Ancient TL

www.ancienttl.org · ISSN: 2693-0935

Montret, M., Miallier, D., Sanzelle, S., Faïn, J., Pilleyre, T. and Soumana, S., 1992. *TL dating in the Holocene using red TL from quartz.* Ancient TL 10(3): 33-36.
https://doi.org/10.26034/la.atl.1992.199

TL dating in the Holocene using red TL from quartz

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Introduction

With a view to dating Quaternary volcanoes, our group has studied the red TL of quartz observed by Hashimoto and Habuki (1987) with quartz grains extracted from Japanese volcanic materials and by Huntley et al. (1988) in quartz grains from southern Australian beaches. More information was obtained for a peculiar orange-red peak, around 360 - 390 °C (5 °C/s heating rate) and 610 - 620 nm, which was observed in almost all the samples, from various origins, tested in our laboratory (Miallier et al., 1991). Several red TL ages of volcanic events between 14 and 800 ka were subsequently measured (Pilleyre, 1991; Pilleyre et al., 1992). At the same time, Hashimoto et al. (1991) applied the red TL to dating tephra layers correlated with the oldest prehistoric sites in Japan and obtained four ages in the range 60-200 ka.

In the work reported in this paper, we tested the red TL method on two known age samples younger than 10 ka, a brick from a Celtic potter's kiln excavated at Bas-et-Lezat (Puy de Dôme, France) and archaeologically dated to 120 ± 40 BC and a soil baked by a lava flow from the La Vache - Lassolas twin volcanoes (Chaîne des Puys, French Massif-Central) around 8.6 ka ago (sampled beneath the Saint Saturnin Castle). A ^{14}C date of 7,650 ± 350 BP (Sa-90) was produced for carbonized wood sampled below the lava-flow (Pelletier et al., 1959) and another ^{14}C date of 7,970 ± 125 BP (MBN-328) was obtained on a peat containing a tephra attributed to the same event by Juvigné and Gewelt (1987). These results fall just outside the limits of the currently available calibration curve; however, all data indicate that the difference between the actual age and the ^{14}C age is around 800-900 years for this period (Stuiver et al., 1986; Vogel, 1987; Bard et al.,1990). Huxtable et al. (1978) obtained a mean TL age of 8,100 ± 800 a using the fine grain and quartz inclusion techniques on the sediment baked by the lava-flow; Guérin (1983) obtained two TL ages for plagioclase grains extracted from the lava itself, of 9,150 ± 550 a and 8,820 ± 870 a, respectively. The weighted mean for these ages is 8,650 ± 450 a (1σ), although it is not possible to average rigorously because of the specific constraints of the ^{14}C technique.

Technique

The gamma annual dose rate was measured in situ using a portable gamma spectrometer (Sanzelle et al., 1988) and the cosmic radiation dose was calculated on the basis of the localization and depth of the samples (Prescott and Stephan, 1982); the beta dose rate from ^{40}K was calculated from potassium contents and the beta dose rate from the U and Th series was derived from thick source alpha counting (TSAC). The internal beta-equivalent dose rate within quartz grains was assumed to be 0.06 ± 0.05 Gy/ka (from the mean contents of U and Th of quartz grains from the same area , and assuming a value of 0.1 for the efficiency of alpha particles as measured using the red TL from quartz. Dose rate values are given in Table 1.

Quartz inclusions were prepared using a procedure based on Fleming's (1970) recommendations. It included gentle crushing, sieving, magnetic separation, hydrofluoric and hexafluorosilicic acid etching and heavy liquid separation. As a precaution, the preparation was carried out under subdued light, although the red TL of quartz seems to be insensitive to sunlight bleaching .

The irradiations were performed with a ^{137}Cs gamma source delivering 38 mGy/s to quartz. TL measurements were made at a heating rate of 5 °C/s in a nitrogen atmosphere, with - or without - preliminary evacuation. We have found that the red TL is liable to be accompanied by a significant spurious signal if the grains have been crushed or if a long time (several months) elapsed between preparation and measurements. However, this spurious TL can be strongly diminished by gentle acid etching and evacuation.

A longpass sharp cut-off filter Schott RG 610 (50% at 610 nm) was used with a bi-alkali EMI 9635QA photomultiplier tube. The use of a red extended PM tube appeared unnecessary; we observed no gain with such a tube (EMI 9558, kindly lent by the Oxford Laboratory), the lower limit of the technique being controlled by the signal-to-noise ratio.

The additive dose technique was used, with quartz grain aliquots of around 9 mg calibrated by volume. For the two samples, a 'second reading' growth curve was obtained with a laboratory reset (380 °C for 16h in air) natural sample .

