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Dating quartz sediments using the 325 °C TL peak: new spectral data

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Shortly after Mike Smith (1987) described the Puritjarra rock shelter in Australia's Northern Territory, the Adelaide Archaeometry group began collaboration with him to find thermoluminescence (TL) dates for the project one of the objectives being to check radiocarbon and thermoluminescence dates one against the other. The oldest C-14 age is 22,440 ±370 years. Samples (PJ1ES series) were taken within the shelter to a depth of 2.1 m which is much deeper than the apparent occupation, and from the dune field nearby (PJ2S series). We have since obtained a sequence of quartz inclusion TL dates from the site.

The TL ages provide consistent seriation with depth within the shelter. Ages agree well (3 ka) for test site PJ3S which was specially selected from the rear of the shelter with less likelihood of disturbance than for other sites. Most other comparisons suggest that the TL age is older than the most nearly corresponding C-14 age by five or six thousand years. This is a serious matter because it could be said to leave unresolved the true age of Puritjarra. However, the possibility of an error in TL methodology is even more serious for it would tend to bring into question TL dates in general.

Our feeling was that incomplete bleaching was the most likely cause for the discrepancy but we carried out extensive independent checks on other possibilities. These included repeat TL resampling of charcoal PL measurements on two samples and independent analysis for U, Th and K. We conclude that bleaching was incomplete for some samples although sampling problems for C-14 still remain possible.

We have carried out bleaching tests on a subset of the original samples as collected. They therefore included clay as well as quartz and it is i n this form that the original material would have been exposed to light. Samples PJ1ES/10 and PJ1ES/75 were exposed in thin layers to Adelaide summer sunlight and were found not to be well even after 21 days of summer sunlight. The PJ2S and PJ3S samples bleached rapidly to low levels and the surface samples from these two sites were found to have been already bleached, indicating long exposure to light in conditions presumed to reproduce the original conditions. This is consistent with the agreement between TL and C-14 ages for PJ3S and geomorphological arguments for the samples PJ2S from outside the shelter. Figure shows bleaching of glow curves for PJ1ES/75 with time as a parameter; it also shows the level to which bleaching proceeds in extracted and etched quartz samples.

It appears that resetting has been only partial a Puritjarra because the quartz used in the dating measurements is "shielded" from exposure to the sun by a thin layer of clay and/or iron oxide on the grains and because the orientation of the shelter virtually excludes direct sunlight.

The key question in dating bleached sediments is whether the materials (e.g quartz) have been exposed to sunlight for "long enough" to reset the TL clock. There are two schools of thought the "total bleach" and the "partial bleach" schools.

The total bleach school (e.g Singhvi et al, 1982) have argued that in the majority of cases the physical environment is such that essentially complete bleaching can be assured, although there is an inevitable element of judgement in this. The Adelaide group has tended to follow this philosophy (e.g Lu et al, 1988) and it was the philosophy used at Puritjarra in the first instance, because the materials were assessed as wind-blown sediments from an open site with long exposure. This turned out not to be true for all samples.

The partial bleach school, largely developed by Wintle and Huntley (1980) argue that since you do not know how much the sample has been bleached, then you should assume the worst and base your dating on a component in the TL that bleaches easily assuming that there is one. It has long been clear that there is such a component in quartz and Spooner et al (1988) confirmed that it is the 325 °C peak.

We have therefore devised a set of procedures which when used provide greater confidence in the degree of resetting. It is very similar to the procedure proposed by Franklin and Hornyak (1990).

Spooner et al (1988) showed that the 325 °C peak in the TL glow curve of quartz is extremely susceptible to bleaching by light of all wavelengths and that this continues to be true even in unseparated field samples; and we have confirmed this for PJ samples specifically. The half-intensity bleaching time by full sun is about twenty seconds for this peak and a few minute of full sun is sufficient completely to remove it. It can therefore be assumed that the conditions for "total bleach" were satisfied for this peak at the time of deposition and that TL ages determined from this peak by total bleach methods will be correct.

Fox (1990) has shown that the peak characteristically emits light peaked at 380 nm and that these wavelengths are selectively bleached. The 325 °C peak can therefore be selectively used for dating using suitable optical filters. Contour plots of the spectral data for a PJ1ES sample, glowed out and re-irradiated with 9 Gy before and after bleaching are reproduced in figure 2. In the unbleached sample, the 380 nm

emission ridges outwards from more intense emission peaking at 480 nm; after 40 s bleaching with 350 nm light it has gone.

