Ancient TL

Further comparisons of quartz OSL additive dose palaeodoses generated using long and short duration pre-heats

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Introduction

Over approximately the past year there has been some debate on the potentially malign effects of long duration pre-heat procedures on OSL palaeodoses (P) generated for coarse-grained quartz samples. This position has, for the most part, been championed by Roberts and colleagues (Roberts et al., 1993a,b, Roberts, 1994). They have specifically suggested that short (duration of minutes) pre-heats at temperatures above 200°C produce correct palaeodoses, whereas long (duration of hours) pre-heats at temperatures below 200°C result in palaeodose underestimation (Roberts et al., 1993a). This has been demonstrated by Roberts et al. for a series of five samples (one from North Africa, and four from Australia). While small in number, their analyses for these samples are used as a basis for hypothesising that long duration, relatively low temperature pre-heats cause dose-dependent sensitivity changes which influence additive dose growth curves.

Being an advocate of long duration pre-heat procedures in quartz optical dating (Stokes, 1992) I find their data of interest. I have accordingly undertaken a somewhat more extensive intercomparison on a series of 45 samples collected from Australia, South Africa, Botswana, Egypt, England, Holland, Mexico, the United States, and Zimbabwe (described in more detail in Stokes (1994)). Sample ages range from a few hundred to approximately 125 000 years. Clearly the samples are of varied provenance, and were collected from a range of depositional environments. The sampling of such a variety of materials was undertaken to attempt to facilitate measurement of as wide a range as possible of the 'spectrum' of quartz OSL behaviour. This paper describes the results of the intercomparison.

Experimental Procedures

All measurements were made on the quartz fraction as routinely refined from bulk sediment samples (e.g. Stokes, 1992). Cleaning procedures included HCl (1N) treatment, wet sieving (90-150 μm), magnetic and heavy liquid (ρ = 2.75 gm.cm^{-3}) separations, and etching using both HF (48%) and H$_2$SiF$_6$ (40%). All separation procedures took place under subdued red light. Cleaned samples were mounted as monolayers on 9.9 mm dia. stainless steel discs using a silicone oil (aliquot masses c. 5 mg). Typically 26 aliquots were prepared for the construction of each growth curve. In a small number of cases 50 aliquots were used (Stokes, 1994).

Optical stimulation was by an Ar$^+$ laser (λ = 514 nm) as described elsewhere (e.g., Smith et al. 1990), operated at a power level of 125 mW.cm^{-2}. OSL of UV-violet wavelengths was detected using an EMI 9635Q photomultiplier; other wavelengths being blocked by the presence of Corning 7-51 and Schott BG-39 glass filters (Stokes, 1992).

Prior to dosing and pre-heating, aliquots were exposed to the laser for a brief (< 20 mJ) period to facilitate normalisation using the so-called 'natural normalisation method'. Samples were then subjected to varying amounts of beta (^{90}Sr/^{90}Y) radiation and pre-heated either using a 160°C, 16 hour or 220°C, 5 minute procedure. All samples were stored for a period of at least 24 hours prior to OSL measurement.

The resulting additive dose measurements had the OSL collected late in the laser exposures subtracted as a background (c.f., Aitken and Xie, 1992), and palaeodoses were extrapolated using either a linear least squares or saturating exponential fitting algorithm. The saturating exponential fitting algorithm was provided by B.W. Smith (details in

Smith, 1983), and is known to produce somewhat more conservative errors on estimated palaeodoses than many other algorithms currently in use elsewhere.

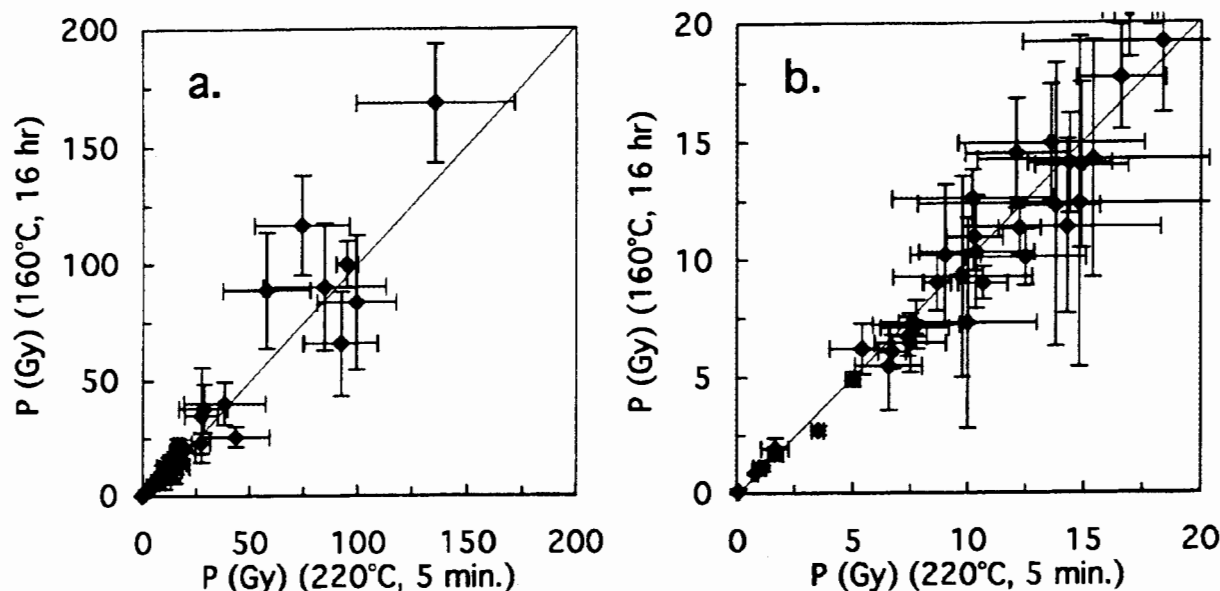The palaeodoses calculated for each pre-heat are plotted in Figure 1.

Figure 1
     Comparison of palaeodoses (in Gy) generated using 160°C, 16 hour and 220°C, 5 minute pre-heat treatments. a. all data, b. data plotted for a restricted range of palaeodoses (0 - 20 Gy). Errors plotted are one sigma

Summary
Palaeodose estimates are found to be in close agreement independent of which pre-heat treatment is employed. This appears to be the case over an age range from a few hundred to over a hundred thousand years. Average ratios of the palaeodoses generated for each of the pre-heats were calculated for all samples. The unweighted mean ratio (220°C, 5 minute/160°C, 16 hour) was 1.02±0.20, whilst the mean ratio weighted inversely proportionally to the scatter in each palaeodose assessment yielded a value of 0.95±0.14. Standard errors on the mean ratios are estimated at 0.03 and 0.02 respectively.

It is inferred from this analysis that there is no consistent contrast in palaeodoses caused by using either a 160°C, 16 hour or 220°C, 5 minute pre-heat treatment. While the corpus of observations remains too small for a robust statisical analysis, there is no specific evidence to suggest any influence of geographical locality, depositional environment, quartz provenance or sample age.

Although Roberts et al. (1993, 1994b) describe contrasts in palaeodoses caused by pre-heating for a small number of high sensitivity samples, it does not appear to be the norm in most quartz varieties. The cause of the apparent contrasts noted by Robert et al. (1994b) remains to be established.

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Rewievers
G. Duller and V. Mejdahl.

Pre-heats, Palaeodoses and Paradigms in the Optical Dating of Quartz: Some Comments on Roberts et al. (1993) 'Cautions on the use of Extended Duration Pre-heats in the Optical Dating of Quartz'

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In a recent issue of Ancient TL, Roberts et al. (1993) described a series of experiments on the OSL of quartz extracted from an archaeological sediment context in Australia. Their discussion ultimately indicated that for their sample, long duration, relatively low temperature (c. <200°C) pre-heats result in palaeodose underestimation. I question their assertion that differences between the OSL growth curves presented are attributable to dose-dependent sensitivity changes, and their implication that palaeodoses generated using an extended duration pre-heat will consistently be in error.

As a result of their study, I have embarked on my own intercomparison of long versus short duration pre-heats on 45 samples. These data are presented in a companion paper (Stokes, 1996). My investigation indicates that there is no tendency for one or other of the pre-heats to give a systematic over or under estimation. Further to these new data, I additionally think that the following points relating to Roberts et al (1993) should be considered in the context of their claims.

a. Despite their suggestion, the two pre-heat procedures are not kinetically equivalent. If, as Roberts et al. (1993) acknowledge, the bulk of the OSL in quartz is derived from the 325°C TL peak would it not be appropriate to predict the extent of thermal depletion from E and s values of that trap? Instead they arbitrarily assign an E value of 1.61 eV. Wintle (1975) calculated a depth of 1.69 eV for the 325°C trap. 160°C, 16 hour and 220°C, 5 minute pre-heats would therefore be expected to reduce trapped charge populations by 12.5 and 16.1% respectively. When considered in isolation, erosion of the 325°C trap should be greater for the 220°C pre-heat than for the 160°C, treatment and not equal.

