THERMOLUMINESCENCE DATING RESULTS FOR THE PALAEOLITHIC SITE MAASTRICHT-BELVÉDÈRE

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ABSTRACT

A brief review of the thermoluminescence technique as it applies to palaeolithic sites is presented and the thermoluminescence age for Unit 4 at Maastricht-Belvédère based on measurement of seven burned flints is reported: 270 ka ($\pm$ 11, $\pm$ 22, Ox TL 712k).

INTRODUCTION

Thermoluminescence (TL) is a phenomenon exhibited to varying degrees by many minerals. Essentially it is the emission of light when a substance is heated, this light being additional to ordinary red-hot glow and usually occurring at a less elevated temperature. In so far as archaeological application is concerned the light is faint and a special high sensitivity photomultiplier is needed for measurement.

TL represents the release of energy that has been stored in the crystal lattice of the mineral, this energy being in the form of trapped electrons. These trapped electrons are the result of exposure to nuclear radiation and there is a weak flux of this from the radioelements (potassium-40, thorium and uranium) naturally present in minerals and soil. The basic notion of TL dating is that at the time of the event being dated the latent TL of the sample was effectively zero. Then during the millenia of burial the latent TL accumulated so that the TL observed from a sample to-day is a measure of age. The erasure of geologically acquired TL at the time of the archaeological event is an obvious essential for dating. In the case of burned flint it is the action of heat that achieves this, a temperature of around 400°C being needed. With sediment it is the action of sunlight in 'bleaching' the TL before deposition; windblown sediment such as loess will have a long exposure to sunlight while airborne and it appears that some types of waterborne sediment have been sufficiently exposed also.

The basic equation is:

$$\text{age} = \frac{\text{palaeodose}}{\text{annual radiation dose}}$$

The paleodose is evaluated from laboratory measurements of the archaeological accrued TL (the natural TL) and the sensitivity of the particular sample concerned, viz:

$$\text{palaeodose} = \frac{\text{natural TL}}{\text{TL per unit dose of nuclear radiation}}$$

Thus palaeodose represents the total dose that the sample must have received since the event being dated. Alternative terminology is equivalent dose (ED) and archaeological dose (AD).

The annual dose, or dose-rate, is determined by both laboratory and on-site measurements. It consists of two parts, the internal dose-rate from radioelements in the sample itself, and the external dose-rate from radioelements in the burial soil (up to a distance of about 25 cm from the sample). For flint the external component is often as much as 80% of annual dose and consequently the reliability of the age can be strongly dependent on an accurate evaluation of it. This component is alternatively called soil dose, environmental dose, gamma dose and gamma-plus-cosmic dose; the cosmic-ray contribution is typically about 20% of the external component, less in deep caves.

An important consideration in respect of the external components is the rapidity with which the sample became buried to a depth of 25 cm. Until that depth is reached it does not receive the full component; while lying on the surface uncovered it receives only half.

For on-site measurements we use (i) TL capsules and (ii) a portable gamma spectrometer. A TL capsule, usually of copper contains a highly sensitive TL powder and is buried in the soil in a situation that represents that of the sample as closely as possible; it is about 8 mm diameter and 40 mm long. Minimum burial time is a few months, a year being preferred; it is placed at the end of a 30 cm long auger hole. The detector of the gamma spectrometer is 65 mm in diameter so that a correspondingly larger hole is required; however in this case the measurement time is only half-an-hour.

The annual dose is influenced by the water content of sample and soil during antiquity. An upper limit to the effect is obtained by measuring the saturation content. The as-dug content can also be measured (using a tightly-tied plastic bag to avoid evaporation during transport to the laboratory): it is then a matter for discussion as to whether the average during burial was bigger or smaller than the as-dug value. Uncertainty about water content is one of the factors that limit the accuracy attainable and the collaboration of a soil scientist is important. For instance on this site our thanks are due to Burrough, Dirksen and Van der Westeringh from the Agricultural University of Wageningen for use of their detailed soil moisture analysis.

The maximum age that can be reached is dependent on the TL characteristics of the sample, its radioactivity, and the radioactivity of the soil. In round terms both flint and calcite can reach about half-a-million years, perhaps more; the limit for sediment is not yet established. The accuracy obtainable varies with circumstances, often being limited by uncertainty in water content. It is realistic to expect $\pm$10% of the age; somewhat better can sometimes be achieved but it is unlikely that the error limits will ever be reduced below $\pm$5% of the age. Although the accuracy is not high it is usually adequate for deciding in which of several possible isotope stages a site should be placed.

In respect of flint it is desirable to have several suitable samples from each context being dated, preferably half-a-dozen or more. The samples need to be well burned and large enough. They should not be exposed to sunlight or fluorescent light; they should be stored in the dark. The ultimate test of whether the burning is sufficient is an integral part of the TL measurement, the plateau test, and not all flints which seem to be well burned to the eye pass this test. The size requirement is a disc of flint roughly 3 cm x 1 cm. The flint is destroyed in the course of measurement.

