

## Editorial

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I do not intend to make editorials a regular feature in Ancient TL. However, I did want to take the opportunity here to mark a number of events. On page 27 of this edition of Ancient TL are details of two prestigious awards that members of the luminescence community have been awarded this year. The Institute of Physics (IoP) awards are made annually, and this year both Professor Ann Wintle and Doreen Stoneham have been recipients. Professor Ann Wintle was awarded the Appleton medal and Doreen Stoneham received the Gabor medal. These medals are in recognition of the enormous contribution that these two scientists have made in their respective fields of endeavour using luminescence.

The bibliography in this edition of Ancient TL will be the last one put together by Professor Ann Wintle, and I would like to thank her for compiling the bibliography for the last 23 years. This bibliography has been the most consistent contribution to the journal, and in terms of the number of printed pages, the most significant. This has proved to be an invaluable service to everyone in the luminescence and ESR community, helping to keep people abreast of the ever increasing number of publications in this area of research. As the fields of luminescence and ESR have grown over this period, the magnitude of the task has increased dramatically. Looking back at the early editions of Ancient TL when Ann first put the bibliography together, it would be a bumper edition if there were 25 references or more. The bibliography in recent editions has regularly included in excess of 150 papers. Ann is also standing down from the Editorial Board of Ancient TL, and I would like to thank her for her sustained support for the journal throughout the years.

Future bibliographies in Ancient TL will be produced by Dr Daniel Richter of the Max Planck Institute, Leipzig, who will be joining the Reviewers Panel from 1st July onwards. Colleagues wishing to have their papers included in the bibliography are asked to send hard copies or PDFs of their papers to Dr Richter at 'Max Planck Institute for Evolutionary Anthropology, Department of Human Evolution, Deutscher Platz 6, 04103 Leipzig, Germany' or by e-mail ([drichter@eva.mpg.de](mailto:drichter@eva.mpg.de)).

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# How many equivalent dose values are needed to obtain a reproducible distribution?

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## Introduction

Since the development of single-aliquot measurement protocols, it has been feasible to rapidly obtain multiple estimates of the equivalent dose ( $D_e$ ) for a single sample. Where a number of  $D_e$  values have been measured, a dose distribution can be obtained; the shape and spread of this distribution may be an important factor in obtaining the appropriate burial dose ( $D_b$ ) for age calculation. Where the  $D_e$  values form a Gaussian distribution which is tightly clustered, some form of the mean is appropriate for the  $D_b$  value. Where the distribution is more scattered, however, a mean value is unlikely to be a good representation of the true burial dose. Scatter in  $D_e$  distributions can arise from a variety of factors including heterogeneous bleaching, post-depositional mixing and beta dose-rate heterogeneity; heterogeneous bleaching appears to be the most common cause of scatter and is most frequently discussed in the literature. Heterogeneous bleaching arises from insufficient exposure of a sediment to sunlight during transport, leading to residual trapped charge remaining in some or all of the grains on deposition; hence an overestimation of the burial dose is calculated for these grains. A relatively large number of studies have focussed on how to obtain an appropriate  $D_b$  value from such a distribution for a heterogeneously-bleached sample since the problem was first identified in a water-lain deposit by Murray et al. (1995). Less attention, however, has been paid to how to obtain a  $D_e$  dataset suitable for such analyses, i.e. how many replicate  $D_e$  measurements are sufficient to obtain a distribution that would result in a reproducible  $D_b$  value. If an insufficient number of  $D_e$  values is used, the final  $D_b$  value could be incorrect, regardless of how one calculated this  $D_b$ . Whilst for a well-bleached sample a relatively small number of  $D_e$  values are sufficient for  $D_b$  calculation, for a heterogeneously-bleached sample it might be expected that more measurements would be necessary to calculate the appropriate  $D_b$ .

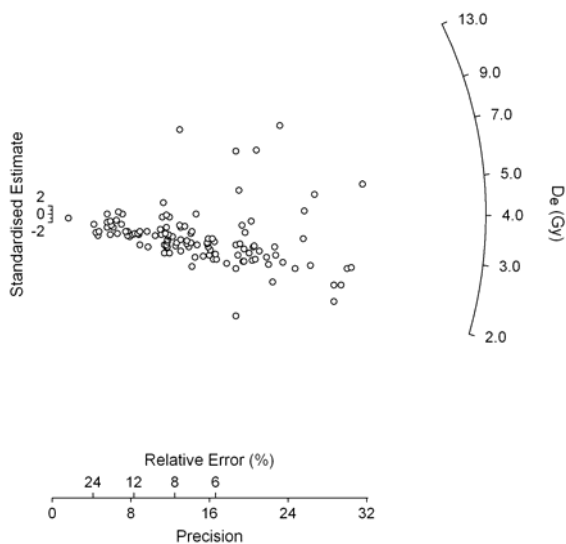
The number of  $D_e$  values used in studies investigating partial bleaching varies considerably; whilst many studies obtain more than 50 values per sample (e.g. Olley et al., 1998; Lepper et al., 2000; Folz et al., 2001; Rowland et al., 2005) others use less than 10  $D_e$  values for some samples (e.g. Colls et al., 2001; Fuchs and Lang, 2001; Srivastava et al., 2001). The quantity of material available for analysis can be a limiting factor in some instances; however, it is still desirable, where possible, to obtain enough  $D_e$  values for a reproducible distribution. The appropriate number of  $D_e$  values is not currently specified in the literature. This study aims to quantify this parameter through the use of a dataset obtained for a heterogeneously-bleached fluvial sample.

## Sample details

Sample Aber/70KLA1, used in this study, was collected from an abandoned channel of the Klip River, South Africa, by augering through post-abandonment organic deposits until continuous sand was encountered. The uppermost part of this sandy deposit was sampled and interpreted as being derived from the final bedload transport event in the channel (Rodnight et al., 2006). The sample was pretreated following common procedures including 10% v.v. HCl and 20 volumes  $H_2O_2$  to remove carbonates and organic matter, respectively. The 212-250  $\mu\text{m}$  size fraction was obtained from dry sieving and sodium polytungstate solutions (densities of 2.62 and 2.70  $\text{g}/\text{cm}^3$ ) were used to obtain the quartz fraction which was etched with 40% HF acid for 45 minutes followed by washing with concentrated HCl. The sample was then resieved and the quartz grains retained were used for OSL measurements. Small aliquots (mask diameter 2 mm and containing  $\sim 30$  grains) were used for all the measurements discussed in this paper. 175 aliquots of this sample were measured using the single-aliquot regenerative-dose (SAR) protocol (Murray and Wintle, 2000) with a preheat for 10 s at 220°C and a cut-heat at 160°C.