As a test for the occurrence of the post pre-heat 'OSL excess' such as that observed by Roberts et al. (1993), I have measured the initial OSL of 28 samples, both prior to and following pre-heating (23 of which were administered both pre-heats). This ratio is plotted in Figure 1. Of the 23 comparable samples, 8 exhibited a post 160°C pre-heat OSL which was in excess of that following the 220°C pre-heat, all others exhibited greater intensities following the 220°C procedure. While a minority (c. 25%) of samples indicate higher OSL intensities following pre-heating, the bulk of the samples indicate nett depletion, in some cases by up to 60%. Clearly, the pattern of trapped charge re-adjustment caused by pre-heating is considerably more variable and complicated than inferred by Roberts et al. (1993), and their data on a single sample is probably misleading. The presence of non-quartz microinclusions in some samples may further complicate the picture.

b. Given the remarkably high degree of precision which they claim, it must be asked why their suggestion of a 15% sensitisation effect is not observable in the TL data presented in their Figures 1 and 2. The influence of a clearly subordinate 375°C peak is not considered to be a satisfactory explanation, particularly given that they acknowledge that the mean TL intensities for the pre-heated aliquots are indistinguishable, yet still use these data to infer sensitisation.

c. In relying on the 110°C TL peak for evidence of dose-dependent OSL sensitivity change the authors assume (as did Stoneham and Stokes, 1991) that the changes are synchronous. Further investigations (e.g., Jungner and Botter-Jensen, 1994; Stokes, 1994) clearly showed this not to be the case, and that

OSL sensitivity changes in quartz are triggered by the bleaching to which aliquots are subjected subsequent to additive dose dating measurements. Of course, although the samples on which I have worked included one from NW Australia (OX$_{OD}$778M8), the Roberts et al. sample may have different characteristics.
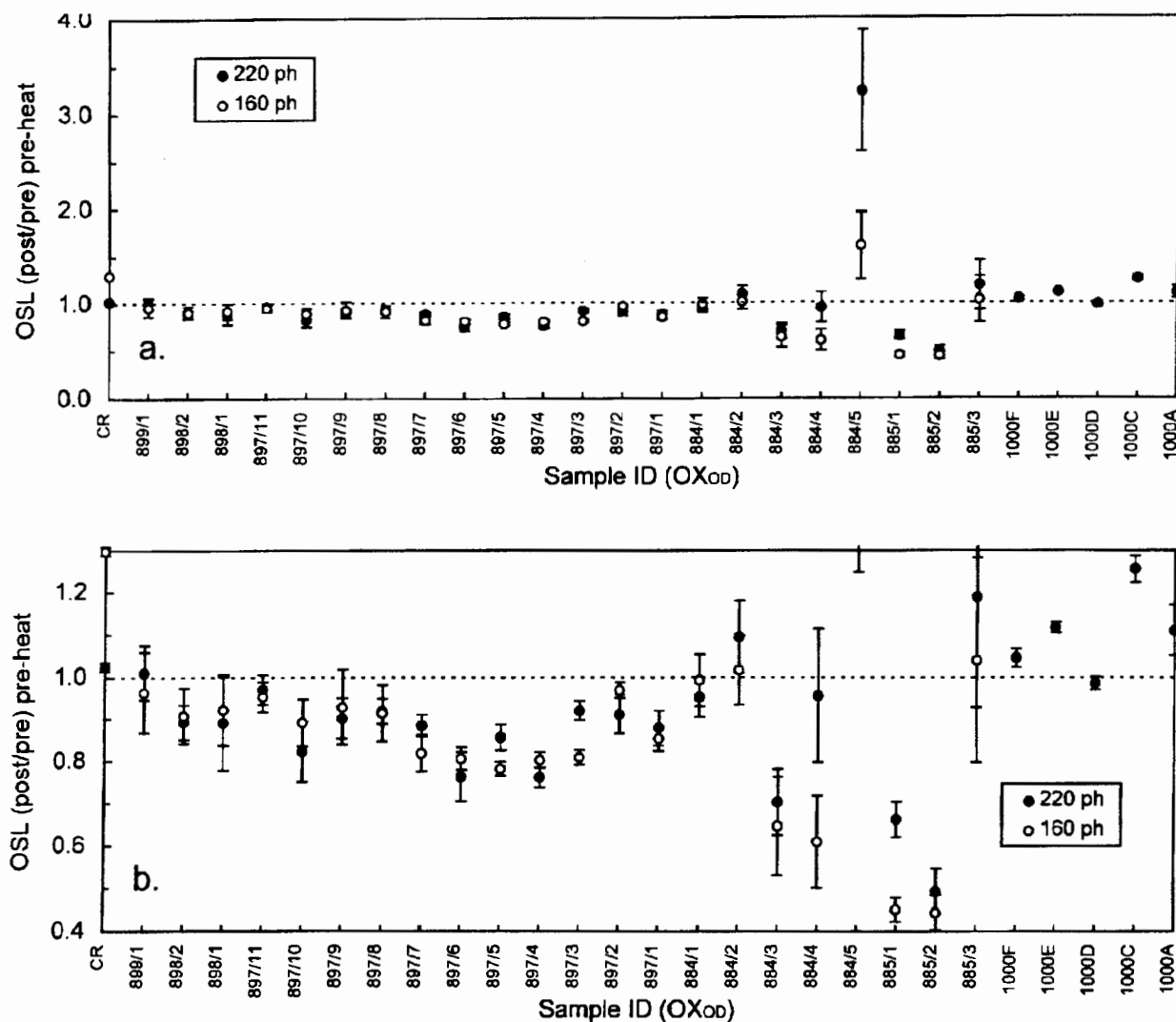

Figure 1.
        Ratio of the natural OSL intensity before to that following 220°C, 5 minute (filled symbols) or 160°C, 16 hour (open symbols) pre-heat (ph) treatments for a selection of 28 samples (CR = chaperon rouge). a. All data plotted; b. Data plotted for a restricted range of y values.

d. In contrast to other workers (e.g., Smith and Rhodes, 1994; B.W. Smith, pers comm.), Roberts et al. dismiss thermal charge transfer as a significant process. While the first two lines of argument which they use are marginal to the discussion at best, their third (and 'most direct') line of evidence is somewhat problematic when it is considered in the context of other data presented in the paper. Despite subsequently indicating that regenerative procedures yield palaeodose estimates similar to those generated using additive dose methods, the data in their Table 2 indicates a 91.8/11.2 fold increase in sensitivity upon bleaching. Such a bleach-induced sensitisation would result in a regenerated palaeodose estimate somewhat closer to 0.12 Gy than the 23.5 Gy which they quote. Even if the numbers which they present were sensible, the experimental strategy is neither direct nor compelling evidence to negate charge transfer.

e. Other researchers have documented that dose-dependent sensitivity changes can occur for quartz

(and other minerals) whether the 160°C, 16 hour or the 220°C, 5 minute pre-heat treatments are used (e.g, Rhodes, 1990; Duller, 1992; Stoneham and Stokes, 1991; Jungner and Botter-Jensen, 1994; Stokes, 1994). All these previous studies have quantified the degree of dose dependent sensitivity change by comparing ratios (after/before dating) of normalisation factors as a function of added dose. While this does not give a direct insight into the timing of the sensitivity changes, it does provide a means of establishing the occurrence and rate of sensitivity change as a function of added dose. If Roberts et al. wish to suggest that dose dependent sensitivity change is responsible for the apparent effects which they describe then they should at least provide the reader with a measure of its extent and demonstrate its occurrence only for the extended duration pre-heats.
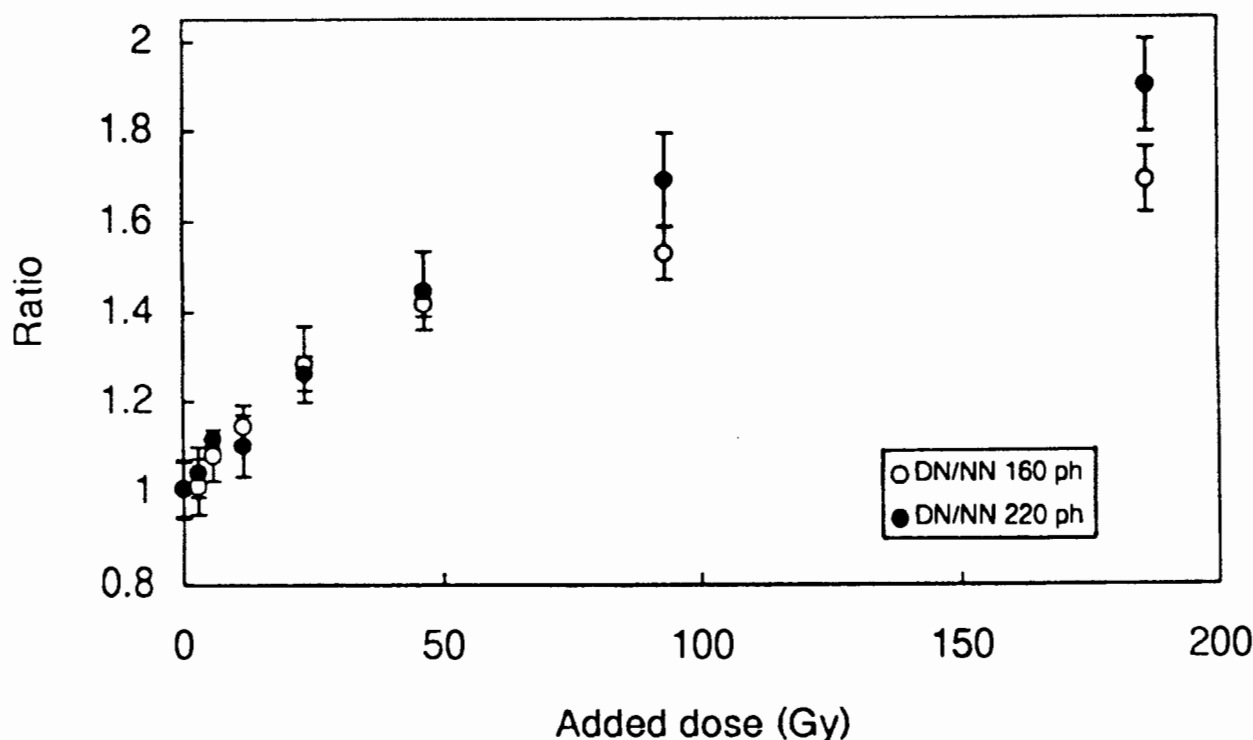

Figure 2

$$Ratio\ of \left(\frac{dose\quad normalisation\quad (DN)}{natural\quad normalisation\quad (NN)} \right) versus\ added\ dose\ for\ a\ dune\ sample\ from$$

Chaperon Rouge, Morroco (CR in Figure 1). Filled symbol - 220°C, 5 minute pre-heat, open symbol - 160°C, 16 hour pre-heat. Each data point plotted is an average of 8 aliquots (scaled to unity for average natural aliquot intensity); error bars - 1 standard deviation. When a linear least squares fit is applied to the data in the additive dose range 0-45 Gy, dose dependent sensitivity changes of 0.95 and 0.92 %.Gy^{-1} are estimated for the 220°C, 5 minute and 160°C, 16 hour pre-heats respectively. Aspects of the behaviour of this sample have also been described by Rhodes (1990), Roberts et al. (1994) and Smith and Rhodes (1994).

