Improvements in infra-red dating of partially bleached sediments – the 'Differential' Partial Bleach Technique

A.K. Singhvi[1] and A. Lang[2]

[1]Earth Science Division, Physical Research Laboratory, Ahmedabad 380 009, India
[2]Forschungsstelle Archäometrie, Heidelberger Akademie der Wissenschaften, Max-Planck-Institut für Kernphysik, Postfach 103980, D-69029 Heidelberg, Germany

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Abstract *- A new approach to estimate optical ages making use of the most light sensitive component of partially bleached sediments is proposed. A differential partial bleach technique is used where the stimulation during measurement substitutes for the laboratory bleach. Results on infrared stimulated luminescence from fine grain sediments of diverse depositional environments, based on this approach are presented. Improvements are obtained in the case of poorly bleached sediments, whereas results for well bleached sediments are consistent with other optical dating results. The differential partial bleach technique is seen as primarily applicable to silt-sized sediments, for which single-grain dating is not feasible.*

Introduction

During the past few years a considerable effort has been expended towards developing methodologies for luminescence dating of fluvial, glacio-fluvial and colluvial sediments (see e.g. Berger, 1988, 1990). In contrast to aeolian deposits such sediments receive a substantially reduced daylight flux and a narrower spectrum for photobleaching prior to deposition. This is due to a combination of sediment load, turbulence in the medium and clumping of sediment particles. In such sediments there are three possible scenarios of pre-depositional daylight exposure, viz.,

(1) that all grains experienced equal but a substantially attenuated bleaching,
(2) that the grains were bleached to varying extent,
(3) a combination of the above, i.e. the grains were both poorly and heterogeneously bleached.

To address to the problem of uniform insufficient bleaching (#1 above), the partial bleach TL method was developed. This was based on an assumption that the luminescence level of a mineral grain comprises several components with different sensitivity to photobleaching (Wintle and Huntley, 1979, 1980; Huntley, 1985). Thus in a uniformly but partially bleached sediment with a complex history of depositional photobleaching it is expected that only a part of the signal was bleached and the other part was not affected. It is then considered that as long as the dating technique measures only such photosensitive signals that were bleached at the time of deposition, reliable age estimates could be expected. A series of sun-exposures are made on different sets of aliquots and additive growth curves obtained for each of these sets as well as for the unbleached set. The equivalent dose (or palaeodose, P, as used henceforth) is determined from the intersection point of the unbleached and the bleached growth curves. In the next step the dependence of palaeodose on varying daylight exposure is examined to deduce the most appropriate bleaching condition, beyond which an overestimation of the age would occur (Fig. 1). Further understanding of the appropriate bleaching conditions were found by Berger and Lutenauer (1987) who measured the net sun spectra in turbid water (~ 500 nm to ~ 690 nm) and based on this suggested that the wavelength < 550 nm and > 700 nm should be blocked during laboratory photobleaching. This can be achieved by filtering the daylight through a combination of Schott OG 570 and KG 5 filters.