The detector of the spectrometer is similar to that used for TL measurements and since the optical response of the spectrometer (without detector) is essentially wavelength independent, these spectral data are reproduced in their uncorrected form to illustrate the relative changes in spectral components of the TL emission which would be detected using a 9635Q PMT (with HA3). The spectrally-corrected response peaks in the red near 630 nm (inset fig. 2a). The easily-bleached "325 °C" peak appears at a temperature of 305 °C with the heating rate of 5 K/s used here. Apart from pre-dosing of the 110 °C peak, there was little change of the spectrum on reheating.

A suitable filter combination for isolating this peak with a 9635Q PMT is a UG-11 plus 7-59 (fig 3) which accepts 320-380nm and largely rejects other wavelengths that do not readily bleach. The UG-11 filter is a compromise between selection of the 325 °C peak and rejection of the neighbouring peaks; it does not fully isolate the former and the glow curve still has some contributions from other temperatures. The data in figure 1 were taken with the 7-59 filter; the inset in fig. 1 shows the enhanced response at 325 °C for the 7-59 + UG-11 combination. A UG-11 filter alone or a UG-2 filter is almost as satisfactory although the latter also transmits in the red.

Further, if as part of the laboratory dating procedures, bleaching is carried out only with light of wavelength longer than about 475 nm (eg using a GG-475 filter) it is possible selectively to remove the 325 °C peak without affecting any of the rest of the TL which can therefore be used as a baseline from which the 325 °C peak rise, and from which the 325 °C peak intensity can be found by subtraction as described by Franklin and Hornyak.

Application of these procedures to an artificially-aged sample recovered the correct dose and produced a reduction of the apparent age of PJ1ES/75 from about 30 ka to 22 ka. We are currently repeating the dating of PJ using the new techniques. We agree with Franklin and Hornyak that the amount of work involved is considerably more than is required for conventional total bleach dating. However this is a small penalty to pay to obtain more accurate dates.

Acknowledgements

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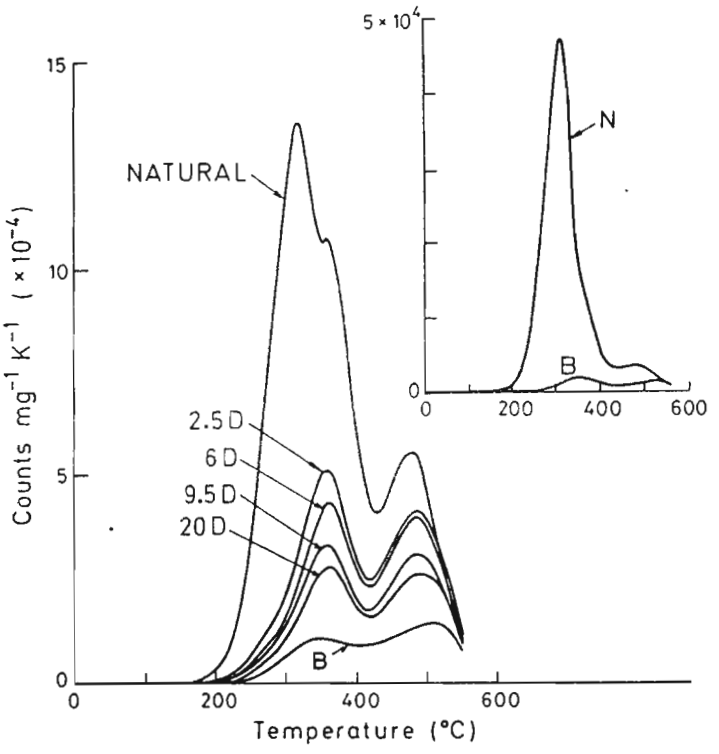

Figure 1
Glow curves for "natural" sample PJ1ES/75 before and after bleaching. Bleaching time are days of natural sunlight. Curve B is for extracted and etched quartz bleached for two days. The curves were obtained with 7-59 optical filter. For comparison purposes, the inset shows two corresponding curves using the filter combination 7-59 + UG-11.

