minimal effect on the latent IRSL.

Laboratory measurements: Transmittance spectra and IRSL

As elsewhere, light illumination in the LUX laboratory is strictly controlled, and only low intensity >600 nm light is allowed in the sample preparation room. Laboratory lamps are filtered through 3 layers of red Lee 106 jacketed around white fluorescent tubes (Spooner and Prescott, 1986). The transmittance spectrum of the Lee 106 cinemoid filter used in our laboratory was measured with a Pye Unicam spectrophotometer (Fig 1). This filter prevents any bleaching of latent luminescence in

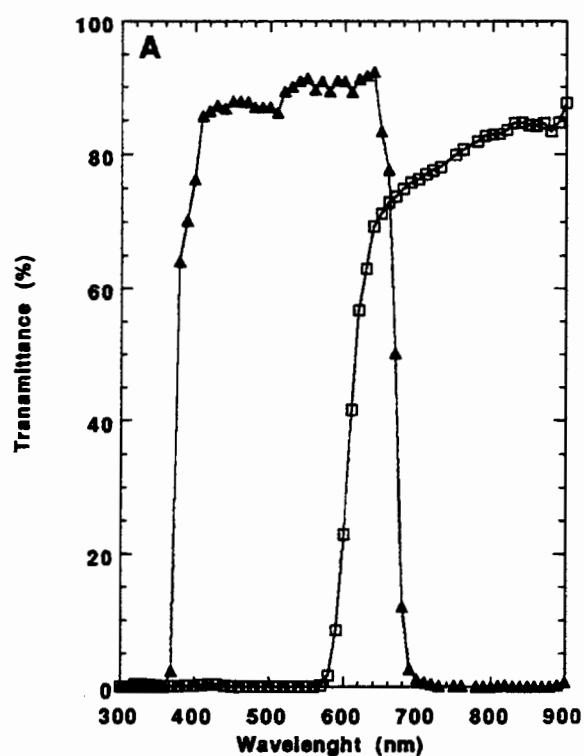samples investigated in the course of our dating programs.

Figure 1.A
Transmittance vs wavelength for the Detector Trimmer glass filter (full triangles) and a layer of Lee 106 (open squares) measured individually on a spectrophotometer;

However, for mounting a single feldspar grain onto an aluminum disk, there is need for the light source to be much closer, for a brief period of time. Some

unwanted light might be shone on the grains at this stage. In order to assess the magnitude of such laboratory bleaching, multiple-aliquots of feldspar from different geological sources have been exposed to a small (4 cm x 3 cm) fluorescent tube filtered as above, for several hours, at a distance of 5 cm. The same type of experiment was performed using an infrared trimming glass filter ("Detector Trimmer", normally used in photographic applications and available from Optical Coating Laboratory Inc, Santa Rosa, Ca. USA; d= 0.5 mm). This filter was tested since, according to the manufacturer, transmission is less than 5% for >690 nm energy. Transmittance spectra have been obtained for this filter as well as for the Lee 106/IR trimmer combination (Fig. 1).
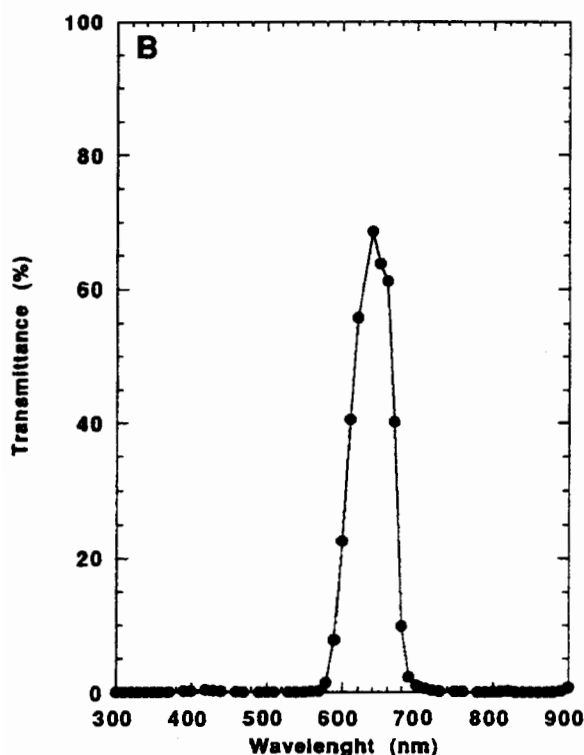

Figure 1.B
Same through both filters at once.

The IRSL measurements were performed on the automated Daybreak 1100 IRSL/TL reader. The 30 IR diodes array runs at 30 mA, with a peak transmission at 880 nm. The luminescence is detected through an EMI 9635QA PM tube and a blue-transmitting Corning 5-60/Schott BG 39 filter combination. Each measurement is luminescence emitted upon a 1 second IR shine ("short shine"). The data presented on Figure 2 have been obtained by measuring the remaining IRSL of 10 feldspar aliquots as they were progressively illuminated under the filtered laboratory lamp for periods up to almost

1000 minutes. These values are normalized to the IRSL before illumination. Erosion of the signal due to successive shines has been corrected by monitoring the IRSL of another set of 10 unbleached feldspar aliquots. The results shown herein are from the natural IRSL signal of ~100 ka feldspar aliquots (20 mg) but the data obtained from other types of sample (e.g. natural and irradiated single grain, single-layered and multiple-layered feldspar grains from other geological sources) were quite similar.

Discussion

The need of stringent light conditions has already been demonstrated by Spooner and Prescott (1986) and recently by Galloway (1991) who described the effect of different unfiltered but colored tubes on the OSL of quartz and feldspar. Some of the recipes used by different researchers are briefly mentioned in Aitken (1994).

As seen on Figure 2, the reduction of feldspar IRSL is relatively rapid when illuminated under the Lee 106 filtered light and reaches 50% after 960 minutes. When the samples are illuminated through the same light source but with the addition of the IR trimming filter, the reduction is only one-third of this and there is no significant bleaching in the first hour.

The data presented on Figure 2 demonstrate clearly that whatever the filter combination used, lengthy illumination could potentially bleach the datable part of the signal used in IRSL. However, the general light level in the laboratory (~ 0.3 lm.m^{-2}) is much lower than the light intensity used for this experiment (~ 30 lm.m^{-2}) so that bleaching of latent IRSL during the routine phases of sample preparation is unlikely. Mounting the grains necessitates only an illumination of mostly a few minutes, so that light filtered through the IR trimming filter combination seems appropriate for this phase of laboratory work. The slower decrease of luminescence intensity under the 600-650 nm filtered light is due to two factors: a) lower bleaching efficiency of the incident energy, and b) lower photon flux.