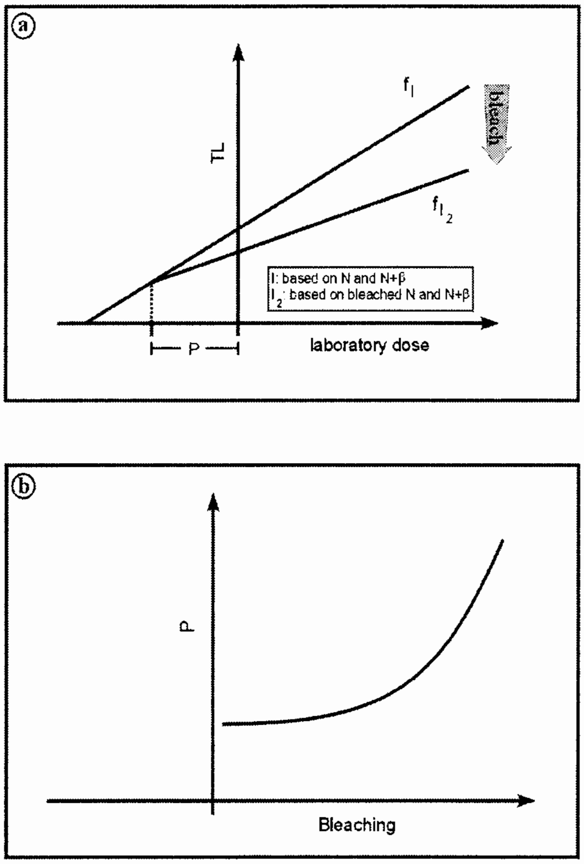

Figure1.

(a) Diagram for P determination in the partial bleach technique used in thermoluminescence dating.

(b) Expected behaviour of palaeodose with bleaching using the partial bleach technique. The palaeodose estimate will increase after the laboratory photobleaching exceeds the depositional photobleach level.

Development of optical dating with direct measurement of the easily bleached component brought the optimism that more sediments of different depositional environments could be dated, than was possible with TL (Huntley *et al.*, 1985; Hütt *et al.*, 1988). However, results of insufficient bleaching of OSL in specific situations has been reported, for example by Duller (1994), Lang (1994), Li (1994) and Rhodes and Pownell (1994). Fuller *et al.* (1994) extended the concept of restricted spectrum bleaching and the partial bleach method in TL dating to infra-red stimulated luminescence to examine deposits from the river Danube. However, from a simulation experiment it was concluded that the partial bleach methodology was inappropriate for IRSL. An alternative approach was suggested by Aitken and Xie (1992), who conceptualised that the total signal comprised an 'easy-to-bleach' and a 'hard-to-bleach' component. The authors used a 'subtraction technique' to obtain the rapidly bleaching component. To achieve this, the 'late light' of each of the shine down curves (i.e. signal appearing after a long shine down) was subtracted from the 'early light' (signals at the early intervals of the shine down). The palaeodose is then determined using the additive dose method but on the rapidly bleaching component only.

We discuss here a simple technique to conduct a partial bleach analysis in optical dating which allows for probing the most photosensitive signal and uses only simple IRSL shine down curves. The new technique comprises construction of a series of partial bleach growth curves for a sample to estimate the dependence of the palaeodose (P) with increasing optical stimulation. A somewhat similar approach was employed by Spooner *et al.* (1990) for a well bleached sample. In the following the basic philosophy of the technique is presented along with the results on sediments of different depositional environments.

The technique

In the conventional partial bleach experiment a minimum of two sets of growth curves are constructed (Fig. 1). These are,

(1) a growth curve based on natural and natural + additive doses and,

(2) a second growth curve based on an identical set of aliquots but with an additional treatment of photobleaching.

Extrapolation of the two curves and the determination of their intersection point provides the paleodose P. In the present approach, the IRSL shine down curves are analysed and IR stimulation itself substitutes for optical bleaching. The intensity $I_{\Delta t_0}$ of the first interval Δt_0 of stimulation is taken to represent the natural (unbleached) signal. The intensity of all subsequent shine down intervals are considered as photobleached signal i.e. $I_{\Delta t_1}$ ... $I_{\Delta t_n}$. (Fig. 2), with increasing photobleach as stimulation proceeds. A set of partial bleach growth curves are then constructed for different intervals using the first interval as the natural additive growth curve (i.e. $f(_{\Delta t_0})$) and the subsequent intervals as the photobleached ones (i.e. $f(_{\Delta t_1})$, $f(_{\Delta t_2})$,..., $f(_{\Delta t_n})$). P is estimated from the intersection points of the growth curves, i.e. P_1 from the intersection of $f(_{\Delta t_0})$ and $f(_{\Delta t_1})$, P_2 from the intersection of $f(_{\Delta t_0})$ and $f(_{\Delta t_2})$, ... and P_n from the intersection of $f(_{\Delta t_0})$ and $f(_{\Delta t_n})$. We call this approach the 'differential partial bleach' (DPB) approach. In a similar approach Aitken and Xie (1992) suggested to use the subtraction technique where the difference of $I_{\Delta t_0}$ and $I_{\Delta t_n}$ is plotted to obtain the equivalent dose.