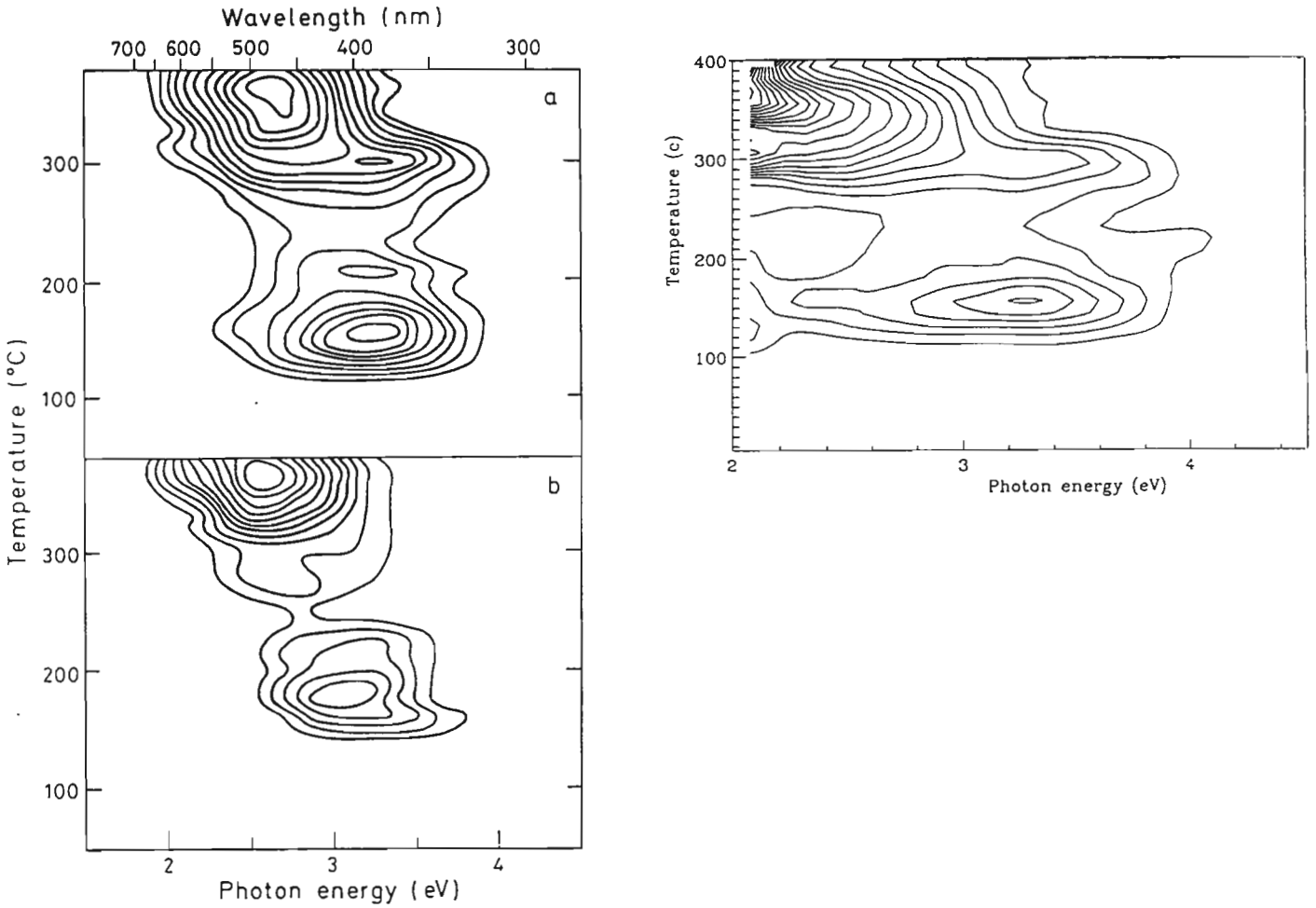

Figure 2.
Contour plots of the spectral data for a laboratory-irradiated PJ1ES sample.
(a) before bleaching (b) after 40s bleaching at 350 nm (2 mJ.cm^{-2}).
The wavelength scale is added for comparison purposes. The spectral intensities per unit bandwidth and kelvin are computed in energy space. The contour plot on the RHS is fig. 2a) corrected for instrument response.

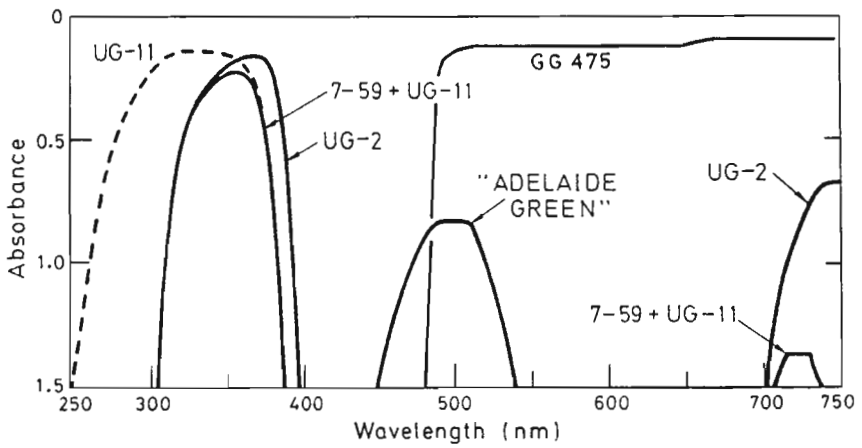

Figure 3.
Absorbance vs wavelength for some of the filters referred to in the text.