In their original paper, Hutt *et al.* (1988) have shown excitation spectra of feldspar that suggest some photoionization does occur with red stimulation. Godfrey-Smith (1991) has measured low luminescence emission for feldspar under excitation by the 647 nm line of a Kr laser (1.92 eV), but the emission was intense enough for estimating paleodoses. Ditlefsen and Huntley (1994) have also shown a low bleaching efficiency for feldspar from ~ 2.0 eV photons. Indeed, the resonance wavelength of the so-called "IRSL defect" is ~ 861 ± 6 nm (Spooner, 1994; see also Bailiff, 1993, and Botter-Jensen *et al.*, 1994). Clearly, a Lee 106 filter

transmits this light of unwanted energy if it is present in the light source. As shown on Figure 1, the IR trimmer has a very steep cut-off in the 650-680 nm range which, combined with the Lee 106 cut-on at 600 nm, leaves only the transmission of 1.9-2.1 eV photons.
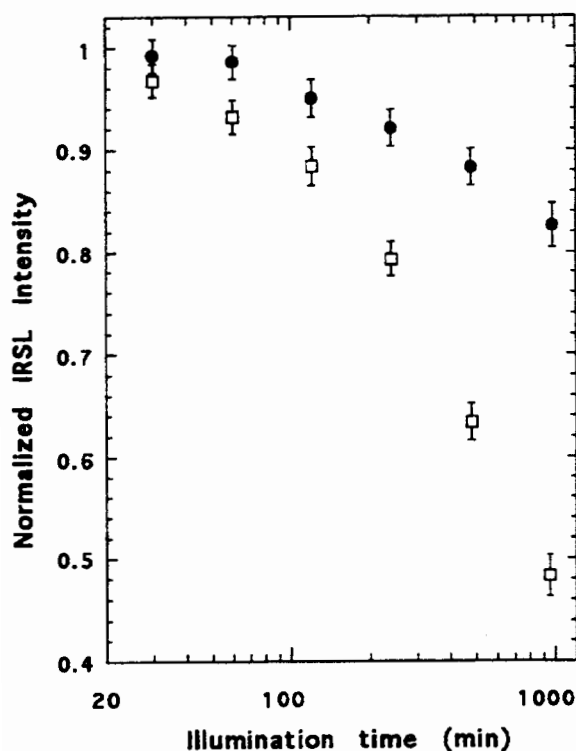

Figure 2.
    Reduction of normalized IRSL intensity for ~100 ka natural feldspar grains, under the illumination of laboratory light filtered through three layers of Lee 106 (open squares), and through the same Lee filters to which has been added the Detector Trimmer glass filter (full circles). Errors are at one sigma level.

The total light intensity was measured using a digital luxmeter (TES 1330). For the experimental design described herein, the light intensity dropped from 32.7 to 25.4 lm.m^{-2}, upon the addition of the glass filter. Therefore, for the Trimmer-Lee combination, measurements were pursued for another 500 minutes. This induced a further reduction of ~ 6-7 %, indicating clearly that the slower rate of bleaching using the Trimmer-Lee combination is related mostly to wavelength but also to absolute light intensity.

Despite the small decrease of visible light due to the addition of the glass filter, the illumination seen

through this filter combination allows agreeable light level for binocular work, with reduced danger of inadvertent bleaching. Finally, it is financially relieving to know that the glass filter described in this note can be purchased in large size at a very low cost (e.g. ~ US$ 130 for 16 sq. in., including cutting charge). Large light sources can thus be conveniently filtered during the critical phase of grain mounting.

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       Laboratory lamps emitting in the red or
yellow spectral region have usually been employed
for preparation of samples for dating. However, the
widespread use of IRSL has accentuated the need to
avoid any infrared light in the spectra from such
lamps. The paper describes an effective and
convenient filter combination that eliminates the
infrared as well as the high energy part while still
leaving a convenient light for sample preparation.

It is reassuring to see that one can work for at least
40 min. without any appreciable loss of signal with
this filter combination.

Scatter in luminescence data for optical dating – some models

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Significant scatter in optically-stimulated (and thermo-) luminescence among similar subsamples (discs or planchets) has been observed by many workers for several years, and a variety of experimental procedures have been suggested to compensate for or to minimize this scatter. However, no general understanding of the cause(s) of this scatter has been presented, thus rendering suggested 'correction' procedures rather ad hoc. In the course of a recent dating study of Holocene samples, we again encountered this problem, and the data are such that we are able to provide some insights here into plausible causes.

The samples consist mainly of K-feldspar grains; other grains were present since the separation was imperfect. Optical excitation was with ~ 15 mW cm^{-2} of 1.4 eV (880 nm) photons, and a Corning 5-57 filter was used in the measuring system in order to favour the 3.1 eV (405 nm) K-feldspar emission band. The preheat was 16 h at 120°C and the bleaching was with infrared (quartz-halogen lamp + Schott longpass RG-715 filter which absorbs strongly below 700 nm and has a high transmission above 730 nm). Both regeneration and additive-dose extrapolation procedures were employed and gave essentially the same equivalent doses. All dose responses were linear as far as could be determined. (In what follows normalization means the division of each measured intensity by $I_N/\langle I_N \rangle$ where I_N is the natural intensity for the planchet and $\langle I_N \rangle$ denotes the average for all the planchets.)

Figure 1 shows two examples of scatter. No distinction between the different symbols shown in Fig. 1B should be made at this point. In both cases the data have been normalized on the basis of luminescence resulting from a short shine before any other treatment.

Scatter generally occurs because the grains are a highly inhomogeneous collection, and most of the observed luminescence arises from a few grains on each planchet. Luminescence variations can be ascribed to intrinsic causes, such as different trap and recombination centre concentrations,

transparency etc., and to external causes, such as different radiation doses, and extents of bleaching at deposition, which are related to the histories of the grains. The normalization used should compensate for much of the intrinsic variations, and when it fails one should therefore look for the cause in the group of external causes.

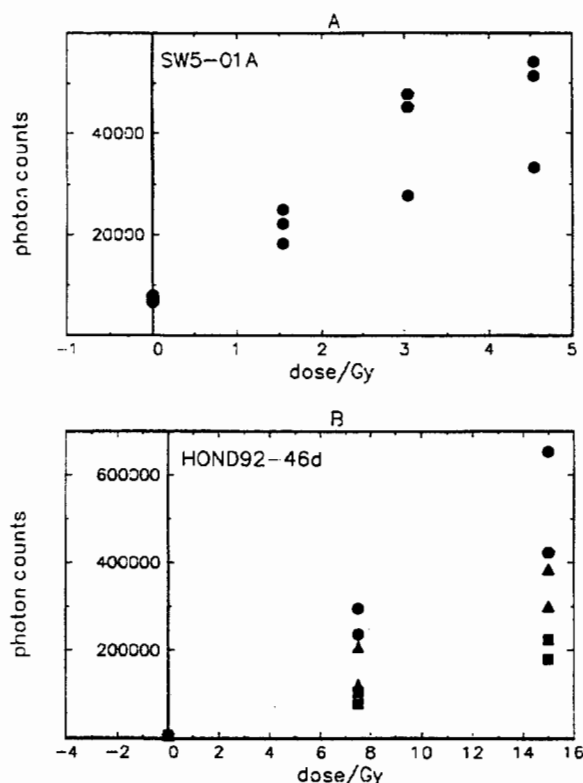**Figure 1.**

N+dose data from A) SW5-01A (Saskatchewan) and B) HOND92-46d (California). Both have been normalized. Masses were ~ 15 mg per planchet. The photon counts are for the first 10 s of excitation. For (B) the circles, triangles and squares are for planchets from groups a, b and c respectively, as defined later in Fig. 4.