Aliquots were rejected if: (1) no detectable OSL signal was present after a regeneration dose had been applied; (2) the  $L_N/T_N$  value did not intersect with the growth curve; (3) the recycling ratio was not consistent with  $1.0 \pm 0.1$ ; (4) the IR-OSL depletion ratio was not consistent with  $1.0 \pm 0.1$  (Duller, 2003); (5) recuperation following a 0 Gy dose was detected (giving a  $L_X/T_X$  value greater than 5% of  $L_N/T_N$ ). 122  $D_e$  values were obtained after these rejection criteria had been applied. The errors associated with the individual  $D_e$  values were calculated in Luminescence Analyst (Version 3.20) from counting statistics, curve fitting and an instrumental reproducibility error of 2.5% (Duller, 2007).

The distribution obtained for Aber/70KLA1 indicated that the sample was heterogeneously bleached (Fig. 1), with a large range of  $D_e$  values present and an overdispersion value of 37% (Rodnight et al., 2006). Rodnight et al. (2006) demonstrated that the Finite Mixture Model (Galbraith and Green, 1990) gave the most reproducible  $D_b$  values when applied to replicate datasets of five samples and burial ages that were stratigraphically consistent with independent age control. Using the Finite Mixture Model on the dataset of 122  $D_e$  values, a  $D_b$  of  $2.38 \pm 0.02$  Gy was calculated for Aber/70KLA1. This was obtained by fitting 5 components to the data set and  $D_b$  was the value of the lowest dose component containing a minimum of 10% of the aliquots

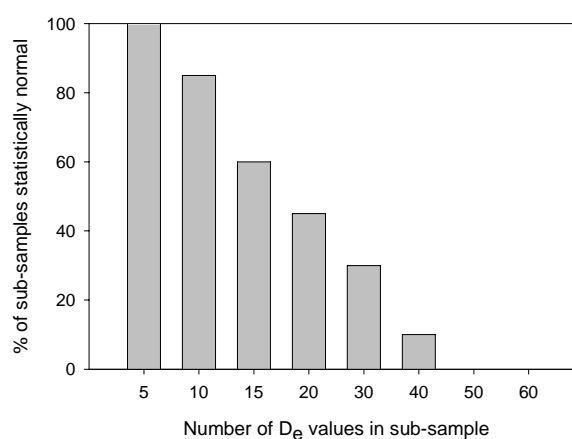


**Figure 1:** Radial plot of  $D_e$  distribution for sample Aber/70KLA1.  $D_e$  values for 122 aliquots are shown. Redrawn from Rodnight et al. (2006).

## Methods

To determine how many  $D_e$  values are needed to characterise the distribution for Aber/70KLA1, sub-samples containing 5, 10, 15, 20, 30, 40, 50 and 60  $D_e$  values were randomly selected from the 122, and this was repeated 20 times for each sub-sample size resulting in 160 datasets. When the sub-sample contains more than 5  $D_e$  values there will be some replication of the  $D_e$  values in the datasets, but the analysis should still allow one to investigate the reproducibility of the distribution for a heterogeneously-bleached set of grains. Each of the 160 sub-samples (of 5, 10, 15, ... 60  $D_e$  values) was tested for normality using the 1-sample Kolmogorov-Smirnov test (SigmaPlot Version 7.0, SPSS Inc.). The results (Fig. 2) indicate that for this sample, at least 50  $D_e$  values need to be measured to be certain of obtaining a dataset that is statistically non-normal. If the dataset contains less than 20 aliquots, there is a greater than 50% chance that a distribution from this heterogeneously-bleached sample will appear normal.

The  $D_b$  value was obtained for each of the 160 sub-samples using a variety of statistical models that have been proposed (Olley et al., 1998; Fuchs and Lang, 2001; Thomsen et al., 2003; Galbraith and Laslett, 1993; Galbraith and Green, 1990) for obtaining an appropriate  $D_b$  value from a heterogeneously-bleached sample. The change in the  $D_b$  values obtained, and the spread of the values for each sub-sample size can be used to assess how reproducible the results are for each method, at each sub-sample size. A brief description of each of these statistical techniques is given below.



**Figure 2:** Bar chart showing the percentage of the sub-sample datasets from sample Aber/70KLA1 that is statistically normal using the 1-sample Kolmogorov-Smirnov test.

*Method 1: Olley et al. (1998)*

The authors of this paper found that they were able to calculate an OSL age consistent with the known age for a poorly bleached flood deposit from the Murrumbidgee River, Australia, by taking the mean of the lowest 5% of 78 measured  $D_e$  values. They subsequently used the mean of the lowest 5% of  $D_e$  values to calculate ages for fluvial samples from a core from the Namoi River; they found that the ages generally increased with depth. Whilst this method appeared to be suitable for the rivers investigated, it would be expected that the percentage of  $D_e$  values used would have to be 'calibrated' for different depositional systems.

*Method 2: Fuchs and Lang (2001)*

Low quantities of quartz were obtained from the samples detailed in this paper, therefore only nine or ten aliquots were analysed per sample. The results showed  $D_e$  values that were scattered more than expected from experimental variation, and this scatter was attributed to heterogeneous bleaching. Aliquots which had been artificially bleached and irradiated yielded a maximum standard deviation of 4% in the  $D_e$  values obtained from these measurements. To calculate a  $D_b$  value from the natural  $D_e$  values, based on only those  $D_e$  values from aliquots consisting of well-bleached grains, the  $D_e$  values for each sample were ranked in order from lowest to highest. Starting with the lowest value, and including one additional  $D_e$  value at a time, the mean  $D_e$  and the percentage standard deviation was calculated. This was repeated including aliquots with increasing values of  $D_e$  until the standard deviation of the mean was 4%, and this mean value was taken to be the most appropriate estimate of  $D_b$ .