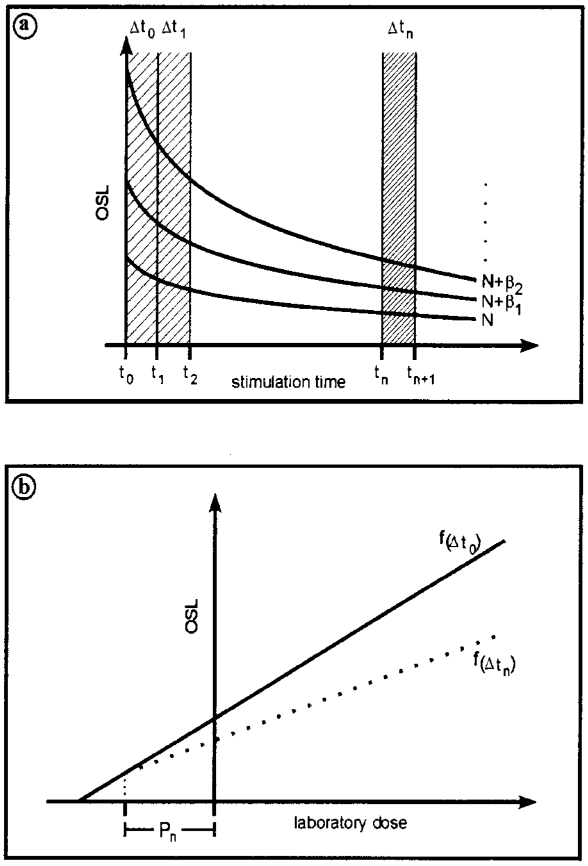

Figure 2.

(a) Schematic of the DPB approach. The intensity $I_{\Delta t_0}$ *of the first interval* Δt_0 *of stimulation is taken to represent the unbleached (natural) signal. The intensity of all subsequent shine-down intervals are considered as equivalent to laboratory bleached i.e.* $I_{\Delta t_1} \cdots I_{\Delta t_n}$.

(b) Growth curves are constructed for each interval and a palaeodose (P) is estimated - as in conventional TL partial bleach technique - at the intersection of the growth curves of unbleached and subsequent bleached intervals.