We consider now two models for the cause of the scatter, both arising from incomplete bleaching at deposition, and give an example which fits one model very well. Consider two trap groups. The first group consists of those traps from which electrons are being excited by the 1.4 eV radiation; these will be referred to as the principle traps. The second group consists of traps from which electrons are transferred to the principle traps by the preheating (see e.g. Ollerhead *et al.*, 1994).

(i) Incomplete bleaching of principle traps at deposition

Consider the case in which the sample was not adequately bleached at deposition, i.e. some grains were well bleached and some were not. That this can occur has been shown directly by Lamothe *et al.* (1994). We assume that the number of grains on the planchets is not sufficient to smooth out this effect, or in other words the grains on a planchet that are responsible for most of the observed luminescence are small in number, giving rise to planchet-to-planchet variations. A set of planchets of the sample, otherwise identical, would have given varying intensities had they been measured then. Three such hypothetical planchets are shown at zero dose in Fig. 2a. The subsequent *in-situ* radiation dose would have led to the increased intensities, had they been measured, for the as-collected (naturals) and for the N+ dose points shown in Figure 2a. Applying normalization on the basis of the as-collected naturals leads to the divergent dose responses for the three hypothetical planchets shown in Fig. 2b. The scatter is similar in form to that shown in Figure 1. This cause of scatter can be recognized by the scatter increasing with laboratory dose, and the relative size of the scatter at high laboratory doses approaching that of the unheated naturals. Planchets with large normalization values will tend to appear low on the graph and vice versa. We show later that this model accounts for the data of Fig. 1b, but not those of Fig. 1a.

(ii) Incomplete bleaching of secondary traps at deposition

The practice of preheating samples prior to measurement has an unwanted effect attributed to the transfer of some electrons from secondary traps to the principle traps. Consider the case in which the principle traps were well bleached at deposition, but that these secondary traps, which are much less light sensitive, were erratically bleached at deposition. The secondary traps will not be sampled during the normalization measurement, but will be in all subsequent post-preheating measurements. The transfer from these traps can then be expected to be variable (because of the variable bleaching), and

the variability should be independent of laboratory dose (assuming the laboratory doses are small enough not to affect the population significantly); this transfer and its variability should also occur and be apparent in samples which have received a laboratory bleach that empties only the principle traps. All of this is illustrated in Fig. 3. This cause of scatter should be considered if the size of the scatter does not increase with laboratory dose. We have not yet observed an example of this behaviour.

Figure 2.
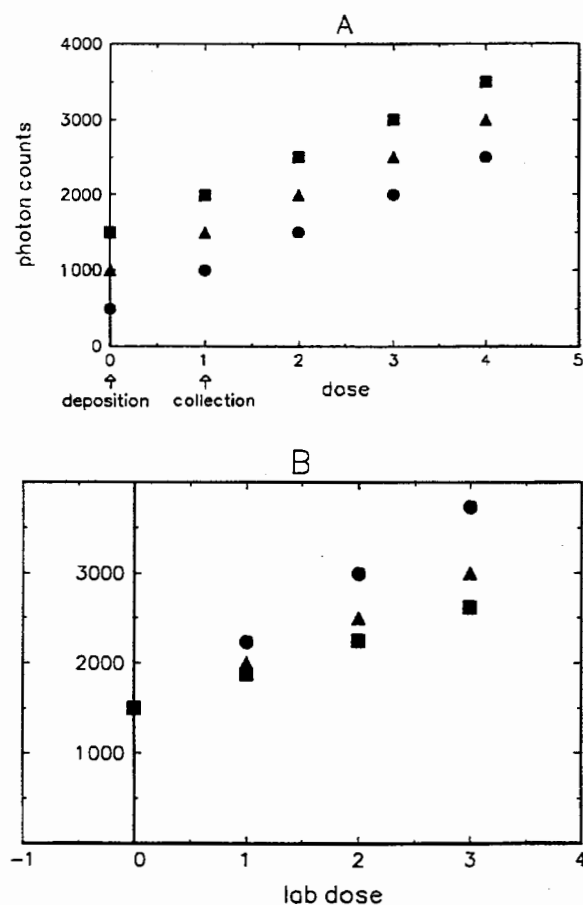
Model data showing scatter to be expected from incomplete bleaching of the principle traps at deposition.

The three photon counts at each dose are for three aliquots dominated by grains that have had different degrees of bleaching at deposition.

A) Shows data that would have been obtained had measurements been made at various times. B) Shows expected resulting data after normalization.
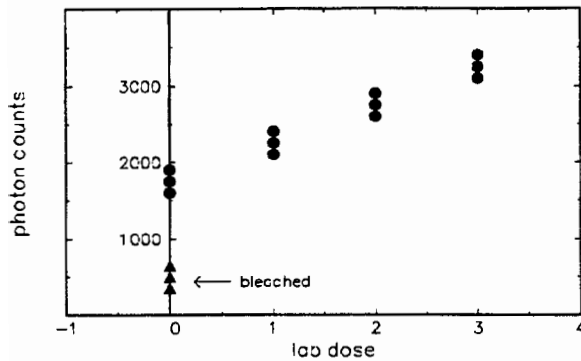
Figure 3.

Model data (not normalized) showing scatter expected from incomplete bleaching of secondary traps leading to a variation in thermal transfer.

Model(i) example - HOND92-46d

We now turn to an example in which the data are in accord with the first model. The sample, HOND92-46d, is the top 1 cm at this site, and includes the actual surface material. The normalization luminescence showed a high degree of variability for the 40 planchets made; the distribution is shown in Fig. 4. In order to test the model the planchets were split into three groups, labelled a, b and c, representing those of low, medium and high intensity, as shown in the figure. Equivalent dose measurements were then made on each group. For each group, the planchets for the naturals were selected from the centre, and other planchets were chosen in pairs symmetrically either side in order not to produce bias. The normalized N + dose data for all three groups are those shown in Fig. 1b.