*Method 3: Thomsen et al. (2003)*

This paper detailed a method which calculated the correct  $D_b$  from single grain  $D_e$  values for quartz extracted from irradiated blocks. To calculate a  $D_b$  value based on those grains from the well-bleached part of the distribution, the ratio of the external measurement of uncertainty ( $\alpha_e$ ) to the internal measurement of uncertainty ( $\alpha_i$ ) was used. The equations for these two measurements of uncertainty are:

$$\alpha_e^2 = \frac{\sum_{i=1}^n \frac{(x_i - \bar{x})^2}{\sigma_i^2}}{(n-1) \sum_{i=1}^n \frac{1}{\sigma_i^2}}$$

$$\alpha_i^2 = \frac{1}{\sum_{i=1}^n \frac{1}{\sigma_i^2}}$$

where  $x_i$  is the dose estimate from each individual grain,  $\sigma_i$  is its uncertainty,  $\bar{x}$  is the weighted mean, and  $n$  is the number of measurements.  $\alpha_e$  combines information on individual estimates of uncertainty for each grain and the deviation from a weighted mean. If there is no other source of error except for the uncertainty on the individual data points then  $\alpha_e$  reduces to  $\alpha_i$ , so for a large, normal population  $\alpha_e/\alpha_i$  tends to unity (i.e. where the overdispersion is 0). In a distribution containing partially bleached grains, this ratio can be used to determine which grains are well bleached, i.e. where the distribution in  $x_i$  is consistent with  $\sigma_i$ . Any additional variance because of heterogeneous bleaching will increase  $\alpha_e$  relative to  $\alpha_i$ . By ranking the individual equivalent doses from lowest to highest and calculating  $\alpha_e/\alpha_i$  for  $n = 2, 3 \dots x$  until  $\alpha_e/\alpha_i = 1 \pm (2(n-1))^{-0.5}$ , only the well-bleached grains are used in the calculation of  $D_b$ . Any grain giving a  $D_e$  above this point is assumed to be partially bleached.

*Method 4: Galbraith and Laslett (1993) – Minimum Age Model*

The minimum age model was developed for samples where heterogeneous bleaching is evident (Galbraith et al., 1999). The model fits a truncated normal distribution to the logarithms of the individual  $D_e$  values, with the truncation point giving the value of  $D_b$ . To describe the distribution fitted to the dataset, four parameters and their errors need to be calculated: (1) the proportion of grains that were fully bleached prior to burial; (2) the truncation point of the distribution (i.e. the logarithm of the  $D_b$  value); (3) the value that would be the mean of the normal distribution fitted to the dataset; and (4) the overdispersion of this distribution.

*Method 5: Galbraith and Green (1990) – Finite Mixture Model*

This model was developed for instances where grains of more than one discrete population are present and where each population has been well bleached and can be described by the central age model (Galbraith et al., 1999). Although this model has been designed for a distribution consisting of a discrete number of populations (components), it can be applied to a heterogeneously-bleached sample. As the model selects populations based on the logarithms of  $D_e$  values that are consistent with one another (within a pre-defined overdispersion value), the  $D_b$  of the lowest population will be essentially derived from a

normal distribution of the lowest  $D_e$  values. Thus, if the dataset contains some  $D_e$  values measured from well-bleached grains, this lowest population should give the appropriate  $D_b$  value for the sample. To analyse a dataset, the model is run repeatedly, starting with only one component, and then with an additional component included each time. The model output includes two parameters which can be used to select the most appropriate number of components for fitting the dataset. The maximum log likelihood (l<sub>lik</sub>) of a fit is likely to improve indefinitely as a greater number of components are fitted, although this does not necessarily mean that the solution is a better one. The Bayesian Information Criterion (BIC) takes into account the complexity of the model as well as the goodness of the fit to the data, and hence reduces down to a minimum at the 'best' fit, before rising as the increase in the l<sub>lik</sub> is outweighed by the additional components (Jacobs et al., 2008). For each of the 160 sub-samples in this study, the  $D_b$  value was calculated from running the model with the number of components that were calculated to have the best-fit (using the BIC). The final  $D_b$  value calculated for each sub-sample was based on the lowest population that contained at least 10% of the  $D_e$  values in the dataset. This 10% value was selected arbitrarily so that populations based on one or two  $D_e$  values only were not used for derivation of the sample  $D_b$ . The number of components found to have the best fit varied between the sub-samples. As an example, the number of components fitted to the 50  $D_e$  sub-samples was as follows: two components (2 sub-samples); three components (6 sub-samples); four components (8 sub-samples); five components (3 sub-samples); and six components (1 sub-sample).

The first two methods do not incorporate the error on the individual  $D_e$  values in the analysis. If the brightness of the OSL signal is variable on an aliquot-to-aliquot basis, however, then the  $D_e$  errors will also vary considerably. Thus it is preferable for them to be taken into account, otherwise a value that may appear to be an outlier can actually form part of the main distribution within errors.

For Method 5 an overdispersion value of 10% was used for each component; since no well-bleached samples were available from the Klip River study area this was based on a dataset of  $D_e$  values obtained from analysis of small aliquots of a well-bleached last glacial maximum linear sand dune from Tasmania (see Rodnight et al., 2006 for further details). For Method 4 this 10% overdispersion was incorporated into the minimum age model so that the results were comparable with those from Method 5.