I have generated such data for one of the samples which they subesquently describe (Roberts et al., 1994). The ratio of normalisation factors measured before and after dosing, pre-heating and laser exposure, plotted as function of added dose is provided in Figure 2. Both sample treatment procedures clearly indicate dose dependent sensitivity changes of the order of 1 %.Gy^{-1} (when measured over a small initial range of added dose). In both cases, at higher doses the degree of sensitisation reduces, as also noted by Stoneham and Stokes (1991).

Clarification of the points raised above would facilitate a more reasoned assessment of the information presented in Roberts et al. (1993). While they present important data which may indicate that palaeodose estimates may partly be controlled by the selection of pre-heat procedures, at least for the sample which they describe and for five subsequent samples (Roberts et al., 1994), to reach

the unsubstantiated assertion that it is caused by dose-dependent sensitivity changes is most unhelpful in the collective long term goal of developing robust procedures for routine use of the optical dating method as a Late Quaternary geochronometer.

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A simple staining method for K-feldspars before sample preparation for IR-OSL dating

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Introduction

Potassium-feldspar (K-Fs) grains are favored for dating the last insolation of sand deposits when IR-OSL (optically stimulated luminescence using near-infrared light) is used (Aitken, 1994; Berger, 1996; Wintle, 1994). For this purpose, the K-Fs grains are floated in a heavy liquid (e.g., sodium polytungstate, available from Geoliquids Inc., 15 E. Palatine Rd., Suite 109, Prospect Heights, IL 60070, USA) at a relative density of 2.58 g/cm^3. In eolian deposits such as sand dunes, the concentration of K-Fs is often very low (<1-5%), because feldspars are less resistant to weathering, comminution and dissolution than is quartz. If the K-Fs concentration is low, one can choose to date only the quartz fraction (e.g., Rendell et al., 1994), using either TL or G-OSL (OSL using green stimulation). However, if one prefers K-Fs grains for their several advantageous luminescence properties (compared to quartz), then K-Fs can be separated from quartz with a little patience.

We have found that a quick visual means of estimating quantitatively the concentration and physical character of K-Fs grains in dune sand is useful for planning efficient subsequent steps in sample preparation. X-ray diffraction is insensitive to detection of K-Fs concentrations below ~5%. The optical-microscopy Becke-line test (e.g., Stoiber and Morse, 1981) can be straightforward, but "not all grains give rapid, unequivocal answers" (Stoiber and Morse, op. cit.). Thus we have found it's use to be ambiguous and tedious for our samples, especially when searching for a minor component such as K-Fs. Other analytical procedures (e.g., scanning electron microscopy) require elaborate and expensive equipment for such low concentrations. Staining of the K-Fs grains followed by moderate-power optical microscopy offers an alternative, simple, visual, quantitative means for estimating concentration of K-Fs grains in sand dunes.

Staining techniques for geological purposes have been used for over a hundred years (e.g., Allman and Lawrence, 1972; Hutchison, 1974). Generally, certain dyes or reagents have the ability to color only a specific mineral, even in the presence of other similar minerals. Specifically, sodium cobaltinitrite (Gabriel and Cox, 1929) colors K-Fs lemon yellow but leaves quartz unaffected, and other feldspars generally uncolored. The disadvantage for IR-OSL sample preparation, in our view, of this and other published techniques for staining K-Fs (and other feldspars) is the relative effort they require. Either polished thin sections or (unpolished) grain mounts (using a heated cement) are required, to be exposed to controlled fumes of hydrofluoric (HF) acid and certain rinsing solutions in specially fabricated etching containers (e.g., Teflon), followed by drying and application of a staining reagent and other steps. We have employed a much easier and quicker procedure, which is a variant and simplification of the procedures compiled by Hutchison (1974).

Our procedure employs loose grains, and thus avoids the need to fabricate grain mounts and polished thin sections. It also obviates use of special containers, HF fumes, and sample heating. Grain-size separation is not necessary, but prior removal of small grains (e.g., <50-100 μm) is beneficial. Only a small fraction of the total sample is needed for staining. Following the usual removal of carbonates (with HCl acid) and organics (with H_2O_2), the general steps to be performed for staining are: removal of rust (iron) stains from the sample to facilitate laboratory staining and easy visual discrimination of stained from unstained grains; staining of the K-Fs grains; visual grain counting, using either plane-polarized transmitted light through a petrographic microscope, or unpolarized reflected light through a binocular low-power microscope. Use of plane-polarized light

can enhance (Allman and Lawrence, 1972) the target lemon-yellow color.

Removal of rust stains

Common yellowish rust stains hamper the further staining of samples, and later visual discrimination of artificially stained K-Fs grains. A saturated solution of oxalic acid dihydrate (a strong reducing agent) is added to a small amount of each sample to remove rust stains. This solution is best for removal of limonite stains. For haematitic deposits, a heated bath of concentrated hydrochloric (HCl) acid may be necessary as a substitute. Other, more elaborate, methods could be used (e.g., citrate-bicarbonate-dithionite or CBD). Our simple oxalic-acid procedure was successful in removing all but the most stubborn stains and individual (opaque) iron-mineral grains.

The oxalic acid solution is prepared, in quantity, on a hot plate in a fume hood. Solution temperature may be 50-80°C. To a chosen volume of distilled water in a beaker containing a magnetic stirring bar, add oxalic-acid powder slowly, permitting it to dissolve.

Remove the solution from heat and cool to room temperature. There should be a small amount of undissolved powder on the beaker's bottom when cool.

Add the solution to the sample in a volume ratio 2:1. Cover the beaker and place in an oven at 40-50°C overnight or a full day. The solution will change to a yellowish color. A bright yellow color indicates that several applications of the oxalic-acid solution are needed. Care is required to prevent reprecipitation of used (yellow) solution onto the sample. This can be prevented by not allowing the sample to cool or evaporate significantly before decanting. Decant the solution and thoroughly wash the sample in distilled water.

Staining of a cleaned sample

Efficient staining is accomplished on bulk grains using a saturated solution of sodium cobaltinitrite (NCN hereinafter, available from standard chemical suppliers), following wash-etching of the sample with concentrated (~49%) HF acid on a fast, hardened filter paper. HF leaches away silica and leaves an etch-residue containing K and other elements. NCN reacts with K to form a yellow precipitate of potassium cobaltinitrite. Unetched grains will not noticeably stain (Bailey and Stevens, 1960). Once stained, the samples should not be shaken or stirred too briskly prior to visual examination, so that none of the coating is removed.

In a fume hood, place a Teflon funnel into a support ring, with a Teflon beaker beneath. Use a fast, hardened (may be ashless) filter paper, such as Whatman 541. The saturated NCN solution is viscous, thus requiring a fast filter paper. Wet the paper with a few drops of distilled water. Add a small amount of cleaned sample. Add just enough HF to wet the sample and to produce just one drop on the lower end of the funnel. Let stand for 15 seconds. Rinse the sample with distilled water. Add a small amount of saturated NCN solution, allowing to stand for ~1 minute. Rinse the sample thoroughly with distilled water, remove the filter paper from the funnel and permit to air dry.

Visual grain counting

To a good first approximation, in the stained sample, K-Fs grains (excepting sanidine) will have a bright (lemon) yellow powdery coating, Na-Ca feldspars (plagioclases) will have a white powdery coating, and quartz will be unaffected. Increasing Na seems to reduce the effectiveness of NCN, and with sanidine grains, only pits and corners will take much of this stain. However, sanidine is unlikely to be a noticeable component in most sand dunes, occurring mainly in deposits derived directly from volcanic materials.

We have tested the above procedures with samples from three temperate-zone sand dunes. We had no specific mineralogical information about these dunes before. Grain counts were performed under plane-polarized light in a petrographic microscope. We classified the grains as "white" (plagioclase), "yellow" (K-Fs) and "total" (unstained, stained and opaque), and counted all grains in each of 4 quadrants of the field of view (moderate power) for 5 subsamples from each stained sample. The results are shown in Table 1.

Sample	Total grains	%Yellow (mean±S.E.) (n=20)	%White (mean±S.E) (n=20)
A	1577	0.62±0.20	5.48±0.70
B	1416	2.37±0.46	5.81±0.64
C	1309	3.38±0.43	4.72±0.51

Table 1 Grain-counting

These data demonstrate both the sensitivity of the method (sample A), and the ability to estimate the relative proportions of K-Fs and plagioclases. Even if half the total number of grains were counted, such data would likely still permit the deduction that sample A is problematical and will require special effort to produce sufficient K-Fs for all expected dating analyses. We note in passing that the statistical distributions of the populations of yellow and white grains were not all symmetrical nor normal.

