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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.

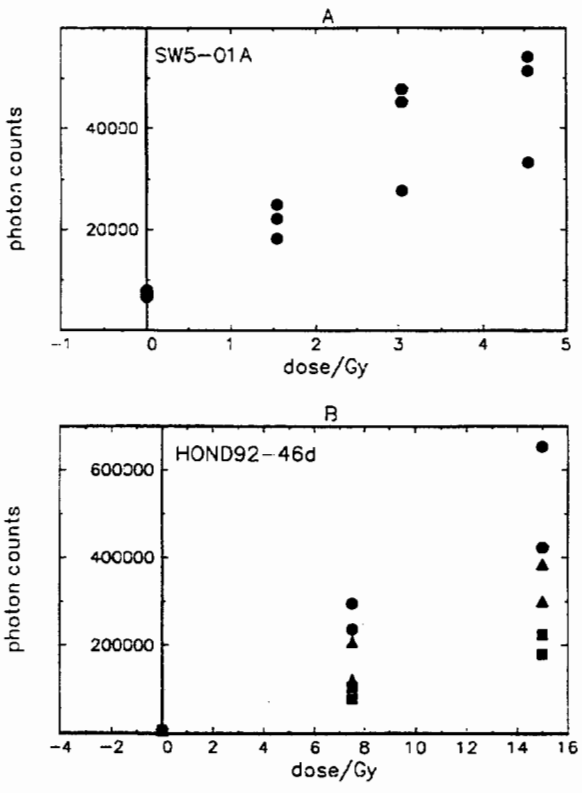

Figure 1.

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.

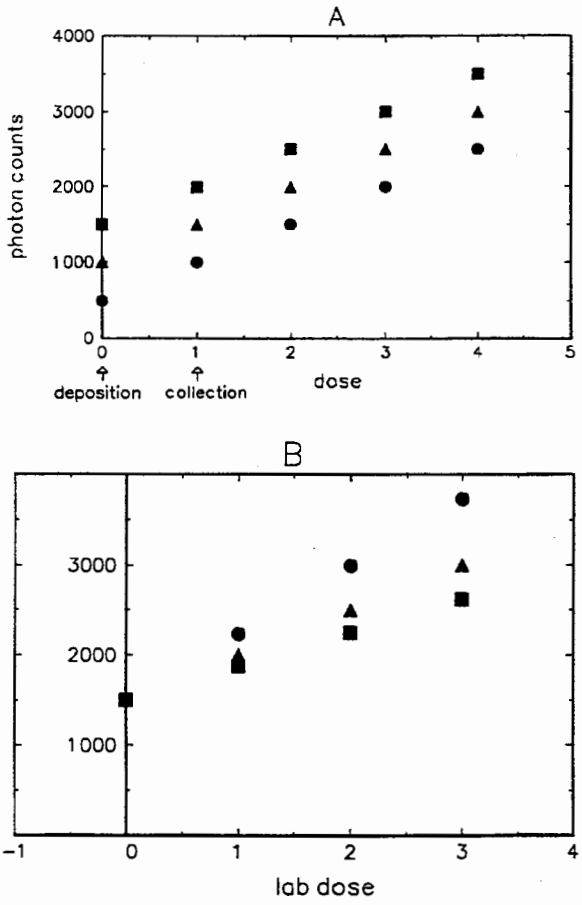

Figure 2.

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.

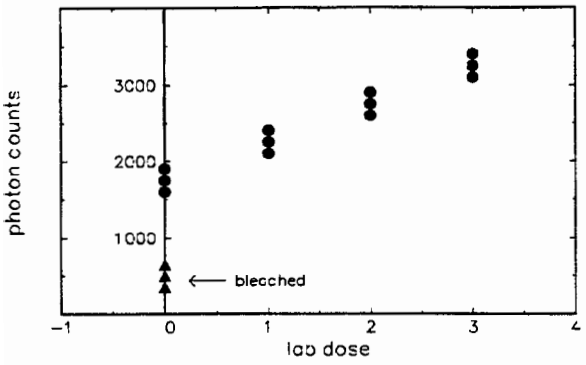

Figure 3.

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

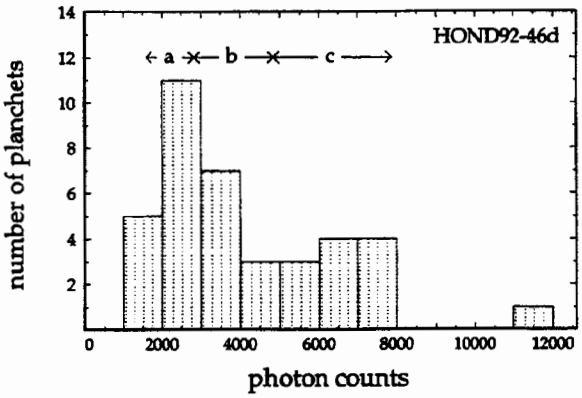

Figure 4.

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.

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.

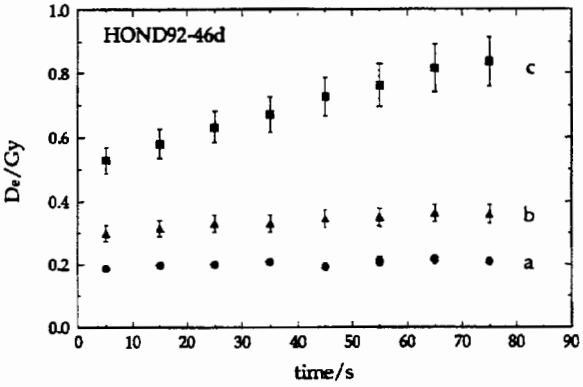

Figure 5.

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, ~ 18 $\mu g\ g^{-1}$, whereas the uranium content is normal, ~ 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 ~ 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).

Acknowledgements

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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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