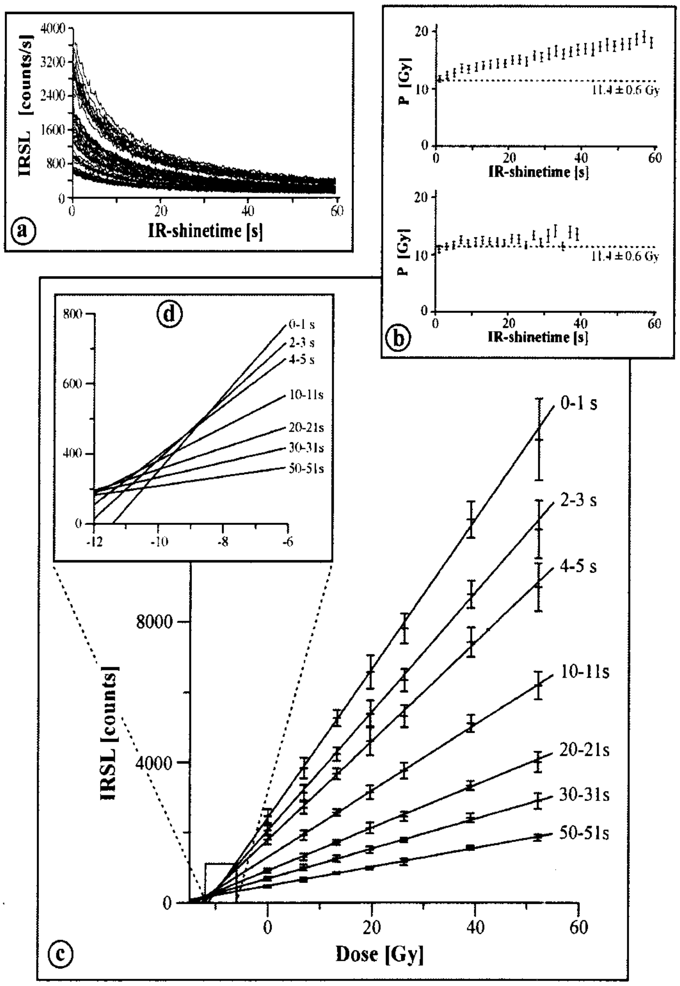

Figure 3.

(a) Shine down curves for sample HDS-180. IRSL in counts per s is plotted versus stimulation time for natural and additive dosed aliquots.

(b) Shine plots for sample HDS-180: P in Gy is plotted versus stimulation time. The upper plot shows P-values obtained using the conventional additive dose technique. The lower plot shows P-values obtained using the additive dose technique after subtraction of the 'late light' (mean signal in the interval 50 s to 60 s) The dashed lines are plotted at the additive P value obtained for the first s of stimulation (11.4 ± 0.6 Gy). No palaeodose plateau is seen.

(c) Dose response for selected intervals: integral IRSL in counts is plotted versus applied laboratory dose in Gy for different stimulation intervals (as labelled). Extrapolated lines are shown for each interval. For clarity, dose response for interval 1-2 s has been omitted.

(d) Enlargement from Fig. 2 (c): The region of intersection of the extrapolated lines is shown.

The samples

A series of silty samples from different depositional environments i.e. (a) fluvial deposits (overbank sediments), (b) colluvial deposits, and (c) aeolian deposits were investigated. Such a choice was made to examine the applicability of the present approach to samples derived from different depositional bleaching environments. It is expected that the present technique would provide an age similar to that of the conventional additive dose IRSL technique for a well bleached aeolian sample and that it should provide ages similar to that of the subtraction technique for sufficiently bleached colluvial samples. On the other hand, it should produce substantially improved ages (i.e. consistent with age controls) in case of poorly bleached proximally transported fluvial sediments. The details of samples and their depositional environments are presented below.

Fluvial deposits (Samples HDS-178 to HDS-184, MAU-EB-4 and MAU-EB-7).

All samples originate from the floodplain of the Elsenz-river near Heidelberg, Germany. These sediments consist of reworked loess, deposited as overbank sediments during floods. Samples HDS-178 to HDS-184 were recovered by drilling immediately besides the present day river channel near the town of Wiesenbach. A chronological framework for the construction of the Elsenz floodplain is provided by Barsch *et al.* (1993). Previous IRSL-studies of floodloam from besides the channel suggested very poor or no bleaching of the mineral grains at deposition (Lang, 1994). Samples MAU-EB-4 and MAU-EB-7 were taken from the floodplain at a construction site near the town of Mauer (Lang, 1996). MAU-EB-4 was taken from near the river channel, and MAU-EB-7 from the distal part of the floodplain. Radiocarbon ages on associated wood and land-snail samples provides following time frame: AD 1446-1621 for MAU-EB-4 and AD 390-530 for MAU-EB-7 (calibrated using Stuiver and Reimer, 1993).