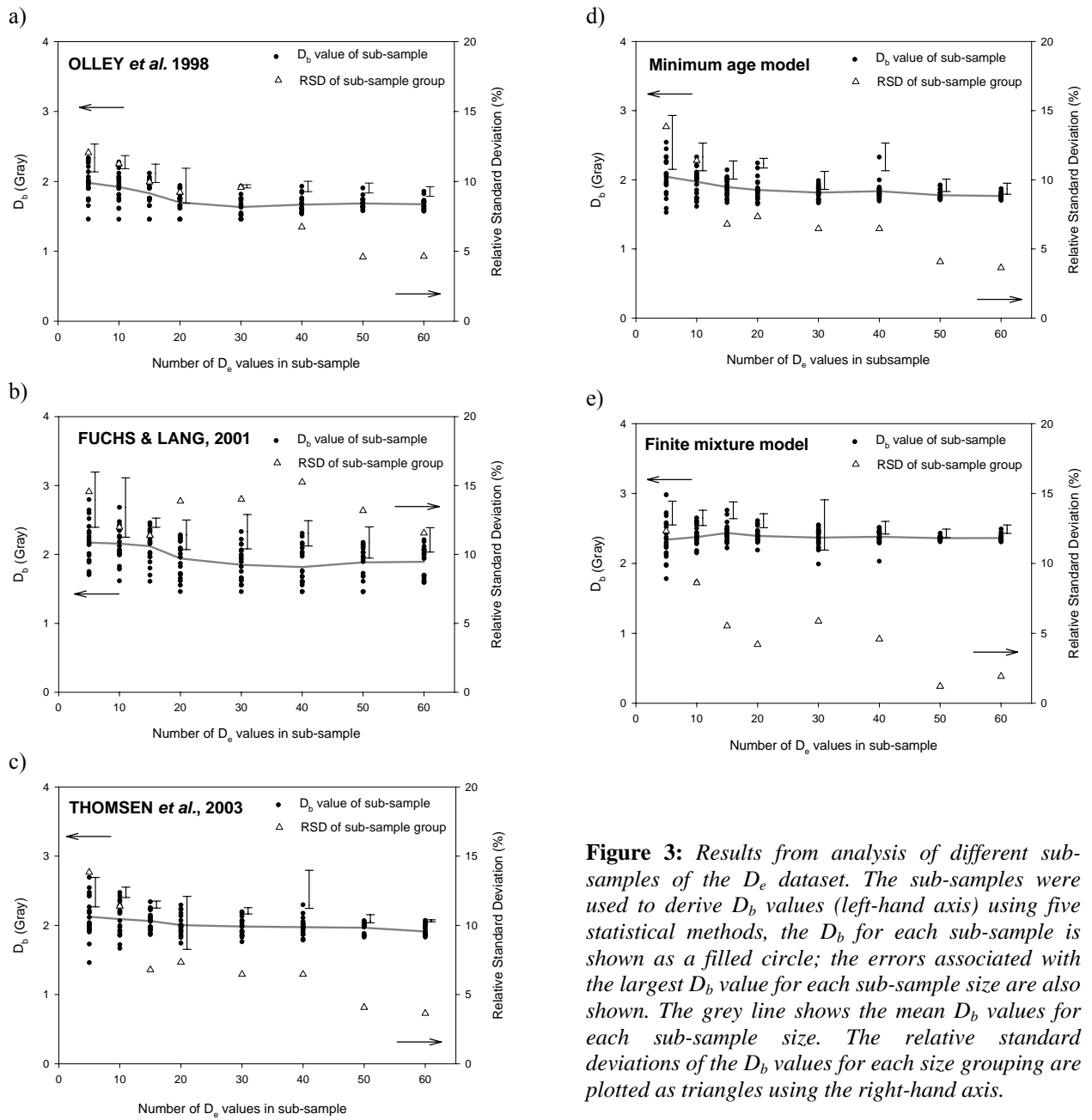
## Results

The results obtained for each method using the dataset for Aber/70KLA1 are shown in Figure 3. The  $D_b$  value calculated for each of the 160 datasets is plotted as a filled circle as a function of sub-sample size. The error bars indicate the absolute error associated with the greatest  $D_b$  value calculated for each sub-sample size. The grey line joins the mean  $D_b$  value for each sub-sample size to indicate whether changing sub-sample size can be associated with the trend in the mean  $D_b$  value calculated. The relative standard deviation (RSD) calculated for the 20  $D_b$  values for each sub-sample size is indicated by the open triangle.

The results using Method 1, of Olley et al. (1998), to calculate  $D_b$  are shown in Figure 3a. When the sub-sample consists of more than 20  $D_e$  values, consistent values are obtained for the  $D_b$ . The  $D_b$  for datasets with  $\leq 20$   $D_e$  values will be derived from the lowest  $D_e$  value only, whilst the  $D_b$  for the 60  $D_e$  sub-sample is based on the lowest 3  $D_e$  values. As this technique takes into account only very few aliquots from the lower end of the distribution, each  $D_b$  value is essentially based on the same few  $D_e$  values. This is demonstrated by the mean  $D_b$  values that fall monotonically for between 5 and 20  $D_e$  values, and then remain constant at around 1.8 Gy. Using this method, the RSD of the results falls to  $<5\%$  when at least 50  $D_e$  values are included in the analysis.

The RSD of the  $D_b$  values obtained using Method 2 (Fuchs and Lang, 2001) show poor reproducibility in the results (Fig. 3b). This poor reproducibility arises because of the sensitivity of the procedure to a high percentage standard deviation in the lowest two or three  $D_e$  values in the sub-sample. For instance, if the first few  $D_e$  values vary considerably, a high relative standard deviation results and the  $D_b$  is calculated from only these very few values. Even when the sub-sample consists of 60  $D_e$  values, the RSD of the  $D_b$  values remains high ( $>10\%$ ).

For sub-samples containing  $\leq 15$   $D_e$  values, Method 3 (Thomsen et al., 2003) tends to select low outlying values for calculation of the  $D_b$ ; however, with larger sub-sample sizes this does not occur (Fig. 3c). In general, the mean  $D_b$  value that is calculated stays relatively consistent at  $\sim 2.1$  Gy, and the RSD of the  $D_b$  values is  $<5\%$  when 50  $D_e$  values are included in the analysis. The errors associated with the  $D_b$  value are, however, highly variable owing to the manner in which the  $\alpha_e/\alpha_i$  ratio is used to derive the errors.



**Figure 3:** Results from analysis of different sub-samples of the  $D_e$  dataset. The sub-samples were used to derive  $D_b$  values (left-hand axis) using five statistical methods, the  $D_b$  for each sub-sample is shown as a filled circle; the errors associated with the largest  $D_b$  value for each sub-sample size are also shown. The grey line shows the mean  $D_b$  values for each sub-sample size. The relative standard deviations of the  $D_b$  values for each size grouping are plotted as triangles using the right-hand axis.

| Method                   | Minimum $D_b$ (Gy) | Maximum $D_b$ (Gy) | Mean $D_b$ (Gy) |
|--------------------------|--------------------|--------------------|-----------------|
| 1. Olley et al. (1998)   | $1.58 \pm 0.08$    | $1.90 \pm 0.01$    | $1.68 \pm 0.02$ |
| 2. Fuchs and Lang (2001) | $1.46 \pm 0.32$    | $2.17 \pm 0.23$    | $1.88 \pm 0.06$ |
| 3. Thomsen et al. (2003) | $1.83 \pm 0.28$    | $2.07 \pm 0.06$    | $1.97 \pm 0.02$ |
| 4. Minimum Age Model     | $1.71 \pm 0.10$    | $1.92 \pm 0.09$    | $1.78 \pm 0.01$ |
| 5. Finite Mixture Model  | $2.31 \pm 0.05$    | $2.43 \pm 0.05$    | $2.36 \pm 0.01$ |

**Table 1:** Summary of results obtained using the sub-samples containing 50  $D_e$  values. Showing the minimum and maximum  $D_b$  values, and the mean  $D_b$  (and its standard error) of the 20 calculated values.

Using Method 4, the Minimum Age Model of Galbraith and Laslett (1993), for  $D_b$  analysis the average  $D_b$  values decrease until a sub-sample size of 30 after which they remain relatively constant (Fig. 3d). A sub-sample size of 50  $D_e$  values or more is needed to reduce the RSD to less than 5%.