Conclusions

We conclude that the simplified staining procedures described here can permit relatively low-effort, low-technology quantitative estimates of both K-Fs and plagioclase concentrations in sand dunes having relatively low (<5%) concentrations of K-Fs. Samples can be processed in batches. The resultant grain-count data can be useful in planning efforts toward separation of K-Fs for IR-OSL dating. For the same reason, the concomitant knowledge of plagioclase concentration may be useful if plagioclase grains will be used for IR-OSL dating.

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Reviewer
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Comments

This is a useful and simple method to determine the presence of IRSL-datable K-feldspar in dune sands. Where K-feldspar abundance is very low, a quantitative estimate of its abundance helps to predict the yield from heavy-liquid flotation. This method will probably not detect K-feldspar inclusions in quartz which are sometimes the last surviving, IRSL-datable component in mature sand (see Huntley, Hutton and Prescott, Geology, 21:1087-1090, 1993).

TL measurements of single grains from selected feldspar samples

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Abstract
The thermoluminescence (TL) properties of single feldspar grains from both sediments and mineral specimens have been measured using an imaging photon detector (IPD). The TL sensitivity of the grains to a known laboratory dose was found and a range of grain-to-grain sensitivities were observed. Analytical SEM and optical analysis were then used to attempt to explain the grain-to-grain behaviour in terms of the physical or optical properties of each grain. However, the range of TL sensitivities found could not be correlated with any of the physical or optical properties measured.

Introduction
Thermoluminescence (TL) and infrared stimulated luminescence (IRSL) from feldspars are increasingly common as methods of dating in quaternary science and consequently many studies have concentrated on the luminescence behaviour of bulk samples of feldspar grains (i.e., the total luminescence from many thousands of individual grains), for example, Duller (1992).

In contrast, little work has been published in which the TL behaviour of single feldspar grains has been investigated, Southgate (1985) made TL measurements on feldspar grains to find individual EDs, but no final results were presented. Grün et al. (1989) measured the TL from fifty feldspar grains and although the TL varied greatly from grain to grain, the mineralogical properties of these grains, as determined with an SEM, were broadly similar. A recent paper by Lamothe et al. (1994) reported measurements on 120 large (500-1000 m) feldspar grains with the natural IRSL from individual grains being measured. A large variability in IRSL intensities was found but the majority of the grains were not examined further.

In this paper, the second glow TL (the TL after a laboratory beta dose) was measured from single feldspar grains prepared from both sedimentary and specific mineral types. The TL was measured using an imaging photon detector (IPD) which is a position-sensitive photon counting device previously used to measure the single grain TL of both zircons (Smith et al.1991) and quartz grains (McFee and Tite, 1994).

Sample selection and measurement
The following sediments and geological sediments were selected for measurement:

Belcroute (a) : a modern beach sand from Belcroute, Jersey. The sediment is assumed to be completed bleached. (Lab ref : 760a1)

Belcroute (b) : an aeolian dune sand from Belcroute, Jersey. The ED of the sediment is around 220 Gy. (Lab ref: 760c1).

Le Gurp : a beach sand from Le Gurp, Atlantic coast of France. The ED (IRSL) is 238 (+57,-66) Gy (measured by the author). (Lab ref: 787a1).

Blassac : an aeolian sediment from Blassac, France. The ED (TL) is 65 (+6,-14) Gy (measured by the author). (Lab ref : 785a1).

Labradorite : a mineral specimen from Madagascar.

Orthoclase : a mineral specimen from Norway.

Microcline : a mineral specimen (Spooner 1993). (Lab ref : Z5).

The labradorite and orthoclase mineral specimens were purchased from Gregory, Botley and Lloyd Ltd. (London). The chemical composition of each mineral specimen was checked using an X-ray Energy Dispersive Spectrometer (EDS) attached to an SEM and was found to be consistent with the stated mineral type.

The sediments were prepared under subdued red light and were first sieved to obtain the 180-220 μm grain fraction. Grains were then left in dilute HCl for several hours to facilitate the removal of any iron oxide coating on the grains. Belcroute (a) and (b), and Le Gurp were density separated using sodium polytungstate, the grains with densities

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below 2.58gcm^{-3} being selected. The sediment from Blassac was density separated to remove only the heavy minerals   (>2.70gcm^{-3}), i.e., this sediment contained both quartz and feldspars. The sediments were then etched for 40 minutes in 10% HF to remove the outer alpha affected layer (Mejdahl, 1985) and were again washed with HCl to remove any fluorides formed.

Two of the mineral samples (labradorite and orthoclase) were supplied as large crystals. These were gently crushed and the 180-220 m grain size selected by sieving. These grains were also left in dilute HCl for several hours to mitigate any possible spurious TL generated as a result of the crushing process (Aitken 1985, p192). The microcline specimen was supplied as 75-125 m size grains and received no further treatment.

Individual grains were picked up on a small needle in dim red light using an image intensifier attached to a binocular microscope, and were placed

in a known pattern onto a rhodium-plated copper planchette.

Grains from all the samples were heated to 450 C at 5 /s to measure the natural TL, these results are summarised elsewhere (McFee and Tite, in preparation).

All the samples were then irradiated on-plate with a ^{90}Sr - ^{90}Y beta source. The following doses were given, which were chosen to allow a reasonably large amount of TL to be obtained from each grain without causing a pile-up of counts within the IPD:
Belcroute (a) and (b), Le Gurp, Blassac and orthoclase each received 30 Gy, labradorite received 60 Gy and microcline 120 Gy.

All measurements were made with quartz optics together with a 1mm BG39 filter, the effective wavelength range being 330-600 nm.

	N	min value	Q1	median	average	Q3	max value
Belcroute (a)	253	217	729	1035	2398	1708	41892
Belcroute (b)	264	26	356	659	1717	1291	33261
Le Gurp	241	852	3521	6623	9312	10936	63506
Blassac	107	126	496	1134	3424	3395	38960
Labradorite	317	182	633	850	1061	1276	4677
Orthoclase	324	232	923	1482	2113	2606	15681
Microcline	206	51	94	149	263	287	4663

Table 1
Descriptive statistics for the sediment and mineral samples. N is the number of grains, min value shows the minimum TL measured, Q1 is the value of the first quartile, median shows the median TL value, Q3 shows the value of the third quartile and max value shows the maximum recorded TL value for each sample.

Results and discussions

The chemical composition of grains from the Blassac sample, which consisted of both quartz and feldspar grains, was investigated using the EDS. The results from grains found to be quartz were excluded from this study.

Around 75% of grains from each sample were sufficiently bright to be observable on the IPD. Table 1 shows descriptive statistics for all the samples and figure 1 shows box and whisker plots for the second glow TL of all the samples. Figure 2

shows second glow TL frequency histograms for the grains from the Blassac sediment and the orthoclase sample. Each count frequency bar shows the number of grains in the range of TL intensity from the value shown to the next value above.

The second glow variations within each sample (figure 1) are very similar, irrespective of the type of sample (sediment or mineral) measured. In addition, outlying grains can be seen in each sample (figure 2). These outliers are usually around 10 to 100 times brighter than the median second glow TL.

When the intensity of such grains is considered in terms of the total light sum from all the grains, such outlying grains contribute about 30-40% of the total light sum. For example, the total light sum from the Blassac sediment was 360,000 counts and the contribution from the brightest 5% (6 grains) was 145,000 counts. Thus, the brightest 5% of the Blassac grains contribute 40% of the total light sum.

The effect of the outlying grains can also be clearly seen in table 1, which includes the median and average TL values found for each sample. In all cases, the average value is much larger than the median value, for three of the samples (Belcroute (a), Belcroute (b) and Blassac), the average values are larger than the values found at the third quartile point (Q3).
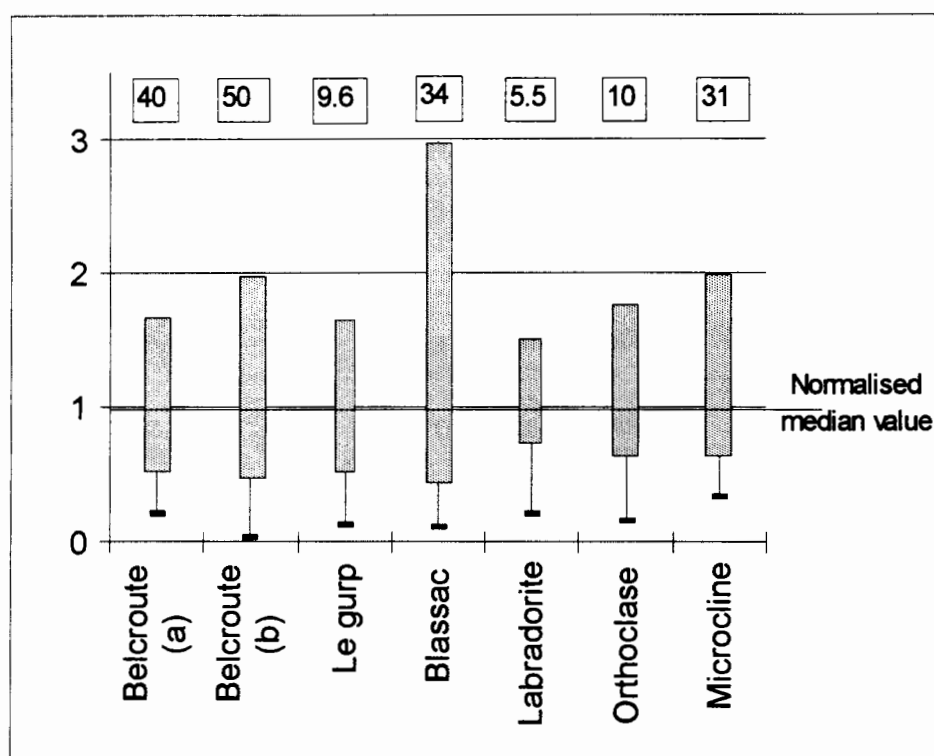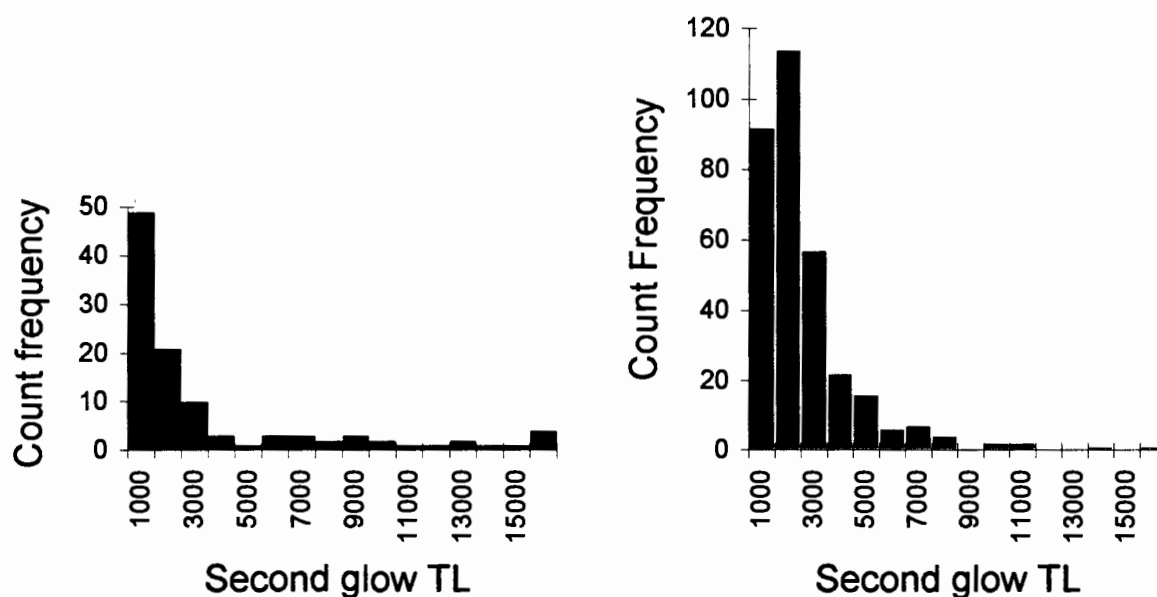