Colluvial deposits (Samples RÖM-1 to RÖM-5)

A set of colluvial sediment samples from Wiesenbach (SW Germany) were taken. The colluvial sequence lies unconformably above a loess horizon and was deposited in response to the construction of a 'dam', such that the material further uphill that was eroded was trapped until filling of the dam was complete. Further details and results of IRSL analysis of these samples using the 410 nm emission and the subtraction technique of Aitken and Xie (1992) can be found in Lang and Wagner (1997).

Aeolian Sediment (Sample RÖM 10)

A sample of Late Würmian pristine loess was also taken at Wiesenbach (see above, detailed description in Lang and Wagner, 1997). Sedimentological characteristics and stratigraphy in the region suggest that the loess should have been deposited after the last glacial maximum. The youngest loess known in this region was laid down 14 to 15 ka ago (Zöller, 1994). Thus the sedimentation age of the sample can be bracketed to 22 to 15 ka.

Measurements

Experimental procedures *viz.* sample preparation, P and dose rate determination are described by Lang *et al.* (1996). Typically 40 - 60 shine down curves were recorded at room temperature for the paleo-dose estimation. A Risø reader TL-DA-12 (Bøtter-Jensen *et al.*, 1991) equipped with an EMI 9635Q photomultiplier and TEMT 484 diodes for stimulation (880 Δ 80 nm) was employed. The net IR power at the sample was ~40 mW/cm^2. Following Krbetschek *et al.*, (1996) the IRSL detection was restricted to 390 - 450 nm. For the construction of growth curves, typically 10 'natural' discs and 6 groups of artificially dosed discs (5 each) were measured. These satisfied the criterion for reducing the error in palaeodose to an optimum level (Felix and Singhvi, 1997). A ^{90}Sr/^{90}Y β-source (dose rate ~1 Gy/min) was used for artificial dosing and preheating of 220°C for 5 min was applied to all samples. No corrections for thermal transfer effects, such as were made by Huntley and Clague (1996), nor of the type made, for example, by Aitken and Xie (1992) and Rees-Jones and Tite (1994). Recent studies by us on mineral separates indicate that this correction may not be significant for samples with P values in excess of half a Gy. Data handling was done using G. Duller's *analyse* software. For regression analysis and calculation of intersection points the commercial software package by *Jandel Scientific* was used. In the error estimation, a weightage proportional to variance and error propagation according to the Gaussian error propagation law were applied. Dose rates were calculated from a combination of data from alpha-counting, beta-counting and high resolution low level gama ray-spectrometry (Lang *et al.*, 1996). In none of the samples was radioactive disequilibrium detected.

Results and discussion

A typical data set for a fluvial sample is provided in Fig. 3. Fig. 3a provides the shine down curves, Fig. 3b provides the shine plots using the conventional additive and late light subtraction techniques and dose response curves for different intervals are plotted in Fig. 3c. The inset (Fig. 3d) shows the different equivalent doses obtained from successive time intervals suggesting the presence of signals of different photo bleachability.
Table 1 provides the P estimates based on the new technique along with the result of conventional IRSL additive dose analysis. P estimated based on late light subtraction, the IRSL intensity in interval 1-2 sec. taken as 'late light' is also provided for comparison. The 'expected P' is calculated using the dose rate of the sample and the estimated age of the sample based on the contextual and stratigraphical information and on the radiocarbon ages. Fig. 4 provides a comparison of the P-values from the DPB analysis and P-values from conventional additive dose analysis.

(a) ***Fluvial deposits*** - Floodloam sequence HDS-178 to HDS-184. In this series, the differential partial bleach technique provides a substantial improvement compared to the standard IRSL additive dose and subtraction procedures using 50-60 s as the late light. The DPB values show a good concordance with the expected values. The only exceptions are HDS-180 and HDS-183, where although the use of DPB technique provides an improvement, still the DPB palaeodose is overestimated compared to the expected P. This would imply that the present choice of analysis intervals are still too long. The examination of sub-second intervals however was limited by the photon counting statistics and will have to await higher efficient measurement devices. Two fluvial samples from the other locality (MAU-EB) show differing results. Whereas P values calculated for MAU-EB-4 show a trend similar to HDS-180 and HDS-183, P values calculated for MAU-EB-7 (the distal sediment) are the same within error limits and fit to the expected P. The conventional additive dose P value is higher.