Results

The brick. Although we had previously thought that around 10 ka would be the lower limit for the red TL to be measurable (Miallier et al., 1991), a red TL peak was obtained for samples from the brick (age about 2,100 a) after a careful background subtraction (figure 2). The signal-to-noise ratio for the natural signal was ~1 at the peak temperature.

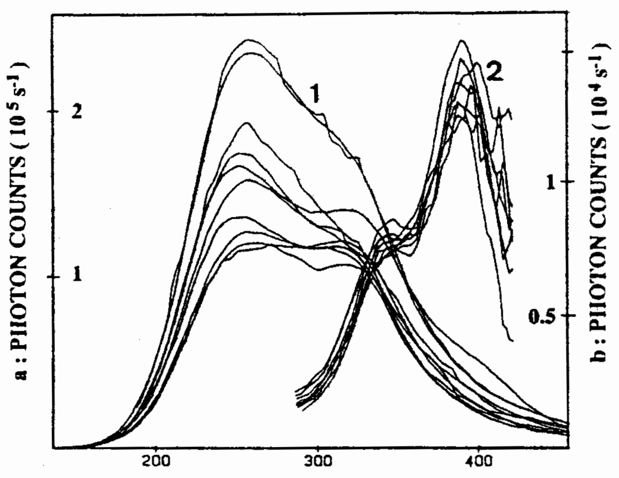
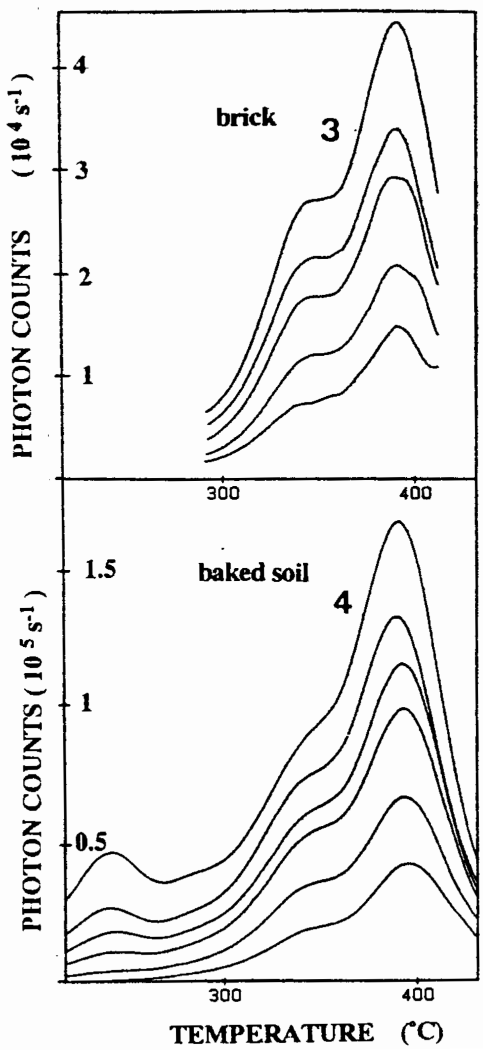


Figure 1. (above)
Individual glow-curves (background substracted) for the quartz of the brick measured using: a) blue filter; b) red filter under conditions described in text.

Figure 2. (opposite)
Glow curves for: a) brick and b) baked soil. The background was subtracted and the curves were averaged over 8 or 10 curves for each dose. The curves shown are, from the lowest intensity curve (natural), those obtained after administration of the following gamma doses: a) 4.53; 9.06; 13.6; 18.13 Gy and b) 22.66; 45.32; 67.98; 90.64, and 136 Gy.

This sample provides an example of the small scatter of the red TL peak: the sample to sample variation of the natural red peak was 6%, whereas it was 27% for the blue signal (figure 1), measured through a blue band-pass Leitz BG 12 filter (the weight dispersion, using the calibrated volume with 200-315 µm grains is usually 2% and this technique results typically in a standard deviation of 1-2% for the TL of good phosphors such as Al_2O_3 or $CaSO_4$:Dy). No difference was found between the curves recorded with or without evacuation. Thus, the red TL appears better distributed than the blue TL and probably less affected by feldspar remains and surface phenomena - which might explain the beneficial effect of HF etching observed by Franklin and Hornyak (1992); moreover, like these authors, we had observed, with samples from another origin, that most of the blue TL could be emitted by less than 10% of the grains (Ousmoï, 1989) and our efforts to normalize with the pre-dose peak were unsuccessful. In the case of the brick, we did not try the dose normalization with the blue filter; this technique can slightly reduce the scatter of the results when the changes of sensitivity are taken