Using the Finite Mixture Model (Method 5), of Galbraith and Green (1990), the results (Fig. 3e) are similar to the Minimum Age Model; the mean  $D_b$  values fluctuate until a sub-sample size of 30 is used, after which they remain relatively constant, and a sub-sample size of 50  $D_e$  values or more is needed to reduce the RSD to less than 5%. As one would expect, however, the minimum age model consistently derives lower  $D_b$  values than the finite mixture model.

The minimum and maximum  $D_b$  values calculated from the 20 sub-samples containing 50  $D_e$  values are detailed in Table 1. This shows that Methods 1 and 4 (Olley et al., 1998, and the Minimum Age Model) calculate the lowest  $D_b$  values for all the datasets, whilst the Finite Mixture Model calculated the highest  $D_b$  values. The errors associated with the  $D_b$  values vary considerable as a result of the different ways in which they are calculated for the individual methods. Between the various methods there is up to 40% difference in the mean  $D_b$  values, demonstrating the necessity of careful consideration when choosing a statistical technique for  $D_b$  calculation.

### Discussion and Conclusions

With the exception of Method 2, the RSD of the  $D_b$  values calculated from sub-samples is always less than 5% for sub-samples containing 50  $D_e$  values. In general, the results from Methods 2 and 3 show more variation in the final  $D_b$  values than the other models; this suggests that techniques based on the inclusion of increasing values until a predefined parameter is reached encounter problems with low, outlying, values. This is particularly demonstrated by the results for Method 2 owing to the fact that the errors on the individual  $D_e$  values are not taken into consideration. Only the  $D_b$  values calculated using the Finite Mixture Model are consistent with the result obtained from the entire dataset of 122  $D_e$  values ( $2.38 \pm 0.02$  Gy, calculated using the Finite Mixture Model) as one might expect.

Sample Aber/70KLA1 has an overdispersion parameter of 37%, other work has detailed  $D_e$  distributions with similar or greater values of overdispersion (e.g. Olley et al., 2004; Arnold et al., 2007), demonstrating that it is not an unusual situation. The results from the normality tests on the sub-samples of Aber/70KLA1 suggest that 50  $D_e$

values are necessary to be certain of determining whether the sample is non-normal; datasets consisting of less than 50  $D_e$  values may appear to be well-bleached when in fact the sample is not. The results from the different statistical models also indicate that, for most of the methods, at least 50  $D_e$  values are necessary to obtain reproducible estimates of  $D_b$  for this sample, and the  $D_b$  values calculated using the Finite Mixture Model were consistent with the  $D_b$  from the entire dataset of 122  $D_e$  values. Therefore, for analysis of samples from a similar depositional environment which may be heterogeneously bleached, 50  $D_e$  values are recommended as the minimum working population.

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## Reviewer

A. Lang



# LED laboratory lighting

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## Introduction

Over the last three decades there have been various recommendations about laboratory lighting. These have changed with changing understanding of the luminescence responses of quartz and feldspars, as well as with changes in the availability of affordable lighting technology (e.g., Sutton and Zimmerman, 1978; Jensen and Barbetti, 1979; Spooner and Prescott, 1986; Smith, 1988; Galloway and Napier, 1991; Lamothe, 1995; Spooner et al., 2000; Huntley and Baril, 2002).

In reviewing the excitation spectrum of feldspar by Ditlefsen (1991) and their own tests on loess, Huntley and Baril (2002) summarize that when one is preparing quartz, the reddest visually comfortable illumination is preferred, whereas when preparing feldspars, wavelengths in the region ca. 530-630 nm would be preferable. With advances in LED lighting technology, affordable and adjustable LED lamps of different colors have become available.

Here, we summarize our own adoption of such lighting by presenting some high-resolution spectra from representative LED lighting that we employ, and a spectrum from our filtered compact-fluorescent ceiling lamps. Since 1984 the first author has used Lee ([www.leefilters.com](http://www.leefilters.com)) number 158 (deep orange) plastic filter to cover fluorescent ceiling lamps and other laboratory lights (see also Spooner and Prescott, 1986; Smith, 1988). The DRI laboratory uses discrete-switchable, ceiling mounted, compact-fluorescent bulbs masked by 4-6 layers of Lee #158.

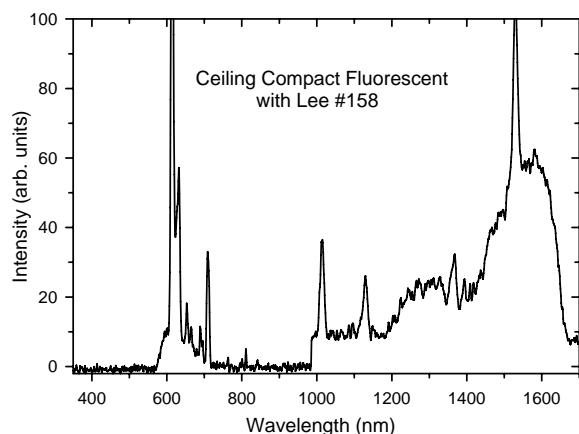
The transmission curve for # 158 in our 24-year-old LEE sample pack indicates <1% transmission below the cut-on wavelength of ca. 550 nm, but we note that the Lee website presently provides a transmission curve for #158 that shows 1-5% transmission below 550 nm. To ensure that such possible transmission (in different batches?) is reduced to insignificance, we always use 4-6 layers of filter, depending on the application.