(b) ***Colluvial deposits***: For all the samples P values calculated are the same within error limits and fit well to the known values. The exceptions are the conventional additive dose P.

(c) ***Aeolian deposits***: All P-values determined for RÖM 10 are the same within the limits of error, again with the exception of the conventional additive dose P.

From these results two groups of samples can be distinguished. Group (1) shows a trend towards lower P values when analysing earlier intervals and therefore exhibit significant improvements with the use of the DPB-technique. Group (2) has all P values that are identical and fall within the error limits, except the conventional additive P estimates. Group (2) comprises the well bleached aeolian loess, reworked sediments like the RÖM-1 to RÖM-5 which also behave like well bleached sediments. Here the DPB estimates are comparable to other estimates. The higher conventional additive P is possibly due to the hard to bleach component of the blue emission (Rieser *et al.*, 1997; Lang and Wagner, 1997).

In view of the fact that, an optical exposure can be finely tuned in respect of the flux, wavelength and duration, the present approach permits the growth curves for situations where the signal sampling interval Δt_0 can be made as small as possible and the flux for IR-simulation may be reduced to the lowest possible level. It is considered that the approach is likely to be limited only by
(1) photon counting statistics
(2) photobleaching during sample preparation (if any), and
(3) signal loss during short shine normalisation runs.

Effects due to scattered illumination light into the detection optics, Raman scattering, thermal transfer and photomultiplier dark counts are common to both the intervals Δt_0 and Δt_n and consequently it can be reasonably expected that they will get eliminated automatically.

The technique implicitly assumes the presence of a spectrum of bleachability in the OSL signal. This is the case for polymineral fine grain samples. The technique's utility for coarse grains (and single grains) remains to be investigated. The method should compliment the single grain technique in the sense that while the single grain technique aims to locate the most bleached grains in a heterogeneously bleached ensemble of grains, the DPB method looks for the most rapidly bleachable signal in such an ensemble.

Table 1**:** *P estimates for the different approaches used. Sample No., sediment type, sampling depth (in case of the fluvial sequence) are also listed. All P values are in Gy.*