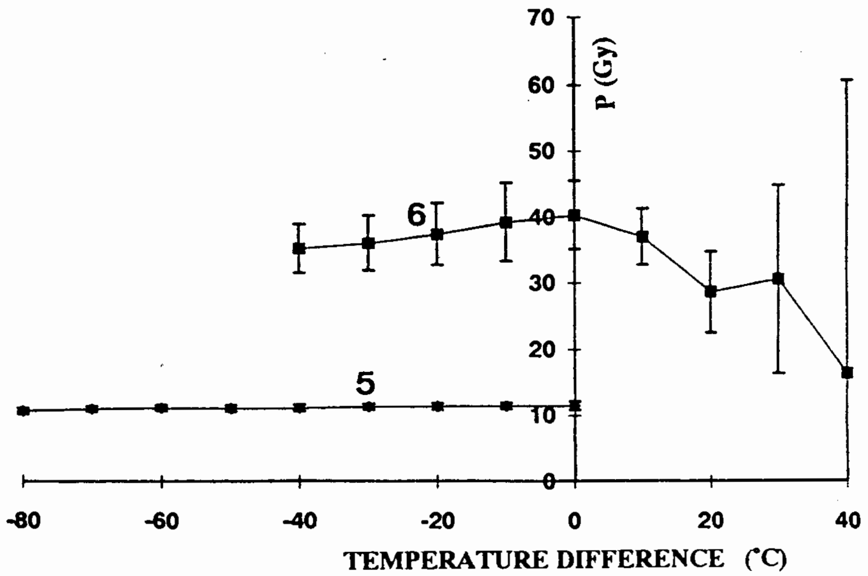


Figure 3.
Palaeodose plateau tests: a) brick; b) baked soil. Above the peak temperature (0 on the X axis), results are less reliable because of the predominance of the thermal background.

Table 1. Summary of results. Quoted errors correspond to 1σ. The evaluations of E and Q were based on the measurement of the peak intensity.

Sample	Grain size µm	E Gy	Q Gy	P Gy	Annual dose Gy/ka	Red TL Age ka	Expected age ka
Brick kiln (Cler 71)	200-315	2.41±0.36	9.11±0.53	11.52±0.61	4.69±0.28	2.46±0.20	2.11±0.04
Baked soil (Cler 207a)	100-200	2.83±0.48	37.5±5.2	40.3±2.3	4.32±0.19	9.34±0.67	8.65±0.45
Baked soil (Cler 207b)	200-315	3.65±0.61	33.7±2.4	37.4±2.5	4.19±0.19	8.92±0.72	8.65±0.45

into account (see Reviewers Comments by J.R. Prescott on the paper by Franklin and Hornyak, 1992).

The growth of the signals vs added dose was linear and the peak did not shift significantly towards low temperatures with increasing dose as is the case at higher doses (fig. 2). A sensitization of 1.3 was found following the second heating. The intercept correction E (terminology from Aitken, 1992) was not negligible and varied with temperature, as did the equivalent dose Q. A good palaeodose plateau (P=E+Q) was obtained (fig. 3), leading to the age (see table 1):

A (Cler71) = 2.46 ± 0.20 ka

The baked soil. The behaviour of the red TL was similar to that of the first sample, but for the shape of the growth curve, which was non-linear (saturating exponential) at high doses. A small shift of the peak with additional dose was indicated but was neglected within error limits (±3 °C). The sensitization was negligible and no change in shape was observed between the first and the second growth curves; an acceptable palaeodose plateau was obtained (fig. 3). The age, representing the average of the results of two grain sizes was (see table 1):

A (Cler 207 a/b) = 9.13 ± 0.72 ka

Conclusion

The two tests reported in this paper show that the red TL peak of quartz can be expected to give acceptable age results between 2 and 9 ka (table 1). This preliminary evaluation should not be extrapolated to older ages because new features will appear at higher doses such as (i) a significant shift of the peak with accrued dose and (ii) generalization of sublinear dose growth-curves which cause difficulties in extrapolations and in the estimation of error. These two topics will be discussed in forthcoming papers. Also, at high doses, low temperature peaks can more or less be superimposed on the 'good one' (~380 °C) and preheating can be used to isolate the peak. The conclusion of the present work that might be more securely extended to older samples is the confirmation (of previous work on the red TL of quartz) that anomalous fading and dose rate effects are either weak or negligible.

For Holocene samples the technique described in this paper is similar to the conventional quartz inclusion technique, the main drawback being the increasing predominance of the thermal background. However, to those TL workers faced with the common problems encountered with the quartz inclusion technique, such as poor reproducibility of the glow-curves and presence of sensitization in the low-temperature peaks overlapping those used for dosimetry, we recommend exploration of the use of red TL to reduce these problems.

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