If the model(i) hypothesis of the cause of the scatter is correct then the three groups of planchets can be expected to contain essentially the same material, in which case the sensitivities (i.e. photon counts/per gray, not normalized) of the three groups should be the same. They are, within the measurement uncertainties, the actual values being 18.0 ± 0.3, 18.9 ± 0.8 and 19.5 ± 0.7 counts mGy^{-1} for the first 10 s of illumination. As mentioned earlier the additive dose data should show an inverse correlation between normalized intensities and normalization values; this too is seen in Fig. 1b in which the group a points are high and the group c points are low. Another logical consequence is that the D_e values should be different for the different groups, and in proportion to the "natural" intensities. This is also borne out and the results are shown in Fig. 5.
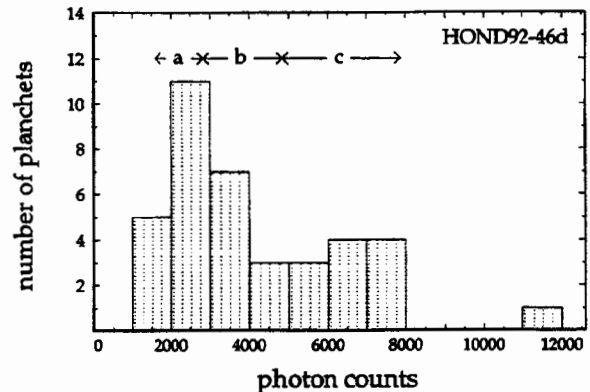

Figure 4.

Histogram of initial normalization measurements for HOND92-46d; two extreme values at 38,000 and 64,000 are not shown. The three groups into which the planchets were divided are labelled a, b and c. Four planchets, the one with the lowest intensity and those with the three highest intensities, were not included in any of the groups.
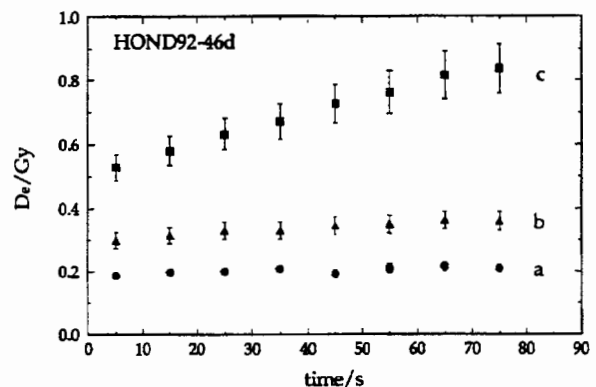
Figure 5.

Equivalent dose determinations for the three groups. The luminescence data used to obtain these were not normalized. Normalization introduced additional scatter as expected from the model.

This would appear to be convincing evidence that for this sample there is a high degree of variability in the depositional bleaching. It is remarkable that it shows up in such an extreme way in the data and one must infer from this that most of the light arises from only a few grains on each planchet. It is very interesting to see that, in the shine plateau plots, group "a" shows almost no rise with time, group "b" shows a slight rise and group "c" shows a rise of over 50%. This behaviour is expected if there are traps with varying degrees of bleachability and the "c" group contained grains that were relatively poorly bleached.

Sample SW5-01

What about the sample for which the data are shown in Fig. 1a? The same explanation cannot apply because there was very little scatter in the normalization measurements; neither did the data conform to the second cause suggested above. In addition, several similar samples from the same area showed very much less scatter. These and the lack of any obvious physical explanation led us to consider our experimental procedures and the resolution has been found there; a repeat experiment with more silicone oil yielded much less scatter in the additive dose data.

Discussion

While the above observations on HOND92-46d fit nicely to the model of incomplete bleaching at deposition, a subsequent observation caused us to pause for further thought. Sample HOND92-46 was collected by pushing a 5 cm high can into the ground. HOND92-46d discussed above, was the top 1 cm of this. The bottom 4-5 cm, called HOND92-46a, was also the subject of measurements. Its luminescence intensity was an order of magnitude larger than that of HOND92-46d, not unreasonably because of it being older. If the two samples were identical except for the age difference then one would expect any measure of the absolute value of the scatter in intensity (e.g. the standard deviation) to be the same, and thus the relative amount of scatter should decrease with increasing age. This was not what was observed, but instead the relative amount of scatter was about the same for the two samples. To explain this and maintain the basic premise of a variability in the bleaching of the grains it is necessary to postulate the existence of a mechanism that mixes the grains vertically; bioturbation by ants, worms or small rodents could do this for example, and in fact is predicted to do so as this is an A-horizon. It would be interesting to model this, and thus describe the mixing quantitatively.

A completely different model that we thought may explain some of the observations on HOND-46 was inspired by the observation that the material at this site has an unusually large thorium content, $\sim 18 \mu g \, g^{-1}$, whereas the uranium content is normal, $\sim 2.5 \mu g \, g^{-1}$, suggesting the presence of monazite, or some other mineral with a high Th content. The resulting β dose rate would then be highly non-uniform. This would lead to different feldspar grains receiving different doses, and a distribution of intensities that did not change relatively with age. We reject this explanation for two reasons. Firstly it is hard to see how the actual

intensity distribution shown in Figure 4 could be produced; this is because the Th β dose rate for this sample composes only $\sim 15\%$ of the total, hence one would expect the distribution to be a spike (for the majority of the grains), with a tail containing at most 15% of the area. Secondly, much older samples at this site show a very much smaller relative scatter, as predicted by model(i).

We conclude then that the scatter found in the luminescence data for HOND92-46 arises from variable and incomplete bleaching of the grains at about the time of deposition. For other samples for which the source of the scatter is sought, the criteria given for models(i) and (ii) should be examined, the possibility of a non-uniform dose rate should be considered, and the experimental procedures used should be examined.

Some of the ideas relating intensity, scatter and equivalent dose have previously been presented by Rhodes (1990), Duller (1994) and Li (1994).

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The increase of paleodose with stimulation time in Fig.5 (c) suggests that, as proposed in Huntley *et al.* (1985), this type of plot is useful in detecting inadequate bleaching at deposition, despite possible interference from non-first-order kinetics (see for an example, and discussion, Roberts *et al.* 1994). Perhaps it is a matter of age.
Incidentally can anybody suggest a better, while still concise, terminology than "rising plateau" ?

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"Efficiencies" of phosphor screens used in Thick Source Alpha particle Counting

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Introduction

Thick Source Alpha Counting (TSAC) has formed the basis for measuring background environmental concentrations of thorium and uranium for many years - for example, Aitken (1985), Huntley and Wintle (1978, 1981), Huntley *et al.* (1986), Jensen and Prescott (1983). The procedure involves placing the sample on a scintillator screen (usually zinc sulphide) viewed by a photomultiplier, with counters recording total alpha counts, 'slow' (within 400 ms) pair counts and sometimes 'fast' pair counts (within 20 ms). The counting system may be made up of discrete modules (eg: NIM), or purchased as dedicated alpha counting equipment (as in the case of the *Daybreak* model 582 alpha counter and printer/controller unit 584); both types are used in this department. Both counters have a screen area of 13.8 cm^2. Other systems, eg: ELSEC, are available but are not in use in this group.

One can measure a "screen efficiency" by counting a standard of known concentration and comparing the count rate observed with that calculated on the basis of the concentration of the standard. In what follows, the ratio of measured to calculated count rates will be referred to as the "efficiency".