Figure 1

Box-and-whisker plots for the sediment and mineral specimens. The maxiumum TL values are shown only at the top of the graph to allow a reasonable scale. The TL from each sample has been normalised so the median TL from each sample has the value 1.

Therefore, the outlying grains do not "dominate" the light sum in the sense that only one or two grains do not contribute almost all of the observed light. However, the contribution of the brightest grains in each sample will be large enough to lead to substantial variations in the bulk TL corresponding to variations in the numbers of these grains present (McFee, 1995).

The TL results found for single feldspar grains are similar to those found previously for single quartz grains by McFee and Tite (1994), with a small number of grains from each sample having a high TL sensitivity. These grains are outliers from the main second glow TL populations with high

intrinsic sensitivity to radiation. Such sensitivity will, at a fundamental level be determined by the concentration of impurities and defects (i.e. trapping/luminescence centre concentration within the grains). However, at a macroscopic level, this high intrinsic TL sensitivity may also be associated with physical characteristics of grains such as:

(1)   grain size
(2)   physical orientation of the grain or grain shape
(3)   degree of weathering
(4)   grain mineralogy
(5)   grain transparency

(a) The second glow TL for the Blassac sediment sample

(b) The second glow TL for the orthoclase sample.

Figure 2
The second glow TL for two samples described in the text. Each count frequency bar shows the number of grains in the range of TL intensity value shown to the next value above

To attempt to identify which of the above explanations were most probable all the grains from which TL had previously been measured were viewed with a low power (x20) binocular microscope in white light and the approximate size of each grain was noted. All the grains measured from the Blassac sediment were also examined at high power (x100 - x500) in both transmitted and reflected white light. Each grain in the Blassac sediment was assigned four indices which provided a measure of angularity, sphericity, transparency (frosty/clear) and the presence or absence of possible inclusions or significant iron oxide staining (it was difficult to differentiate between these two properties using the binocular microscope, even at high powers). The distribution of grain sizes observed was not sufficient to explain either the distribution around the median TL for the majority of the grains nor the presence of outlying grains with very high TL values. For example, the maximum second glow TL for the Belcroute (a) sediment is some forty times larger than the median values which, to be explained solely in terms of grain size, would require a grain of 700 m diameter. Such a grain was not observed. However, it was apparent that, due to the crushing,

the labradorite and orthoclase grains exhibited a great range of shapes, many of them not being even approximately spherical and it was difficult to estimate their true volume. For the labradorite and orthoclase grains the maximum observed second glow TL could be explained in terms of a grain volume equivalent to a spherical grain only some 300 m in diameter. Thus, the effect of grain size on the outlying second glow TL values for the labradorite or orthoclase samples could not be ruled out.

A statistical comparison between the sensitivities of grains from the Blassac sediment and their morphological or optical indices found that the range of TL behaviour amongst these grains could not be explained in terms of physical orientation or grain transparency. As the Blassac grains had a very similar appearance to the grains from all other samples, both sediments and mineral types, it is expected that neither physical orientation nor grain transparency could explain the range of second glow TL measurements observed for the other samples.

A random sub-sample of grains from the Belcroute sediments, as well as all the grains from the Blassac sediment were examined using an

analytical SEM. The grains were encapsulated into resin blocks, which were then polished. The grains were examined with a Cameca SU30 SEMPROBE in both back-scattered and secondary emission mode, with an accelerating voltage of 15 kV and an operating current of 10nA. The grains were viewed in backscattered mode, the shape of the grain and the effect of weathering being noted. Regions of the grain which consisted of different feldspathic minerals such as perthitic intergrowths could be easily identified as areas of differing contrast on the back-scattered image display of the grains. It should be noted that, because of the small penetration depth of the incident electrons, only a thin surface layer (3-5 m) could be examined.
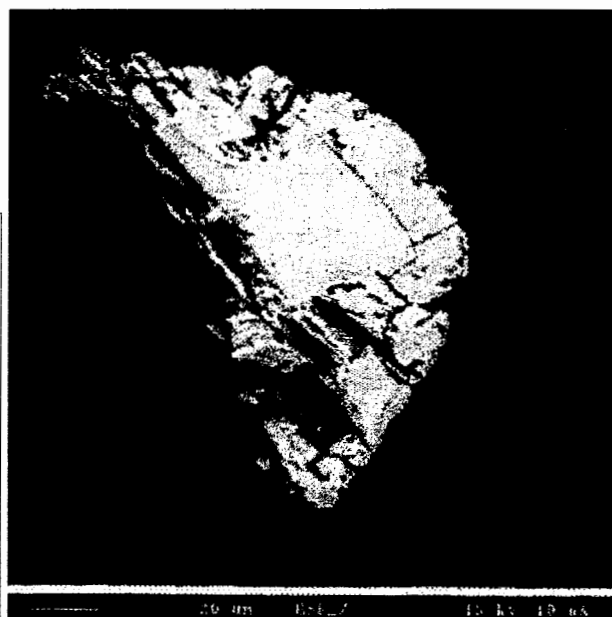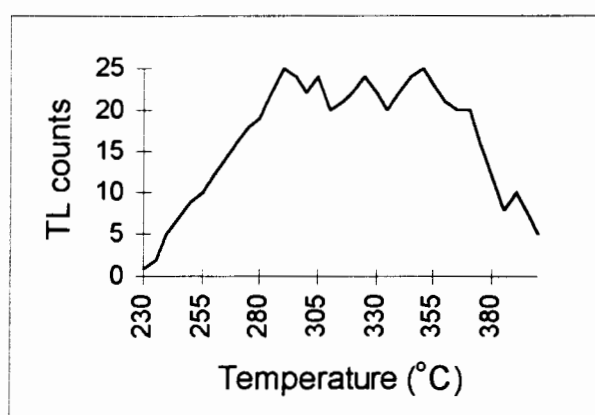

Figure 3

The backscatterd electron image of a grain of feldspar from the Belcroute (b) sediment, along with the second glow TL obtained from the grain.

It was found that the 10% HF etch had removed much more of the outer surface of the feldspar grains that had been previously expected from consideration of the TL literature. This loss of much of the outer surface meant that it was not possible to see any unambiguous signs of weathering. In addition, it was seen that one or two grains had been heavily attacked by the HF etch and were severely degraded with loss of much of the mineral structure. For example, figures 3 and 4 show the backscattered electron images from two grains from the Belcroute (b) sediment, along with the second glow curves associated with each grain. Figure 3 shows a feldspar grain which typifies most of the grains examined with the SEM in that it is intact but signs of attack along the cleavage planes are clearly visible. Figure 4 shows a second feldspar grain which shows signs of an intense attack by HF such that most of this grain has been dissolved away. The darker areas of this second grain were investigated using the EDS and were found to be reprecipitated silica. It is likely that, as a result of the HF etch, most of the K, Ca and Na ions in the feldspar matrix migrated into the HF solution leaving the silica to be reprecipitated. Small areas of surviving K-feldspar are visible in the photograph as regions of brighter contrast. However, this grain had virtually no second glow TL.

In summary, it was found that the effect of the "routine" HF etch was to completely remove all traces of weathering from the grains, and in a few cases the grain was almost destroyed by the HF etch. At the start of these studies such a severe HF attack was not expected but this effect has recently been confirmed by other authors (eg Duller 1994) and consequently any etching of feldspars has been abandoned. Thus, because of the HF etching, it is not possible to identify the effect of weathering on the TL of the majority of the grains measured in this paper. Nevertheless, grains which were severely attacked by the HF etch were found to have an extremely low TL, both natural and second glow (for example, figure 4). Therefore, it is likely that grains which had undergone severe chemical weathering

would tend to show very low TL. These grains would not contribute any significant TL to the overall light sum when conventional TL measurements are made using bulk samples of grains.