		P	**P**	**P**	**P**	**P**	**P**	**P**	**P**
	Technique:	(convent.)	(subtr.)	(subtr.)	(DPB)	(DPB)	(DPB)	(DPB)	'expected'
	Interval:	0-1s	(50-60s)	(1-2s)	20-21s	10-11s	2-3s	1-2s	
Sample	Sed.-type								
HDS178	Floodloam (70 cm)	6.1± 0.3	5.3±0.3	3.1±0.4	5.0±0.2	4.3±0.3	3.7±0.2	1.3±0.3	1-2
HDS179	floodloam (140 cm)	6.6±0.2	5.7±0.2	1.7±0.4	5.8±0.3	6.5±0.4	4.50.3±	2.4±0.4	2-3
HDS180	floodloam (260 cm)	11.4±0.6	10.6±0.5	6.8±0.5	9.9±0.8	9.5±0.7	8.5±0.6	6.6±0.5	2-5
HDS181	floodloam (365 cm)	5.1±0.3	4.5±0.2	3.3±0.6	4.4±0.4	4.4±0.4	4.3±0.3	3.8±0.3	3-7
HDS182	floodloam (390 cm)	5.0±0.3	4.5±0.3	4.1±0.5	4.6±0.5	4.4±0.4	4.3±0.4	4.0±0.3	3-8
HDS183	floodloam (650 cm)	36.5±1.7	35.4±1.7	33.2±1.9	35.2±2.2	34.6±2.4	31.2±2.2	35.9±5.6	10-20
HDS184	floodloam (680 cm)	17.3±1.0	16.3±1.1	16.9±3.1	16.1±1.2	16.0±1.3	16.9±1.4	15.3±1.4	12-25
MAU EB 4	floodloam	24.0±1.5	22.5±1.5	16.3±1.4	21.1±1.5	20.1±1.5	17.0±1.4	15.9±1.4	1.5-2.5
MAU EB 7	floodloam	4.6±0.2	4.3±0.2	4.7±0.6	4.2±0.2	4.1±0.2	3.9±0.2	3.9±0.2	4.5-6.0
RÖM 1	colluvium	4.2±0.3	2.0±0.1	2.0±0.5	2.1±0.2	2.0±0.2	2.2±0.2	2.2±0.2	0-4
RÖM 2	colluvium	5.2±0.2	2.6±0.1	1.4±0.2	2.4±0.3	2.4±0.2	2.2±0.2	2.1±0.2	0-4
RÖM 3	colluvium	5.5±0.4	3.3±0.2	3.8±0.5	3.3±0.4	3.4±0.4	3.8±0.4	4.0±0.4	2-6
RÖM 4	colluvium	8.4±0.4	8.0±0.4	7.0±1.3	7.9±0.5	7.8±0.5	8.2±0.5	7.4±0.5	6-11
RÖM 5	colluvium	14.2±1.2	11.7±1.0	10.7±2.2	9.9±1.2	11.3±1.3	11.7±1.4	12.0±1.3	7-12
RÖM 10	loess	69.6±2.7	62.4±2.2	56.5±5.8	59.7±2.8	57.8±3.0	56.8±2.9	57.7±3.1	50-75

(convent.): P is determined using the 'conventional' additive dose technique and the IRSL signal of the first second of stimulation (0-1 s intervals).

(subtr.): Subtraction technique P estimates using the integral 50-60 s or 1-2 s (as indicated) for determination of the 'late light'.

(DPB): P estimates were determined using the differential partial bleach technique as described in the text. P was calculated from the intersection point of the growth curve constructed for the first interval of stimulation and the growth curve of the specified interval.

'expected': P is calculated using calibrated ^{14}C ages, stratigraphy of the sediments and dose rate determinations as described in the text.