Like Bowman (1976), Jensen (1982) and Jensen & Prescott (1983) found that the scintillator screens were less than 100% efficient and this view has been supported by Akber (1986, personal communication). Consequently, the Adelaide group has always used a "measured" efficiency less than 100% for calculating thorium and uranium contents. Huntley and Wintle (1981), Huntley *et al.* (1986), Aitken (1985) and the *Daybreak* manual seem to suggest that 100% efficiency is assumed. Examination of published counting procedures suggested that other workers assume 100% efficiency, and this procedural discrepancy prompted the investigation reported herein.

Method

Finely divided and diluted uranium or thorium ore in radioactive equilibrium with its daughters is placed in a cell on a scintillator screen. A "calibration curve" is then obtained by plotting count rate vs discriminator setting which is then extrapolated to zero discriminator setting. A linear extrapolation is commonly used but we find that a quadratic better represents the data set, as measured by the correlation coefficient.

Plotting a calibration curve for a known standard allows the determination of the appropriate discriminator setting for that screen. As is conventional, the discriminator is set to a level such that the count rate is 82% (uranium standard) or 85% (thorium standard) of the extrapolated intercept value. An example of such a calibration curve is shown in figure 1. The measured count rate at this point is compared with that calculated from the known uranium and thorium content of the standard and the conversion factors of Aitken (op. cit) or Huntley *et al.* (1986). This ratio is what is defined in the following tables under the heading "efficiency".

A selection of screens from different batches purchased from Wm. B. Johnson[1] since 1979 was tested against a variety of different standards. Within this text, "screen 5" refers to screens from batch 5. Extensive tests were carried out on the most recent screen batch purchased in 1993 ("batch 11"), and one purchased in 1980 ("batch 5"). The certified reference standards used were all from the US Department of Energy, New Brunswick Laboratory, Argonne IL 60439, USA.

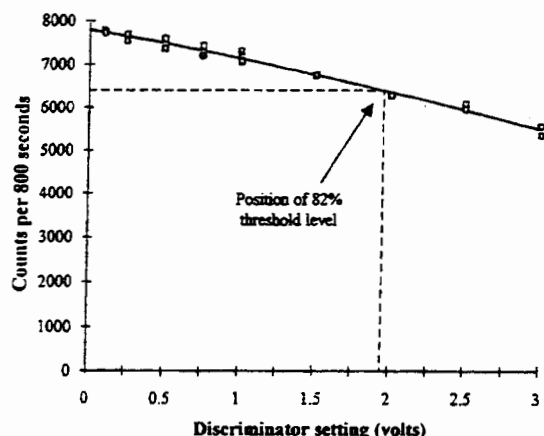[1] Wm. B. Johnson and Associates, P.O. Box 472, Ronceverte, WV 24970, USA

Figure 1
        Calibration curve for screen 5 obtained on the Daybreak *counter using NBL 42.4 standard. The counting errors lie within the boxes.*

Direct measurement of the efficiency

The efficiencies obtained are shown in table 1 with the standard used to obtain them and the nature of the measurement. For those labelled "fitted calib curve", the fitted calibration curve was used to obtain the count rate measured at the appropriate discriminator setting. For those labelled "counted data", it was assumed that the fitted calibration curve measured on another screen from the same batch using the same standard was applicable, and the discriminator was set appropriately; at least 10^4 counts would be accumulated at this setting. In both cases, the resulting measured count rate and that expected from the given standard were then used to calculate the efficiency. The conversion factors of Huntley *et al.* (1986) were used.

As NBL 109, NBL 105 and NBL 104 have very low uranium and thorium concentrations, a complete calibration curve was impractical for these standards. To obtain count rate data for these, the discriminator was set as for NBL 106, which was observed to be of the same colour (and thus have the same reflectivity) as the others.

For each measurement a new disc from the appropriate batch was used and sufficient time was allowed for radon emission to reach equilibrium in the sealed cell, although there was little evidence that this made any difference. Measurements were made on both *Daybreak* and University of Adelaide NIM systems. The quoted errors are statistical counting errors only. These results give weighted mean efficiencies of 89.3 ± 2.8% for screen 5 and

86.5 ± 1.6% for screen 11 (± sample standard deviations).

The NBL 101 result for screen 5 is well outside the interval defined by Chauvenet's Criterion for rejection of outliers (Taylor 1982). We were unable to explain this result. Omitting this result gives a weighted mean efficiency for screen 5 of 88.9 ± 1.8%.

The above results clearly show that the properties of screens can vary significantly from batch to batch. Moreover, by studying the curves produced by standards of different colours, it was found that the discriminator setting for 82% uranium / 85% thorium threshold could vary by up to 25% between grey and white standards. This confirms results obtained by Huntley (1978) and Jensen (1982) concerning sample colour and reflectivity.

It is also evident that it is not possible to reproduce efficiency measurements to better than about ± 3%. This could be due to small variations within each batch which may be very difficult to control on a mass-produced scale, or to differences in filling the cell.

Indirect measurement of the absolute efficiency

Differences among different screens were also assessed by a more rapid but less rigorous method. This uses a permanent standard ("Hanstand"), comprising an approximately 2% natural uranium source encapsulated in a silicone plastic. The source rests on a thin ring just above the scintillator screen to avoid contamination. This provides a convenient way of transferring the standard between screens and for repeating measurements on the same screen. The standard allows easy determination of the relative efficiencies of screens with respect to a chosen reference; it cannot be used to find the efficiency directly since its concentration is not accurately known, and the standard is not in direct contact with the screen. However, the absolute efficiencies can be calculated by relating the relative measurements to the measured absolute efficiency of a reference screen as detailed previously. This indirect method gave similar results to those of the direct method.

Discussion

It may be questioned whether the "efficiency" described here is an "actual" or an "apparent" efficiency and whether it is really associated with the zinc sulphide screens or with

some other feature of the method. There seems to be no doubt that it is at least partly screen-related because application of the same procedures to different batches leads to different results. That screens can vary considerably is clear from figure 2 which shows that screens can differ for two different reasons, as evidenced by the differences in both slope and intercept.
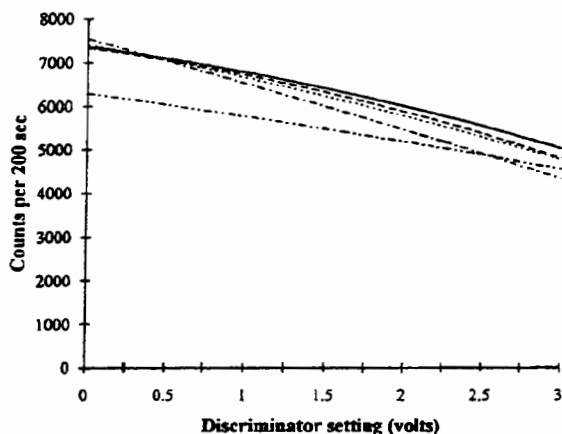

Figure 2

    Calibration curves for selected screens on the Daybreak counter using the Hanstand standard. Experimental points are omitted for clarity. They are better than ±2% throughout.