The possible effect of grain mineralogy on the corresponding TL was also investigated on the grains from the Blassac sample. Previous measurements on bulk feldspathic samples, containing several thousand grains, have suggested that there is a large variation in TL sensitivity between different feldspar types. For example, Spooner (1993) found a difference of up to six orders of magnitude in the second glow TL sensitivities. A factor of 10 times was observed in this study between the median TL intensities of grains from the microcline and orthoclase geological samples. Thus, a range of TL sensitivity may be expected within a sediment sample containing different feldspar types such as will be present in the Blassac sample.
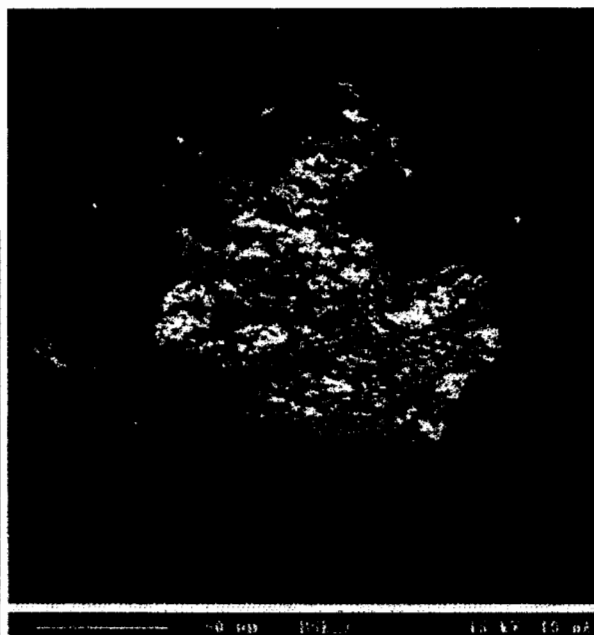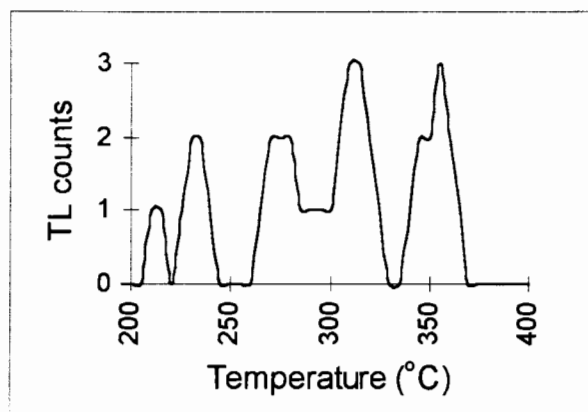

Figure 4
        The backscattered electron image of a heavily degraded feldspar grain from the Belcroute (b) sample, along with the second glow TL obtained from the grain.

All the grains from the Blassac sediment were examined with the EDS and the percentage oxides present of silicon, aluminium, potassium, calcium , sodium, manganese, magnesium, iron, phosphorous and titanium were determined. The elemental concentrations obtained by the EDS measurements were converted into the percentage of the three end members of the feldspar ternary diagrams using the procedures outlined in Deer, Howie and Zussman (1983, p515), that is, orthoclase (high potassium), anorthite (high calcium) and albite (high sodium). It should be noted that to completely characterise each grain it would also be necessary to obtain information on the crystalline phases present, using X-ray diffraction. Although this data could not be obtained, nevertheless, in a general study of single grain luminescence the information obtained from the chemical composition alone is sufficient to allow the grains to be assigned approximate positions on the ternary diagram.

Figure 5 shows the feldspar composition for grains from the Blassac sediment plotted against the corresponding second glow TL for each grain. The sample contains a range of feldspar types within the single population of grains measured. However, it is difficult to see any corresponding general trend in the TL, but instead there is a wide variation in TL intensity for feldspar grains independent of chemical composition. This suggests that, within any one sample, mineral type by itself may not be the dominant cause of variations in TL sensitivity.

Conclusions
        In summary, the bulk TL sensitivity varies on a sample-to-sample basis, as expected. However, the second glow TL variations within each sample are very similar, irrespective of the type of sample

(sediment or mineral type) measured. Also, any outliers observed are not several orders of magnitude brighter than the majority of grains in each sample but are usually only some twenty to thirty times brighter than the median second glow TL in each case. The outliers within the labradorite and orthoclase samples can be explained simply in terms of grain size effects. However, the outlying grains from the other samples cannot be so explained, nor can they be explained in terms of grain-to-grain differences such as physical orientation or grain transparency.
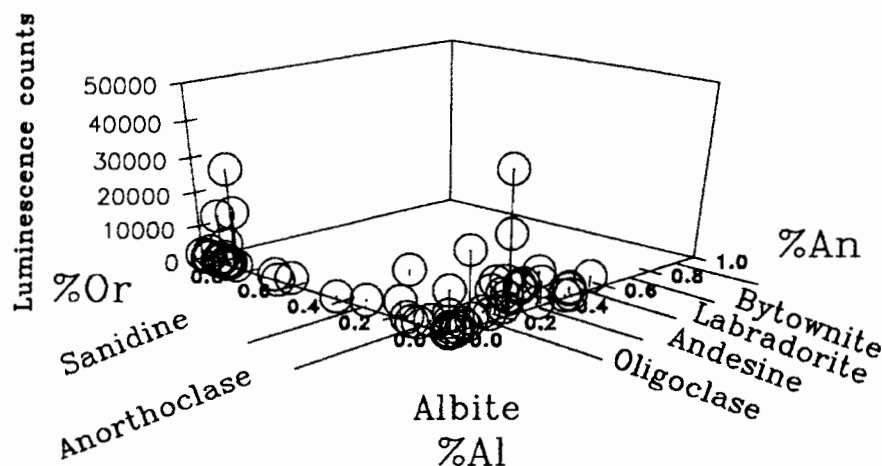

Figure 5
The second glow TL for single mineral grains from the Blassac sample plotted against the corresponding mineral composition for each grain.

Investigations of grain mineralogy found that, whilst there was a variation in both the TL sensitivities and the grain mineralogy within a sediment sample, variations in the TL could not be correlated with differences in mineralogy of the corresponding grains. The effect of weathering on the TL sensitivities of the sediment samples could not be investigated because of the unexpected strength of the HF etch. Nevertheless, it was possible to see that grains which had been significantly altered chemically by the etch, and which could correspond to heavily weathered grains in nature, had negligible TL.

Therefore, the results presented in this paper suggest two important findings about the second glow TL behaviour of single feldspar grains. Firstly, grains having a high second glow TL do exist within most samples and these grains are bright enough to contribute almost half of the total light sum. Secondly, the grain-to-grain variation within a sample which at a fundamental level will be determined by the concentrations of traps and luminescence centres present, does not appear to be related in a simple manner to observable physical parameters such as grain shape or the mineralogy of each grain.

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Uranium and thorium in the constituents of fossil teeth

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Abstract
In the course of ESR dating studies we have analysed the constituents of 67 teeth from 12 archaeological sites in Australia, China and South Africa for uranium and thorium by ICP-MS. We find that there is no relationship between the uranium and thorium concentrations in either dentine or enamel and that there is also no correlation between the thorium concentrations in adjacent dentine and enamel layers. There is some relationship between the uranium concentrations of adjacent enamel and dentine layers, the U concentration in dentine being about 20 times higher than in enamel. There is also hardly any connection between uranium and thorium concentrations and time which precludes any predictions of the mode of uranium and thorium accumulation.

Introduction
For recent ESR dating studies, we have switched from neutron activation analysis to inductively coupled plasma mass spectrometry, ICP-MS, for the analysis of elemental concentrations in teeth. Due to the small sample size, in the range of a few hundred mg, it is difficult to determine Th concentrations, therefore, it was usually assumed that the constituents of teeth are basically Th free and only uranium was determined. The application of ICP-MS allows the scanning of a variety of elements in one run with very high sensitivity (better than 1 ppb) on small samples (< 100 mg). In the context of ESR and U-series dating, it is not only important to know the concentrations of radioactive elements in the sample and its surroundings but also whether and how radioactive elements, particularly uranium, migrate into the samples (see Grün 1989, Millard 1993, McDermott et al. 1993). It is well known that fossil bones and teeth have significantly higher concentrations of uranium than recent specimens and this effect has been used for crude dating purposes (Oakley 1980).
The geochemistry of uranium and thorium has been described in detail in Ivanovich & Harmon (1992). Basically, U^{6+} is water soluble, thus mobile, whereas Th is water insoluble and is quantitatively absorbed by particles. Therefore, percolating water contains traces of uranium but is virtually free of thorium. When bones, teeth and other organic material are buried, the decomposition of the organic constituents produces a reducing environment in which U^{6+} is reduced to the insoluble form of U^{4+}. This leads to an enrichment of uranium in the initial stages of the fossilisation of bone material. Furthermore, U^{4+} and U^{6+} can be incorporated into apatite (replacing Ca^{2+}), which is the main mineral constituent of bones and teeth.
Both ESR and U-series dating results are strongly affected by mobilisation of uranium (McKinney 1991, Grün et al. 1987). In order to address the problem of uranium uptake for ESR dating, two models are used for calculating ESR age estimates (Ikeya 1982):

1) Early U-uptake: the measured uranium concentration was accumulated by the specimens shortly after burial;
2) Linear U-uptake: the uranium was continuously accumulated over time.