## Instrumentation

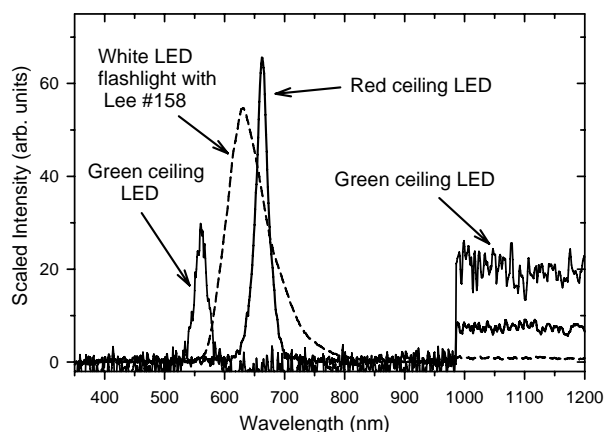
We measured optical fluxes ( $\mu\text{W}/\text{cm}^2$ ) from our various light sources using a NIST-traceable calibrated radiometer (Model IL-1400a, International Light, [www.intl-lighttech.com](http://www.intl-lighttech.com)). Filter F-15957 and a cosine diffuser are mounted over the UV stabilized silicon photodiode detector (SEL033). We measured spectra over the range 350-2500 nm, using an Analytical Spectral Devices (ASD) Inc. FieldSpec® Pro spectroradiometer ([www.asdi.com](http://www.asdi.com)). This spectroradiometer consists of 3 detector arrays (and diffraction gratings), with response boundaries at ~980 nm and ~1800 nm. The response function has a 10-20% variation over the interval 520-800 nm, and the response falls off by a factor of 4 between ~800 nm and 950 nm. The response increases again above 1000 nm and becomes essentially flat in the range 1400-2400 nm. We have applied no response corrections to the spectra presented below, primarily because the spectroradiometer response is essentially flat in the main region of interest (520-800 nm) and because we detected no emissions from our laboratory lights in the region 800-1200 nm.

## Laboratory lights

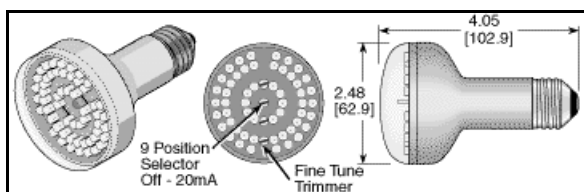
The use of 4 to 6 layers of Lee #158 filter on the ceiling lamps ensures that the intensity at our laboratory bench surfaces ranges spatially from ~30  $\text{nW}/\text{cm}^2$  to ~100  $\text{nW}/\text{cm}^2$ . Under some working conditions we raise this general illumination to a localized maximum of ~230  $\text{nW}/\text{cm}^2$ . A typical spectrum from our ceiling lamps is shown in Fig. 1. The step response at ~990 nm manifests a detector boundary offset between separate detectors within the FieldSpec® that cover different portions of the spectrum. This offset is unimportant here because we are interested only in the relative signals within each portion. It is clear that: 1) a few layers of Lee #158 adequately blocks light below the cut-on wavelength; and 2) the compact fluorescent lamps emit nothing significant in the range 720-1000 nm, the range encompassing the feldspar near IR resonance (~880 nm, e.g., Hütt et al., 1988; Aitken, 1998).



**Figure 1:** Spectrum from a filtered (Lee #158) compact fluorescent ceiling lamp, mounted in a reflector-floodlamp configuration (Lumatech Lighting, Model 1051x for 5W compact fluorescent bulbs, [www.lumatechlighting.com](http://www.lumatechlighting.com)), showing the typical Hg emission lines superimposed on a fluorescent-lamp emission continuum.



**Figure 2:** Spectra for three types of LED lamps. The relative noise in the green-lamp spectrum reflects our use of a relatively short signal-integration time for this lamp.



**Figure 3:** Format of the adjustable screw-base, colored LEDtronics darkroom safelight bulbs. The dimensions are in inches and mm (parentheses) (image from [www.ledtronics.com](http://www.ledtronics.com)).

For close-in work around the laboratory when samples are not exposed, or to momentarily read beaker labels, we employ LED flashlights ('Long Life LED Light', Item 809-1050-0, The Brinkmann Corporation, [www.brinkmann.net](http://www.brinkmann.net)) covered with 3-4 layers of Lee #158. These also emit nothing significant in the range above 800 nm (Fig. 2). However, these LED flashlights and our filtered ceiling lamps are probably unsuitable for direct or long exposures of feldspars because of their relatively broad emissions (Figs. 1 and 2).

### Adjustable LED bulbs

For feldspar preparations, either of two recently available LED lamps with adjustable-intensity could be used. The LED darkroom safelights that we employ are available from LEDtronics, Inc. ([www.ledtronics.com](http://www.ledtronics.com)). In the adjustable-intensity category with standard screw bases for 120V AC operations, only two from LEDtronics are suitable for luminescence dating laboratories: a green and a red lamp (LEDtronics R20 darkroom bulbs, BSD-1293 series). Yellow (peak emission ~590 nm) non-screw-base LED miniature bulbs are available from LEDtronics, but not in the favorable format that is available for red and green lamps. This format is shown in Fig. 3. The attraction of this format lies not only in the convenient screw-in base, but also in the availability of a position-selector switch and of a fine-tune trimmer. We use only the trimmer to reduce the intensity. The spectra from our installed LED bulbs are shown in Fig. 2. These red and green LED lamps have narrower emissions than those outlined by Mauz et al. (2002) and thus do not require additional filtering. They are rated at having  $> 10^5$  hours operating life, very low power consumption, and vibration resistance.

Adjustable-intensity LED bulbs with screw bases are also available from Kurtzon ([www.kurtzon.com](http://www.kurtzon.com)), and perhaps other firms, at similar peak wavelengths to those of the red and green LEDs in Fig. 2. These Kurtzon bulbs, however, have significant emissions at the long-wavelength side of the peak, that are not present in the spectra from the LEDtronics bulbs. For example, the Kurtzon red (660 nm) LED has undesirable emissions (12% of the peak intensity) in the range ca. 850-950 nm, and again above 1000 nm. Thus, caveat emptor, not all LED bulbs are alike. In a 2001 brochure, Kurtzon stated that 585 nm ('yellow') and 610 nm ('orange') LED screw-base bulbs were available, but they do not appear to be presently available. Kodak (via distributors) provides a LED screw-base darkroom safelight, but no spectral emission information is readily attainable, and it is likely that this is a bulb manufactured by LEDtronics (which supplies Kodak, among other firms).

For aid in loading single grains of quartz into Risø single-grain discs we use the LEDtronics red LED bulb inserted into a standard goose-neck desk lamp, and trimmed to reduce the intensity 20 cm below the bulb center to a level of  $5 \mu\text{W}/\text{cm}^2$ . This seems to be near a minimum comfortable-viewing level with this bulb. While being loaded, the discs are exposed to typically 2-3  $\mu\text{W}/\text{cm}^2$  of this red LED at their off-axis positions. This red illumination provides a surprisingly more comfortable viewing experience of contrasts and textures than the normal broadband red safelight of typical photographic darkrooms.