—··—·· *Screen 4 with Hanstand,*
———— *Screen 5 (early sheet) with Hanstand,*
········· *Screen 5 (last sheet) with Hanstand,*
————— *Screen 6 with Hanstand,*
—·—·—· *Screen 11 with Hanstand.*

Table 2 shows results of measurements of efficiency carried out over the years on ten (out of our twelve) batches. Differences can be observed, although in recent years these have been small, with the exception of batch 11 which exhibits a markedly different slope. D.J. Huntley kindly made independent measurements on screens 6 and 11. In relative terms we get identical results, although we differ on the question of the derived efficiency.

Another possibility is that the factors given by Aitken (1985) and independently by Huntley *et al.* (1986) for converting alpha counts and pairs counts to concentrations are in error. Huntley's concentration-to-alpha-count-rate factor for uranium is 3% larger than Aitken's, his thorium-to-alpha factor is 2.6% larger, and for thorium-to-pairs his factor is 3.0% larger. These differences are consistent with Huntley's use of more recent alpha particle data. For consistency, we have used Huntley

et al. (1986) factors throughout, including measurements made on the Daybreak system. These are not the same as those in the built-in Daybreak software, which appear to be the same as those of Aitken (1985).

Huntley *et al.* (1986) give count rate data for several standards: NBL-106, NBL-108, BL-2 and BL-3, and two measurements of DL-1a, all incorporated into a lithium borate glass. For the first, the observed count rate exceeded the expected rate by 4% while for the others the observed count rate was about 3% lower than expected. Huntley *et al.* quite reasonably conclude that the observed and expected count rates are not significantly different. Huntley and Wintle (1981) found that for four of their standards, DL-1, BL-3, NBL-106 and NBL 108, the observed counting rate fell below the expected counting rate whereas for two samples, DL-2 and BL-2 the discrepancy was in the other sense. Those measurements were with finely powdered samples (as in the present work). The Huntley and Wintle (1981) conversion factors were superseded by those of Huntley *et al.* (1986). Correcting for the difference leaves the high count high and the low counts low. In the latter case the deficiency is smaller than the one we observe. Unfortunately, the only one of these samples common with ours is NBL-106, for which we obtain a count rate that is low. Huntley notes that thorium pairs count rates come out consistently low and we return to this point below.

Jensen (1982) found the same screen efficiencies for both finely powdered samples and those incorporated into lithium borate glass, provided the calibration curves were measured independently for each method of sample preparation.

A third possibility is that the counting apparatus does not measure what we think it measures. Perhaps the best answer to this is that we get the same value for the efficiency for the same batch on three different counters. Over the years we believe we have come to understand our apparatus well. However, at the urging of Huntley we rechecked our discriminator thresholds with a precision pulse generator. The zero offsets, 0.2% of full scale, were well within the manufacturer's specification of <0.5% of full scale, which implies a systematic error of the order of 0.2% (depending on the slope of the discriminator curve) in threshold estimation and hence in efficiency. No counts are being lost through exceeding discriminator upper levels. At the suggestion of Victor Bortolot we remeasured the screen area and the point to point uniformity of one of our photomultiplier cathodes. These are not the source of the discrepancy.

Table 1 - Directly determined efficiencies

Screen batch	Standard used	Source	Count rate $(cm^{-2} ks^{-1})$	Efficiency
5	NBL 42.4	fitted calib curve	594.6	89.0 ± 0.6%
	NBL 42.4	fitted calib curve	579.1	86.7 ± 1.0%
	NBL 42.4	counted data	601.4	90.0 ± 1.1%
	NBL 42.4	counted data	610.8	91.3 ± 1.2%
	NBL 42.4	fitted calib curve	586.6	87.8 ± 1.5%
	NBL 42.4	counted data	606.9	90.8 ± 1.2%
	NBL 42.4	fitted calib curve	588.0	88.0 ± 1.5%
	NBL 105	counted data	1.175	89.7 ± 2.1%
	NBL 105	counted data	1.213	92.5 ± 2.1%
	NBL 101	fitted calib curve	1298	101.0 ± 2.0%
	NBL 106	fitted calib curve	357.2	85.4 ± 1.7%
	NBL 109	counted data	3.730	85.6 ± 2.6%
	NBL 109	counted pairs data	0.141	87.6 ± 2.5%
11	NBL 42.4	fitted calib curve	569.5	85.2 ± 0.7%
	NBL 42.4	counted data	557.9	83.5 ± 2.0%
	NBL 42.4	fitted calib curve	565.6	84.7 ± 1.4%
	NBL 42.4	counted data	553.8	82.9 ± 2.0%
	NBL 42.4	fitted calib curve	587.1	87.9 ± 1.0%
	NBL 42.4	fitted calib curve	603.3	90.3 ± 1.0%
	NBL 42.4	fitted calib curve	590.0	88.4 ± 1.1%
	NBL 42.4	fitted calib curve	586.7	85.9 ± 0.8%
	NBL 42.4	fitted calib curve	578.5	86.6 ± 0.5%
	NBL 104	counted data	11.37	85.9 ± 1.5%
	NBL 104	counted data	11.37	85.9 ± 1.7%
	NBL 104	counted data	11.22	84.8 ± 2.9%
	NBL 104	counted data	11.22	84.8 ± 3.0%

Concentration of standards used:
(all standards in silica unless otherwise stated)

NBL 42.4:	0.52 ± 0.006% U in dunite
NBL 101:	1.00 ± 0.01% U
NBL 104:	0.0103 ± 0.0004% U
NBL 105:	0.00102 ± 0.00001% U
NBL 106:	1.00 ± 0.01% Th
	0.035% U (nominal)
NBL 109:	0.0104 ± 0.0003% Th
	0.00037% U (nominal)

Table 2 - Efficiency factors of screens as used by the Adelaide group previously

Batch	Date Acquired	Efficiency factor
1	Sept 1977	95 ± 2%
2	Nov 1977	81 ± 2%
3	Oct 1978	79 ± 2%
4	June 1979	79 ± 2%
5	Dec 1980	89 ± 2%
6	May 1982	92 ± 2%
7	unknown	90 ± 2%
8	Apr 1986	92 ± 2%
9	Aug 1987	89 ± 2%
10	Aug 1988	89 ± 2%
11	1993	85.9 ± 2%

The usual prescriptions for calibration suggest measuring discriminator curves, with a linear extrapolation to zero to find the intercept. Over many observations we have found that a quadratic fit is better and we always use it, as we have here. However, for the present paper we recalculated four of our calibration curves using a linear extrapolation, covering discriminator settings to about twice the 82% threshold. The extrapolated value would be increased by a percent or two but this is not enough to account for the discrepancy, although it reduces it. The effect of colour (or more strictly reflectivity) has been extensively discussed by Jensen (1982), Huntley (1978) and by Huntley and Wintle (1983). In the present case all calibrations were carried out directly or, in a few cases where counting rate was low, on screens of similar colour.