It is clear that both models can only approximate the real U-uptake, as this will strongly depend on the geochemistry of the sediments, water availability, temperature, bacterial activity etc. One would expect, however, if linear uptake is the closer approximation to a general U-uptake history, that older samples have generally higher U-concentrations than younger samples.

Grün et al. (1988) proposed to combine ESR and U-series analyses which then allows the simultaneous estimation of the mode of uranium uptake and the age of the sample. The model was successfully tested with a variety of samples from Israeli archaeological sites (McDermott et al. 1993). The open system modelling (Grün & McDermott 1994) revealed that most of these samples accumulated the uranium a short time after burial, however, some samples showed late uranium uptake. The main conclusion of this study was, that the uranium uptake was not predictable.

ICP-MS Analysis

The elemental analysis was performed on a Fisons VG PlasmaQuad. Homogenised enamel (50 mg) and dentine (30 mg) samples were dissolved in 4ml 6N nitric acid and 3 drops of concentrated perchloric acid and refluxed in capped teflon bottles on a hotplate at 150°C for at least 4 hours. The solutions/suspensions were then evaporated down to a residual pellet, resuspended and dissolved in 2% nitric acid and refluxed at 120°C for at least 24 hours. Whist the enamel samples are readily soluble using the above procedure, dentine samples vary in their ease of solubility due to the presence of metallic oxides, organics and other compounds. Partly insoluble dentine samples were treated with 1 to 2 ml 4N hydrochloric acid when approximately half of the 6N nitric acid had evaporated. In many samples this treatment results in complete dissolution, in others, the addition of several drops of perchloric acid and/or hydrogen peroxide, refluxing for several hours at 150°C alternating with 15-20 minute ultrasonic bath sessions, completes dissolution. The inclusion of hydrochloric acid requires evaporation to a pellet, resuspension/dissolution in 6N nitric acid, evaporation to a pellet and resuspension/dissolution in 2% nitric acid. Milli-Q water is used throughout and all reagents apart from hydrogen peroxide are the refined products of sub-boiling distillation. The 2% solution is transferred, along with several 2% nitric acid rinsings of the teflon bottle, to a 125 ml polypropylene bottle to which is also added a spike (internal standard) to give a final concentration in 50g (taken to final weight with 2% nitric acid) of approximately 1ppb ^{233}U and 7ppb ^{115}In. The sample is now ready for ICP-MS. Blank preparations, for each batch of samples put through the dissolution procedure are prepared in the same manner as for assay samples. Additional blanks are prepared to check polypropylene bottles, ICP-MS uptake tubes, teflon bottles and fresh reagents.

The judgement of whether a sample is in complete solution is based on visual appraisal; it is therefore important to consider that the sample may not be in complete dissolution. To address this issue repeats can be performed and UV-VIS spectrum analysis can be made. Only a few dentine samples have resisted the dissolution procedures as described above, in these cases the dissolution procedure has been repeated on a new sub-sample and if insolubility persists, the suspension is centrifuged and the supernatant removed for analysis along with the supernatant from several 2% nitric acid washings of the pellet. The latter analysis is unsatisfactory due to the unknown chemical composition of the pellet.
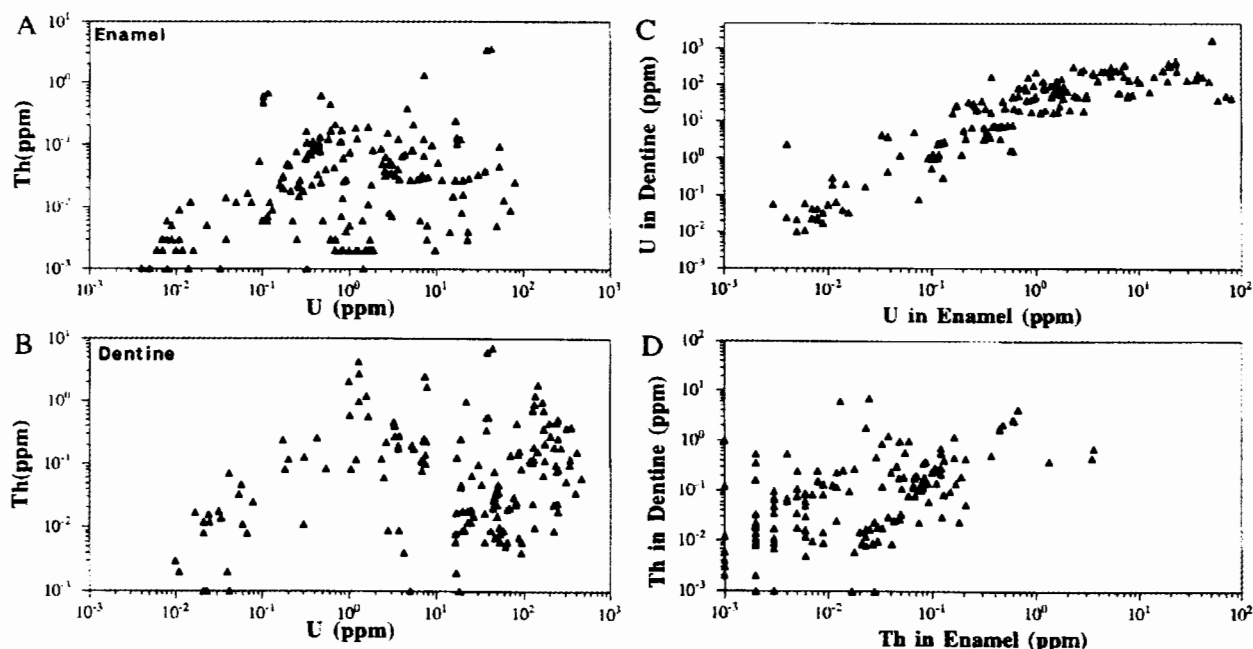

Figure 1

Plot of Th versus U concentrations in enamel (1A) and dentine (1B) and plot of U(1C) and Th (1D) concentrations in dentine versus the respective concentrations in enamel. Figures 1A, 1B and 1D show that the measured Th concentrations show hardly any relationship to the other parameters. The U concentration in dentine is approximately 20 times higher than the U concentration in an adjacent enamel layer (r = 0.89).

Dissolution using a high performance microwave digestion unit will be considered for future dentine samples as the higher temperatures attainable are suitable for dissolving refractory minerals.

The random errors for fully prepared solutions which have been analysed in different session are < 2% for Th concentrations of between 22 and 34,149 ppb and for U concentrations of between 256 and 7,325 ppb
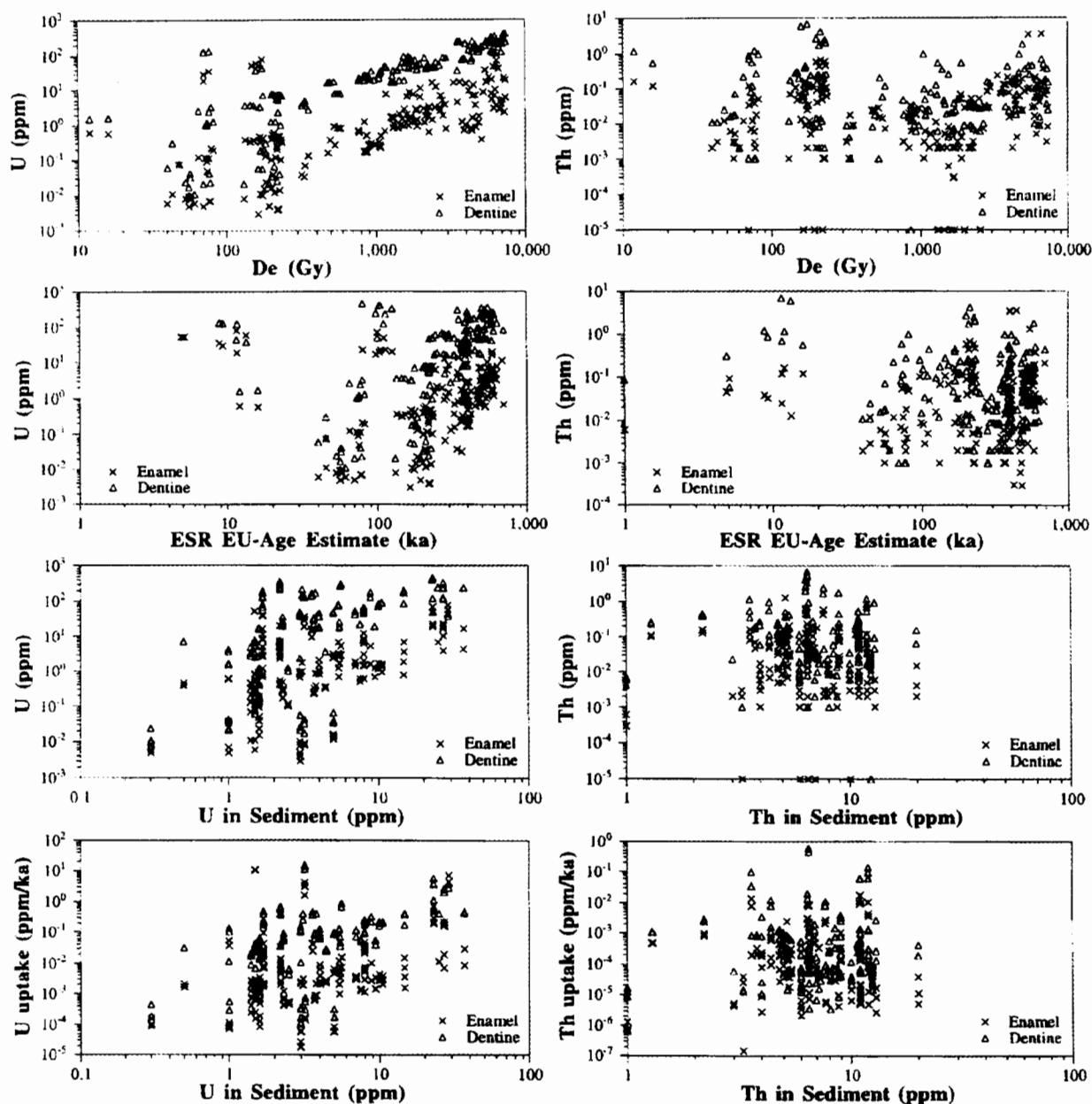

Figure 2
    Relationship between U (left) and Th (right) and the measured dose value (top row); the ESR age estimate (second row), the respective element in the sediment (third row). The bottom row shows the relationship between U and Th uptake versus the concentration of the respective elements in the sediment. There is a reasonable correlation between the U-concentrations and the measured dose value (enamel: r = 0.66; dentine: r = 0.77). All other U relationships have r values between about 0.35 and 0.6 indicating some weak relationships. Th in teeth is basically not related to any of the shown parameters (r < 0.2).