### Sample signal-loss tests

We have tested the effects of a 15 hr exposure to the red and green LED bulbs on 'natural' polymineral 4-11  $\mu\text{m}$  grains of a 11.5 ka loess from South Island New Zealand (Berger et al., 1996). Four sample discs were centered 20 cm below the desk-lamp red LED ( $5 \mu\text{W}/\text{cm}^2$ , trimmed intensity as stated above) and 4 discs were centered 131 cm below the ceiling mounted green LED (74  $\text{nW}/\text{cm}^2$ , untrimmed intensity). The red LED exposure reduced the first 5 s of IRSL (readout at  $30^\circ\text{C}$ ) by  $13\pm 4\%$  and the green, by  $4.3\pm 4.7\%$ . Assuming a linear reduction, these changes correspond to 0.9%/h loss of IRSL for this red exposure and  $\sim 0.3\%$ /h for this green exposure. Clearly, feldspar IRSL would be negligibly affected by either bulb if exposures under these irradiance conditions are short enough (e.g., 15-20 min. red, and 30-40 min. green). These unequal effects on IRSL partly reflect the different irradiances of these particular exposures and partly the different spectral sensitivities of feldspar discussed by Huntley and Baril (2002).

We also exposed Risø calibration quartz (150-250  $\mu\text{m}$ , previously drained in a SAR run, re-irradiated to  $\sim 6$  Gy) under the same irradiance geometries as above, but for 19.5 hours. With a preheating of  $250^\circ\text{C}$  (10s) and a cut heating at  $180^\circ\text{C}$ , the red LED reduced the SAR  $L_0/T_0$  by  $2.5\pm 2.8\%$  and the green LED, by  $1.4\pm 2.1\%$ , statistically barely detectable. Assuming a linear reduction, these changes correspond to a negligible  $\sim 0.1\%$ /h reduction in quartz  $L_0/T_0$  from the red and a negligible  $\sim 0.07\%$ /h from the green. We deduce that exposures of quartz to these configurations of these LEDs for up to a few hours (red or green) would have no significant effect.

In summary, these adjustable-intensity LEDtronics lamps are long-lived, cool running, affordable (ca. \$180 each), provide precisely defined and wavelength-stable emissions, and have little or no effect on feldspar IRSL and quartz PSL (photon-stimulated luminescence) under convenient configurations.

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### Reviewer

A.K. Singhvi

**Reviewer comments:** It is useful to be reminded of an important part of luminescence dating. LED illuminations are the future, as besides giving a controlled spectrum they also reduce scattered light in the laboratory.



## Thesis Abstracts

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**Author:** Sébastien Huot  
**Thesis Title:** Investigations of alternate and innovative ways of performing luminescence dating in an attempt to extend the age range  
**Grade:** PhD  
**Date:** December 2007  
**Supervisor:** Andrew Murray  
**Address:** Department of Earth Sciences, University of Aarhus, Denmark

At the present stage in its development, optically stimulated luminescence (OSL) using quartz has been demonstrated to provide accurate ages from ~ 5 years (Ballarini et al., 2003) to ~ 125 ka (Murray and Olley, 2002). A handful of dates from known age sediments have been published beyond 125ka. However, most of these were the consequence of a low environmental dose rate (the luminescence age is calculated by dividing dose by dose rate) rather than because of an unusually large accumulated dose. This project concerns investigating the possibility of extending the age range of luminescence dating. More precisely, it is about finding and/or validating a different measurement approach that will enable us to measure a much larger equivalent dose.

The primary objective of this project consists of investigating the reliability of alternate ways of measuring the luminescence from quartz and K-feldspar minerals. Part I discuss a novel approach for measuring the luminescence from quartz (isothermal thermoluminescence). It showed a potentially higher saturation growth curve, which could enable the measurements of older sediments. Sensitivity changes, between the measurement of the natural and regenerated luminescence prevented accurate dating. Despite intensive investigations into material from a variety of sources, it has proved impossible to develop a reliable measurement procedure.

Efforts were concentrated on the luminescence from feldspars during the second part of the thesis and those are presented in Part II. Feldspars have long been known to have an extended dose response, but athermal fading of the dosimetry signal has prevented their use. A novel approach was investigated, namely time-resolved luminescence, which potentially holds a means to circumvent athermal fading. Investigation showed it was not the case, however.

Methods have been proposed to correct for this fading, but with limited testing. The application of such correction methods to known age samples with

doses of up to ~ 160 Gy shows promising results. It is important that evaluation of fading rates be as precise and accurate as possible, because of the problem of multiplication of errors. A reduction of the scatter encountered in fading measurements was obtained by using helium instead of nitrogen as the atmosphere in our measurement system. The gas improves thermal contact and makes thermal pretreatment more reproducible, and thus reduces variability in the feldspar luminescence signal.

The potential of sodium feldspars (Albite) was briefly explored in extending the age range. Albite has very similar luminescence properties as potassium feldspars, the latter being the common mineral used in dating. Albite has the advantage of having a simpler dosimetry, compared to potassium feldspars, due to the absence of internal K, thus extending the age range and reducing uncertainties associated with the determination of internal K.

Application to dating burial ages from sediments was achieved through collaborative efforts with visitors coming to the Nordic Laboratory for Luminescence Dating. Most projects contained at least one reliable independent age to ascertain the validity of our dating procedure.

**Author:** Daniel A. Bush  
**Thesis Title:** Application of luminescence dating to the study of archaic age anthropogenic mounds from the lower Mississippi River Valley  
**Grade:** PhD  
**Date:** January 2008  
**Supervisor:** James K. Feathers  
**Address:** Luminescence Laboratory, University of Washington, Seattle, USA

Intense solar bleaching of minerals in active surface soils is a widespread phenomenon. The process of soil turbation distributes soil-bleached grains to some depth. These grains have been viewed variously as a source of contamination in optically stimulated luminescence (OSL) dating of deposits and as a source for chronological information about the soil itself. This work investigates the possibility of applying OSL dating to buried soils using coarse-grained quartz. It focuses on buried soils with reliable

independent age assessments from earthen mounds of archaeological import.

Special attention has been given to developing a soil genesis model for the distribution of solar-bleached grains through the depth of a soil horizon. In this model the soil surface is viewed as a bleaching platform that is continuously stressed and loaded by subsurface turbation mechanisms creating surface relief. Subaerial impact- and transport-forces, such as rain, wind and floral growth, impact surface relief causing disaggregation and single-grain dispersion. This surface "bleaching zone" offers frequent and thorough presentation of mineral grains to solar radiation. In this model, soil turbation distributes bleached grains through the soil horizon and brings unbleached grains to the surface.

Absorbed doses in single-aliquots and single-grains of quartz are determined using the single-aliquot regenerative dose (SAR) protocol. Modern active soils similar in composition and environment to paleosols at depth are analyzed as controls. The concentration of zero-age grains decreases with depth. Minimum age analysis of grains from within 5 cm of the surface results in near-zero ages.