Most TSAC systems provide a pairs counting facility (see, e.g. Aitken 1985, Huntley and Wintle 1981):. The measurement of "slow" pairs, (i.e. coincidences between succesive alpha particles from ^{220}Rn and its daughter ^{216}Po in the thorium chain), allows an estimate of the concentration of thorium. Although the small number of coincidences, relative to the total alpha count, means that the statistical accuracy in the estimation of thorium is small, it is better than no estimate at all. It is well known that the contribution of uranium and thorium to the dose rate is determined essentially by the total alpha count rate, and is very insensitive to the relative contribution of uranium and thorium to that count rate. "Accidental" pairs arise from the random coincidence of two alpha particles within the resolving time of the apparatus; and the rate of these is proportional to the square of the total count rate.

The use of a uranium source of sufficiently low concentration allows the accidental pairs rate calculation to be checked, since all slow pairs can be attributed to random coincidences. Our checks of this gave results consistent with the known resolving time. In addition, for a thorium source of low enough concentration the pairs rate, after allowing for accidentals, gives an independent estimate of the absolute efficiency ε. For counting at 100% efficiency the pairs rate is proportional to the total count rate but, if ε is less than 100%, then the pairs rate is proportional to ε^2, on the assumption that each alpha particle is independently detected with efficiency ε. In table 1, the last entry for batch 5 shows an efficiency calculated in this way. It compares favourably with the immediately preceding entry for the total count measured at the same time.

It may be noted that this measurement was extended for enough time to record 2,500 pairs.

So far as finding the thorium concentration by pairs counting is concerned, an efficiency of less than unity is not very serious if the data are to be used for dose rate calculations, for the reasons given earlier. However, if the thorium concentrations are to be compared with those found by other means e.g. neutron activation analysis or alpha spectrometry, the efficiency factor cannot be neglected. The late John Hutton (personal communication) had carried out an extensive comparison of thorium analyses made by various methods and arrived at an empirical correction to be applied to pairs counts. It has turned out to be almost the same as the ε^2 efficiency factor.

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PR D. Huntley

This work presents a convincing statement that all is not well. I am not convinced that the problem entirely lies in the ZnS screens rather than elsewhere. The different "efficiencies" obtained for different ZnS batches are strong evidence for the former; there is, however potential for other problems. The uncertainties in the alpha particle ranges appear to be of the order of 5%, as judged by differing values published over the years. In this respect calculations using the later ranges of Ziegler (1985) are needed. My experience with uranium standards is that the suppliers will not provide quantitative information about the degree of secular equilibrium, and this is most important. In addition there is the question of radon escape. McCorkell (1986) showed that it varied from 1.7 to 15.5% in the Canadian standards, was 7.5% in one DOE (USA) standard, and up to 34% in others. It was negligible in glassed standards. Does anyone know where the radon goes in a sealed TSAC cell? The situation with thorium standards is much better because of the short half lives, apart from the potential for radon escape. A glassed thorium standard should relieve this problem, and thus be the best. If the ZnS is imperfect why did Huntley et al. (1986) get such good agreement (~3%) between measured and calculated count rates on glassed thorium standards? And why did Aitken (1985, pp 306-7) get such good agreement with powdered standards? I don't know.

Turning now to the slow pair count rates, we have, over the years, found measured pair rates to be consistently lower than those calculated from Th concentrations determined by NAA. For this reason I use the figure 2.05r instead of 2.31r that is in Table 3 of Huntley et al. (1986).

This is a good opportunity to point out that the corresponding constants for fast pairs in that table are too large because no allowance was made for the fact that the first alpha decay is to an excited state which engenders a delay of the emission of the second alpha particle.

Some notes on the experimental side :

a) For the same purpose as the use of "Hanstand" we have used a glassed standard for many years. This is placed directly onto the ZnS and does not significantly contaminate the ZnS when left on it for a few hours. It is stored in a desiccator.

b) If the amplifier gain is set relatively large, some pulses will exceed 10 V (this will be the case for Fig. 1). Whether or not these pulses are counted will depend on the details of the electronics, and presents the potential for missed counts.

If the amplifier gain is set relatively small, the discriminator zero error (i. e. zero discriminator setting does not correspond to zero volts) becomes amplified and it is necessary to determine where zero volts is on the discriminator.

Additional references

McCorkell, R. H., 1986 Radon, emanation rate studies: interlaboratory standard reference materials and the effects of powdering and pelletizing. CIM bulletin 79, 66-69. (CIM = Canadian Institute of Mining and Metallurgy).

Ziegler, J. F., Biersack, J. P. and Littmark, U. 1985, The stopping and range of ions in solids Vol. 1, Pergamon Press, N. Y., (the software has been available from Ziegler) as referred to in Brennan and Lyons, Ancient TL, 7, 32, 1989.

Thesis Abstracts

Abstract

This study combines methodological and applied luminescence research of the quartz optical dating method, using an ion argon laser, with application to Late Quaternary desert sequences of the Southwestern United States. Methodological investigations are undertaken on sample pre-heating, sensitivity changes caused by dating procedures, aliquot normalisation, and dosimetry. These are complemented by investigations of colour filtering of the measured optical emission, sample pre-treatment, and the relationship between quartz petrography and scatter in dating measurements.

Three general types of sensitisation are defined: Type 1 sensitisation which represents sensitivity change during dosing (1A) or pre-heating (1B) which effect trapping efficiency; type 2 sensitisation which represents sensitivity change during pre-heating which effect luminescence emission efficiency; and, type 3 sensitisation which represents sensitivity changes occurring during optical bleaching. Dose dependent sensitivity changes during quartz optical dating are of type 3 and should not affect palaeodoses based on additive dose dating procedures.

Normalisation methods and causes of scatter between aliquots are discussed. A petrographic analysis of a suite of samples exhibiting contrasting OSL behaviour indicates wide ranging genetic forms of quartz, mineral inclusions and quartz overgrowth features which may contribute to growth curve scatter. The significance of a residual α-dosed grain rim is additionally identified as a possible scource of scatter. Natural, dose, equal pre-dose and zero glow monitoring forms of normalisation are investigated. Two types of equal pre-dose normalisation methods appear to represent the best alternative to natural normalisation.

The efficiency of signal zeroing is evaluated by an examination of depositional ages for a range of (primarily aeolian) sedimentary environments. A dating intercomparison exercise is undertaken, comparing optical dating results against numerous alternative dating strategies over a time period ranging from a few hundred, to over one hundred and twenty thousand years. The results suggest good agreement between methods.

A dating investigation is indertaken focusing specifically on the late Quaternary evolution of selected sand seas and other aeolian deposits from the Southwestern United States. These results, in combination with previously published radiocarbon and other chronologies, provide a refined regional picture of late Quaternary aeolian sedimentation and aridity.

Notices

1. MITTELEUROPÄISCHES LUMINESZENZ-UND ESR-DATIERUNGS-KOLLOQUIUM

24 - 26 November 1995
Heidelberg
For further details please contact
Dr. A. Lang or Prof. Dr. G. A. Wagner

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DEPARTMENT OF EARTH SCIENCES
UNIVERSITY OF AARHUS
DENMARK

ASSOCIATE PROFESSOR IN LUMINESCENCE DATING

Applications are invited for a position as associate professor in luminescence dating. The position is open from January 1, 1996 and the annual salary is up to Danish kroner 300.000 plus about 15% for retirement.