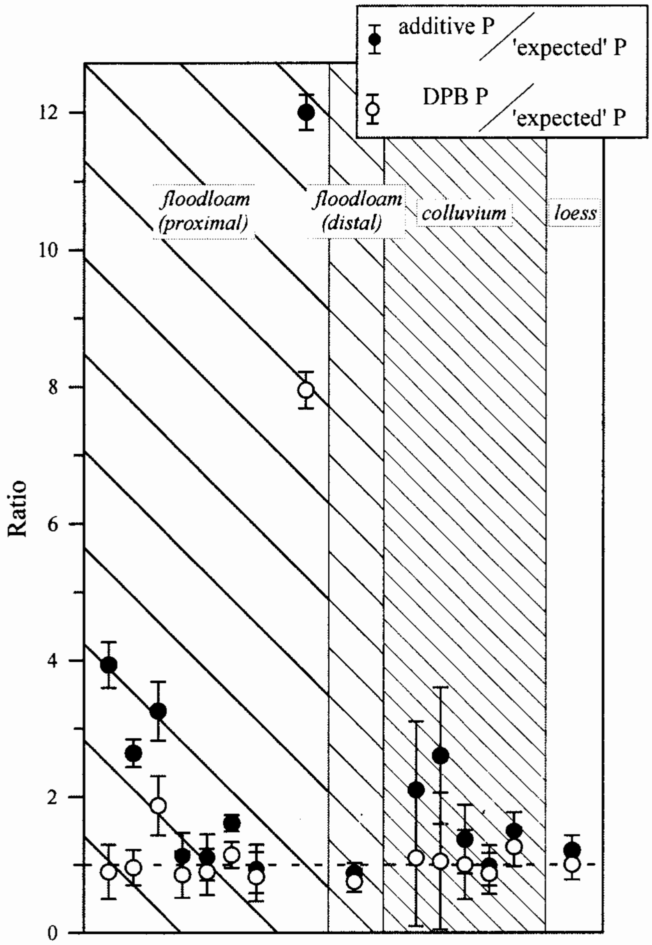

Figure 4.
Comparison of the P estimates from samples of different depositional environments using the DPB and the 'conventional' additive dose techniques. The results are presented as ratios. P estimates for DPB were obtained from the intersection point of the 0-1 s growth curve and the 1-2 s growth curve. The samples are (from left to right):

floodloam (proximal): *HDS 178, HSD 179, HDS 180, HDS 181, HDS 182, HDS 183, HDS 184, MAU EB 4*

floodloam (distal): *MAU EB 7*

colluvium: *RÖM 1, RÖM 2, RÖM 3, RÖM 4, RÖM 5*

loess: *RÖM 10*

Conclusions

The consistency of results obtained for a variety of sediments from different depositional environments enables a reasonable conclusion that the DPB technique will prove useful for the samples that experienced very limited daylight exposure prior deposition. Despite the pessimism of Fuller *et al.* (1994), it appears that the partial bleach approach as described here at any rate is not only useful but is perhaps essential when dating sediments which may be poorly bleached. This approach, as also the case with the single grain analysis being developed by e.g. Lamothe *et al.* (1994) and Murray and Roberts (1997) or single aliquot technique (e.g. Duller, 1995), should imply a significant advance in the dating of fluvial, colluvial and glacial/nival deposits.

As of now we consider the limiting factors to be only the photon counting statistics when dealing with sub-second intervals, and the luminescence intensity lost in short shine normalisation procedures. For the moment the technique should prove useful especially in the case of 'bright' samples. For polymineral samples and low P values the DPB technique is limited until more efficient light measurement devices are available. As for now, using a different detection window (560nm; Krbetschek *et al.*, 1996) may reduce the problems due to the relative large scatter in IRSL and in addition, may probe a more easy to bleach emission. However, the long-term stability of this signal is yet to be demonstrated. It would be interesting to see to what extent the technique is effective with using visible wavelengths for stimulation and to samples other than polymineral. It would also be of interest to examine cases where samples show a complex shine down curve - rising initially and decaying subsequently (Rieser et al. 1997) and to apply the DPB technique to samples artificially partially bleached in the laboratory.

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Reviewer :
Martin Aitken

Comments

1. In essence this is an extension of the subtraction method of Aitken & Xie (1992) ; it differs in the way the data are handled --- as the authors indicate. A further development that might be worth considering would be to use the successive adjacent time intervals instead of always using the first time interval ; this should give better separation of components of differing bleachability though of course these harder-to-bleach components will have suffered a minor amount of bleaching. However, in terms of obtaining the palaeodose for the easiest-to-bleach component there would be no advantage.

2. Because the same data are used for each it is to be expected that the palaeodose obtained by subtraction using a given time interval for the late-light will be the same as that obtained by DPB technique for that interval. The Table enables this comparison to be made for the 1-2 s interval and it will be seen that there is agreement except in respect of HDS178 and RÖM 2 ; these are only two of several low palaeodose samples, and also since the discrepancies are in opposite sense, this feature does not seem likely to be indicative of a systematic effect associated with low palaeodose.

3. I disagree with the authors' expectation that the technique will eliminate thermal transfer effects. My own expectation, consistent with observations (unpublished) I made some years ago using green-light stimulation, is that the shine-down curve for the transferred component will have the same shape as that of the dating signal (the « natural ») ; hence the transferred component will constitute the same percentage of the signal for all time intervals and this will lead to an overestimation of palaeodose by that percentage irrespective of whether the DPB or the subtraction technique is used. Of course for many samples the transfer component is, in any case, barely significant.