Dose depth profiles were studied from 35 cm long cores through 9 buried paleosols from the interior of prehistoric earthen mounds. The position of the buried soil surface within the core was determined from field measurements and confirmed by extensive laboratory soil analysis. Dose distributions with paleosol depth conform to our model and meet expectations of aged buried surfaces. OSL minimum ages from these buried soils demonstrate good agreement with previously published ages. It is concluded that many soils are competent mechanisms for thorough exposure of near surface minerals to solar radiation.



## Research theses online

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Recent visitors to the website of Ancient TL ([www.aber.ac.uk/ancient-tl](http://www.aber.ac.uk/ancient-tl)) will have noticed that there is a new section providing access to a number of research theses that may be of interest to those involved in luminescence and electron spin resonance research.

There are currently nine theses ranging in time from 1981 to 2006, and covering a wide range of topics. These PDF files have been provided by the authors and made available to the general community. Theses often contain a great deal more work than that which is published and I hope that the web page on the Ancient TL web site will be a valuable service, making it much simpler to obtain material that is often difficult to access.

A list of the theses that are currently available is given below in alphabetical order. Further contributions are gratefully received and should be sent, either by e-mail or on CD, to me. There is no restriction upon what language the theses are written in. Please ensure that the entire thesis is contained within a single PDF file.

### Geoff Duller

Baril, M.R. (2002) *Spectral investigations of luminescence in feldspars*, Unpublished PhD thesis, Simon Fraser University, (5.2 Mb)

Bøtter-Jensen, L. (1999) Development of optically stimulated luminescence techniques using natural minerals and ceramics, and their application to retrospective dosimetry, DSc thesis, University of Copenhagen (1.8 Mb)

Duller, G.A.T. (1992) *Luminescence chronology of raised marine terraces, south-west North Island, New Zealand*, Unpublished PhD thesis, University of Wales, Aberystwyth (4.0 Mb)

Murray, A.S. (1981) *Environmental radioactivity studies relevant to thermoluminescence dating*, Unpublished DPhil thesis, University of Oxford (25.2 Mb)

Olley, J.M. (1994) *The use of  $^{238}\text{U}$  and  $^{232}\text{Th}$  decay series radionuclides in sediment tracing*, Unpublished PhD thesis, Australian National University, Canberra (34.0 Mb)

Rodnight, H. (2006) *Developing a luminescence chronology for late Quaternary fluvial change in South African floodplain wetlands*, Unpublished PhD thesis, University of Wales, Aberystwyth (12.5 Mb)

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### Editors' Note

As described in the editorial, the editor would like to take this opportunity to thank Prof Ann Wintle for putting together the bibliography for Ancient TL for 23 years, providing an invaluable service to everyone in the luminescence and ESR community.

Future bibliographies in Ancient TL will be produced by Dr Daniel Richter of the Max Planck Institute, Leipzig, who will be joining the Reviewers Panel from 1st July onwards. Colleagues wishing to have their papers included in the bibliography are asked to send hard copies or PDFs of their papers to Dr Richter at 'Max Planck Institute for Evolutionary Anthropology, Department of Human Evolution, Deutscher Platz 6, 04103 Leipzig, Germany' or by e-mail ([drichter@eva.mpg.de](mailto:drichter@eva.mpg.de)).

## Institute of Physics Awards 2008

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In 2008, two eminent scientists in the field of luminescence were honoured with the award of medals by the Institute of Physics. The Appleton medal was awarded to Professor Ann Wintle and the Gabor medal to Doreen Stoneham. The awards were formally made on 24th January 2008 in London. Excerpts from the citations accompanying each award are reproduced here.



*Professor Ann Wintle receiving the Appleton medal from the President of the Institute of Physics, Peter Saraga.*

### **2008 Appleton medal and prize Professor Ann Wintle**

The Appleton medal and prize for distinguished research in environment, earth or atmospheric physics has been awarded to Professor Ann Grace Wintle, Professor Emeritus in the Institute of Geography and Earth Sciences, University of Wales Aberystwyth, for her outstanding contribution in the development and application of luminescence properties of minerals as a geological dating tool applicable to the past one million years.

In 1979, Wintle showed that the TL signal from mineral grains in deep sea cores could be extracted. This seminal discovery opened up a whole new dating technique for Quaternary sediments which can

extend back beyond the range of radio-carbon dating and is applicable to a wider range of sediments.

Since 1991, Wintle became involved in applying the more precise technique of optically stimulated luminescence (OSL), first from feldspars and then quartz. During the past 10 years, she has been at the forefront of developing methods for dating quartz, carrying out basic experiments to characterise the OSL signals.

Her research has also included applications such as dating coastal sediment in Africa, including sands from caves with early human remains from 70,000 years ago, and wind blown silt deposits in China going back to about one million years.



*Doreen Stoneham with the Gabor medal, accompanied by her husband Marshall Stoneham*

### **2008 Gabor medal and prize Doreen Stoneham**

The Gabor medal and prize for distinguished work in the application of physics in an industrial, commercial or business context has been awarded to Mrs Doreen Stoneham, Director and Company Secretary of Oxford Authentication Ltd, for her successful establishment of a world-leading company that authenticates ceramics for the art world.

Oxford Authentication Ltd, founded by Mrs Stoneham, leads the world in authenticating ceramic antiquities. Its certificates are accepted by all leading auction houses, museums, collectors and art dealers.

The technique used is thermoluminescence (TL) which gives an objective guide to when the ceramic

was last fired. It was pioneered at Oxford University's Research Laboratory for Archaeology and the History of Art (RLAHA), where Stoneham worked successfully developing research laboratory approaches into reliable commercial methods for combating art forgery. She developed a method of authenticating high-fired ceramics like stoneware and porcelains using a novel sampling approach.

Since TL depends on the radiation that the sample has been exposed to, it can be used as a dosimetry technique. Building on her extensive experience with ceramics, she was able to devise new dosimeters to measure radiation exposure at nuclear sites. As a result, she was an expert witness in crucial

international evaluations of the Chernobyl and Hiroshima nuclear events.

When in 1997, RLAHA decided to move its research away from TL, Stoneham seized the opportunity to start a company to continue providing the commercial authentication service. Building on a reputation for unmatched technical expertise and for vigilance in refining techniques to beat the forgers' tricks, Stoneham has won the trust of the international legal and art worlds. The result is that company has a world-wide clientele.