The post is related to the laboratory for luminescence dating which is a joint Nordic facility involved in age determinations of geological and archaeological materials.

A candidate with a background in geology and/or physics and documented research experience in luminescence dating methods, including thermoluminescence and optically stimulated luminescence will be preferred. In addition to performing routine dating determinations, the selected candidate will be expected to carry out research and development in the field of luminescence dating. Within a few years the appointee will be expected to assume full responsibility for the day to day running of the laboratory. The appointed candidate will be expected to supervise students at MSc and Ph. D levels.

As the dating laboratory is partly run on a commercial basis the appointee will be expected to be responsible for contact with users.

The laboratory is currently situated in the Research Centre at Risø on Zealand near Copenhagen, but in the long term it may be transferred to Aarhus.

Applications should include a curriculum vitae giving evidence on which the evaluation of the applicant's scientific and teaching qualifications can be based, a complete list of publications together with three copies of each of those publications which the applicant selects as the most relevant for the application. Other supportive material should also be submitted in three copies.

The selection committee may include further material from the list of publications in its evaluation of the applicant. In that case the applicant must, upon request, submit the material to the selection committee.

The selection committee's written evaluation of the applicants will be sent in full to all applicants.

Applications should be addressed to The Faculty of Natural Sciences, University of Aarhus, Ny Munkegade, Building 520, DK-8000 Aarhus C, Denmark, and marked 212/5-30.

The deadline for the receipt of all application material is October 23, 1995 at 12.00.

Obituary

Professor **Mieczyslaw F.PAZDUR**

We record with sadness the sudden death on 11th May 1995 of Professor Mieczyslaw F. PAZDUR.
Since many years ago he was the Head of Department of Radioisotopes and Head of Radiocarbon Laboratory of the Silesian Technical University of Gliwice in Poland. His main research was concerned with TL dating of sediments and with improvement in radiocarbon measurement techniques.

He was very well known in the scientific communities of both techniques and he published many results in geology as well as in archaeology. Despite financial and political difficulties during his country's period of communism, he succeeded in setting up one of the most important dating laboratories in the world.

After 1989 he had to take charge of many responsibilities in his University and in the direction of scientific research in Poland. Before his death he had taken on the organisation of the next International Radiocarbon Conference.

His death is a hard loss both for the scientists or for his numerous friends.

J. Evin
Laboratoire du Radiocarbone, Université Claude Bernard, Lyon, France

Letters

Letter to the Editor of Ancient TL

Terminology in Optical Dating

I am in process of writing a book on Optical Dating, albeit against advice from some members of the community who rightly or wrongly say the time is not yet ripe. One question concerns terminology and I would appreciate the opportunity to seek opinion from your readers.

The present situation as I see it is that the overall title Optical Dating, established by the first paper on the technique (Huntley *et al.* 1985), is so firmly embedded that it would be confusing to discard it even if one wanted to (see below). Although 'optical' tends to signify 'visible' to most people, the Oxford English Dictionary unambiguously indicates inclusion of the infrared (as well as the ultraviolet); hence there is no fundamental objection to dating by IRSL(infrared-stimulated luminescence) being included under Optical Dating.

OSL (optically-stimulated luminescence) is also well embedded but now with some ambiguity as to whether or not it includes IRSL; it was introduced by the Oxford group to keep in line with what, in 1986 at any rate, was the practice in Luminescence Dosimetry (see Mathur *et al.* 1986) --- OSL referring to the use of luminescence in which detrapping is involved, with PL (photostimulated luminescence) being used for emission from phosphate glasses in which there is no detrapping. On the other hand certain dating groups prefer PL in place of OSL on the basis that it is more meaningful to physicists. Time-dependent recombination luminescence and light-induced emission have also been used, as well as GLSL (green-light-stimulated luminescence).

The principal need is to decide whether OSL is an umbrella term for GLSL and IRSL or whether it refers only to GLSL.   An umbrella term is convenient when discussing common characteristics of the two.

Possibility A (suggested to me in discussion with John Prescott & Alan Franklin): Retain Optical Dating as the overall title and use it also for the umbrella term, with more specific indication of wavelength being given when appropriate, e.g., OD(514 nm), OD(880 nm), the terms OSL and IRSL being allowed to atrophy

Disadvantage (to my mind):   When discussing characteristics of the luminescence it is more appropriate to talk of OSL rather than OD, though in such contexts JP & AF suggest use of 'luminescence' in full.

Possibility B: Retain Optical Dating as the overall title and use OSL as the umbrella term for GLSL and IRSL, but replacing these by OSL(G) and OSL(IR) with stimulation wavelength being put in the parenthesis when more precise specification is appropriate, e.g., OSL(514), OSL(880).
Disadvantage: By usage OSL is now tending to signify GLSL.

Possibility C: Retain Optical Dating as the overall title and use PL for the umbrella term, reserving OSL for green-light stimulation, GLSL not being used.
Disadvantage: Many earth scientists would be mystified by PL, nor would they appreciate the subtle implication attributed to OSL, having become accustomed to regarding 'OSL Dating' as synonymous with 'Optical Dating' and inclusive of IRSL.

Whichever of these I use for the book readers should not think that I am suggesting any that this should be the editorial diktat in respect of other publications. I hope to complete the text during the coming winter/spring and publication, by Oxford University Press, is likely to be late 1996/early 1997; for various reasons it is not possible, regrettably, to delay long enough for reference to be made to papers in the Canberra proceedings but I would appreciate receiving any pre-prints, in respect of those proceedings or in respect of other journals, that anyone cares to send. Appropriate acknowledgement would of course be made together with opportunity to vet the relevant passage.

Martin Aitken
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Addendum

Addendum to :

D.J. Huntley, O.B. Lian, HU Jinsheng and J.R. Prescott (1994) "Tests of luminescence dating making use of paleomagnetic reversals" appeared in *Ancient TL* (12, pp. 28-30).

I wish to draw interested readers to two matters related to the article mentioned above. The first is that D.J. Easterbrook, who made the paleomagnetic measurements on the Salmon Springs section, considers the whole sequence to be of reversed polarity, and that the observed normal polarity is a magnetic overprint. This conclusion was based on measurements at several sections (D.J. Easterbrook, J.R. Roland, R.J. Carson and N.D. Naeser, Geological Society of America Special paper 227, pp. 139-165, 1988). The age of the peat should therefore be considered to be between 0.78 and 1.06 ±0.11 Ma. The details are in O.B. Lian, J. Hu, D.J. Huntley and S.R. Hicock, Can. J. Earth Sci. in press (1995).

The second point is that optimism raised by the 680±130 ka age for the West Naracoorte dune, using optical dating on inclusions within quartz grains, has been tempered by the results of measurements on several other dune samples from the same area which have yielded ages which are much younger than their known ages. A resolution is being sought.

D.J. Huntley

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(beginning of September 1995) compiled by Ann Wintle

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