Results and Discussion

The teeth analysed were collected from a variety of sites ranging in their estimated geological age between about 5,000 years to greater than one million years. Figure 1 shows the results of 197 adjacent enamel and dentine layers. The correlation coefficients are given for the logarithmic values as shown in the diagrams. Figures 1A and 1B show that there is no relationship between uranium and thorium either in enamel (r = 0.29) or in dentine (r = 0.22). Relative high Th concentrations (> 0.5 ppm) were measured in 8 enamel samples and 17 dentine samples. There is a clear relationship between the uranium concentrations in enamel and dentine (r = 0.89, Figure 1C). The average uranium concentration in dentine (92±204 ppm) is about 20 times higher than the average U concentration in enamel (5.7± 2.6 ppm). It also seems that dentine reaches saturation in the range of about 100 to 1000 ppm. Figure 1D shows that there is a weak correlation (r = 0.51) between the Th concentrations in dentine (average: 0.32 ± 0.87 ppm) and the adjacent enamel layers (0.01 ± 0.38 ppm), the Th concentrations in dentine being about 30 times higher than in enamel.

Figure 2 shows the relationship between the U and Th concentrations with the past radiation dose, D_E, the approximate age of the samples and the concentrations of the respective elements in the sediment. The only clear relationship exists between the measured D_E and the uranium in enamel (r = 0.66) and dentine (r = 0.77). This is hardly surprising as the influence of the external gamma and beta dose rate from the sediment on the total dose rate diminishes with increasing U-concentration in the constituents of the teeth. Th, on the other hand, has only a minor influence on the total dose rate, and, hence, no relationship between Th concentrations and D_E value can be observed; the relationship between Th and the dose is for enamel: r = 0.11 and for dentine: r = 0.037.

One would generally expect that there is either a relationship between the uranium in teeth and age, the uranium in sediment or the combination of the two. As shown on the left had side of Figure 2, however, there is hardly any relationship between these parameters. The best relationship exists between the uranium concentration in dentine and age (r = 0.61), followed by correlation of the uranium in enamel and age (r = 0.35). The relationships between the U in dentine and enamel and U in sediment or uranium uptake vs U in sediment have all r-values in the range of 0.4 to 0.5 which indicate that there is only some weak influence between the chosen parameters. The diagrams of the Th concentrations in the constituents of teeth versus the other parameters clearly demonstrate that neither time nor the Th concentrations of the environment relate to the measured Th concentrations of the teeth. The highest correlation coefficients can be found between Th concentrations of the teeth and age (enamel: r = 0.19; dentine r = 0.11). The other relationships have r-values of less than 0.05.

Our results clearly imply that there is no relationship between the thorium concentration and any other parameter considered in this study. The thorium concentrations are usually very low (enamel: 0.01±0.38ppm; dentine: 0.32±0.87 ppm) even in those samples that show high U-concentrations. The question is whether Th can produce a significant contribution to the total dose rate. One has to keep in mind that the alpha and beta dose rates of 1 ppm Th is only about 28% and 20% of the corresponding U dose rates. By far the highest Th/U ratios were measured in sample 1030 from Florisbad, where the average Th/U ratios (of three subsamples) in enamel and dentine are 5.3 ± 0.6 and 2.5 ± 0.6, respectively. The measured Th concentrations increase the total dose rate by 7%. All other samples from Florisbad have much lower Th/U ratios (in the range of 0.01 to 0.2) so that no obvious explanation can be given for the unusual Th/U ratios of sample 1030. Considering that all other samples have significantly lower Th/U ratios, the effect of moderate Th concentrations, as found in this study, can generally be neglected.

The results also show almost no relationship between the uranium concentration and the age of the samples. This supports the findings of Grün & McDermott (1994) that it is not possible to postulate a particular mode of uranium uptake for age calculation without the combination of ESR and U-series analyses.

Conclusions

High thorium concentrations that may have an influence on the total dose rate seem to be exceptional. The results clearly imply that it is not possible to use a particular U-uptake model for calculating ESR age estimates of tooth enamel.

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Reviewer
H.P. Schwarcz

Comments

The U content of fossil teeth and bone has long been thought to give a crude age estimate. K.P. Oakley championed the FUN method of dating (Fluorine, Uranium, Nitrogen). Oakley determined U by beta counting. This gave surprisingly good estimates of the relative ages of bones. This was apparently a case of "the right answer for the wrong reason", since most of the increase in β-activity must be due to the growth of ^{230}Th and its daughters from the parent U isotopes, whose concentration hardly altered.

The present paper attempts to place this concept on a firmer footing, comparing the U and Th contents of tooth components with each other, and with age, dose, and environmental concentrations. The data are from a wide range of sites and faunal types; some of the variation may be depend on these variables. Surely, if the LU model (or any other continuous uptake model) is generally valid there must be some correlation between U content and age. The data shown on Fig. 2 suggest that any such correlation, if present, is overshadowed by intersite differences, and vagaries of the sample materials. It is encouraging, at least, that U shows a considerably stronger dependence on age and environment than does Th, confirming our general conception of Th as a geochemically immobile element.

The ICP method for U and Th determination are useful additions to our literature on analysis of these ever-interesting elements.

Thesis Abstract

Thesis title: Alluvial response to environmental change: Luminescence dating of the Quaternary sediment systems.

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Thesis submitted for the degree of Doctor of Philosophy at the University of Wales, September, 1995.

Abstract

Geomorphological investigations of Quaternary fluvial deposits in the Guadalope basin, northeastern Spain, have revealed an extended record of riverr and drainage basin response to long tem environmental change. Investigatiommns have focused on a 13 km long reach centred on the town of Castelserás, a 4 km long reach at Mas de Las Matas, a 2.5 km long reach at Forcall and a cross section of the valley at Aguaviva. A study was also made of a 1 km long reach in the neighbouring Rio Regallo catchment.

Luminescence dating, utilising a partial bleach methodology with infra-red stimulated luminescence (IRSL), was tested on alluvium collected from the River Danube. The IRSL age of 7 ± 1 ka agreed with archaeological evidence indicating a Mesolithic age. Modelling of the adopted procedure suggests accurate IRSL ages can be determined where residual luminescence signal is 10% or less. The presence of a residual signal necessitates the use of the partial bleach methodology to avoid overestimation of IRSL age.

Luminescence dating of 39 sediment samples from alluvial units in the Guadalope and Regallo basins provides a geochronology indicating that deposits span the Middle Pleistocene through to the historic period. The period c.227-130 ka was characterised by large-scale (locally up to 40m) aggradation and incision cycles associated with tributary stream and slope input of sediment. More gradual alluviation between c.122-30 ka was followed by renewed dissection and aggradation at c.24 ka, immediately preceding the Last Glacial Maximum. At least 4 river terraces were formed during this period, with heights above present river level of between 10 and 15 metres. River instability continued after the glacial maximum and into the Holocence, when the most recent major alluvial unit was formed 3 to 4,000 years ago.

The sedimentology of the alluvial fills indicates that coarse gravel, braided rivers dominated the fluvial system during the Pleistocene. Single thread, meandering rivers (gravel bedded were more common during the Holocene. Sediment geochemistry of the samples analysed using ICP-MS indicates that sediment sources within the catchment have not changed significantly during the Late Quaternary. LOI analyses highlight the importance of local sediment delivery to the trunk river by tributary streams.

Progressive entrenchment of the Guadalope valley floor during the Pleistocene would appear to reflet fong term regional uplift, with channel aggradation and dissection episodes linked to the climate changes shown by vegetation and oxygen isotope records. Rates of downcutting increadsed during oxygen isotope stages 2 and 3. This change probably occurred in response to a reduction of coarse sediment delivery to the main channel from tributary catchments, an increase in trunk stream competence and / or catchment uplift.

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Notices

The first conference on

NEW DEVELOPMENTS IN PHOTODETECTION

is organised at the BEAUNE Palais des Congrés (Côte d'Or, France)
from **june 24th to 28th 1996**, and brings about 200 people.

The main topics will be :

**photomultipliers
photodiodes
hybrid photodiodes, avalanche photodiodes,
gaseous photodetectors, ccd detectors,
germanium,
related electronics ...**

The meeting is organised by DSM-Saclay/CEA and CNRS/IN2P3
and sponsored by CEE and various manufacturers

Dead line for registration : **May 15th**

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Letters

Errata

It is with a red face that I must apologize for using the word « principle » instead of « principal » throughout a recent article (Huntley & Berger, Ancient TL 13-1 5-9, 1995) when referring to the « principal trap ».

It has come to my attention that there is a wrong number in Berger and Huntley « Test data for exponential fits » Ancient TL 7, 43-46, 1989. In table 1 under STRB87-1 the 3rd unbleached count is given as 21040.6. The correct figure is 20140.6.

D.J. Huntley

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