Further information about application of TL dating to palaeolithic sites of NW Europe will be found in Aitken, Huxtable and Debenham (1985).

FLINT SAMPLES FROM MAASTRICHT-BELVÉDÈRE

These are all from the layer of fluviatile sediment containing the archaeological occupation, layer K in our nomenclature, which is Unit 4 as established by Vandenberghe et al., 1985.

Initial measurements began with a 'Maas egg' (Bv 161), for which the TL signal was in saturation, and a tiny scap of flint too small for routine analysis (Bv 1296). However the latter was large enough to confirm that the TL age was consistent with a date in excess of 200 ka. Further excavation produced a total of seven flints suitable for routine dating. Three of these were from the Site C area where we have gamma spectrometer and capsule measurements; they are K4 (Az-12/9), K5 (Cz-19/15) and K6 (Bz-20/2). K4 was from a less homogeneous part of the layer than the other two. We also have K11 (22/22-44) which was from a different part of the pit, Site F; a capsule measurement was made here.

K12 (dw 84/1) and K13 (dw 84/2) are from a manifestation of Layer K/Unit 4 about 25 m SE of the first Site C operations. Finally K14 (1984 11/bf) and K15 (1984 12/bf) and a 'loess-doll' K16 are from another part of layer K. We do not have gamma spectrometer or capsule measurements for these latter samples and we have assumed the Site C values apply. The TL method used for burned flint has been described by Huxtable and Jacobi (1982). Table 1 gives the measurement data and the ages evaluated for the flints. One flint (K15) had not been drained sufficiently by its first heating to be dated. The 'loess-doll' (K16) from layer K yielded a palaeodose of 45.5 $\pm$ 4.5 krad evaluated in the plateau region of the glow curve from 325° to 400°C. This is unlike the behaviour of calcite where growth in this region of the glow curve is non linear. The a value was 0.17 and the calculated age was 175 $\pm$ 35 ka. It would be unwise to infer anything from the discrepancy between this TL age and the flint dates as the 'loess-doll' might have been formed during a later pedogenesis.

GAMMA DOSE-RATE FROM THE SOIL

As will be seen from table 1 this is a dominant component of the annual dose. The value used for all flints is that measured with the gamma spectrometer in Unit 4 at the Site C area. The flints were found about 20 cm below the top of this layer and a greater distance above its base. The gamma spectrometer measurement was made 20 cm below the top and at this depth it is estimated that 95% of the dose came from layer K. The dose-rate evaluated for the Unit 5.1 layer above K was higher by 10%.

Table 2 gives the potassium, thorium and uranium concentrations evaluated from gamma spectrometer measurements in various levels of the Belvédère pit.

Dose-rates evaluated from buried capsules were 74 mrad/a for occupation layer (K) of Site C and 84 and 80 for the occupation layer in which K11 was found at Site F. It should be noted that these values include a cosmic ray contribution and that the overburden is not now the same as during antiquity. In calculating the age it is assumed that the average overburden was 2 metres; there is no significant effect on the calculated age for quite large variations in overburden thickness. A more important consideration is that since the flints were at a depth of 20 cm below the top of the layer, and further since the layer above was deposited fairly rapidly, there is a good assurance that flints were receiving the full gamma dose effectively for the whole burial period. If the overburden had been insufficient the calculated age would be less than the true age.

Laboratory measurements of soil alpha radioactivity gave a dose-rate that was 10% lower than the gamma spectrometer evaluation; this was based on thick source alpha counting and alpha spectrometric measurement of polonium-210. The agreement is considered satisfactory, the gamma spectrometer evaluation being preferred as more direct. Comparison of polonium-210 evaluation with thick source alpha counting indicated (45 $\pm$ 5%) radon escape, and that the degree of escape before excavation was not significantly different from that obtaining at the time of the gamma spectrometer measurement. Although the radon escape is strong, because there is no evidence for downward diffusion of uranium in table 2 it is considered justifiable to assume that there was no important interference from geochemical leaching.

SEDIMENT DATING

The results for sediment have been obtained by Debenham (see Aitken, Huxtable and Debenham, 1985), where the difficulty in respect of sediments in excess of 100 ka, and to some extent 50 ka is mentioned. For the younger sediments, the upper part of decalcified loess (our ref. *a*) gave an age of 17.2 $\pm$ 3.5 ka and the upper part of the calcareous loess (our ref. *d*) an age of 17.5 $\pm$ 3.4 ka; for the layer (our ref. *g*) presumed to be the Nagelbeek horizon the age obtained was 13.3 $\pm$ 3.0 ka.

CONCLUSION

Based on the 5 flints closest to Site C and from it i.e. K4, 5, 6, 11 and 13 the average age obtained is 270 ka ($\pm$11, $\pm$22, OxTL 712 k). Inclusion of K 12 and 14 also would reduce this age by only 3%. The first error limit quoted (see Aitken and Alldred, 1972; Aitken, 1976) gives the standard error derived from the scatter of the individual ages about the mean value. The second one is the predicted error limit taking into account all quantifiable sources of uncertainty. It is the latter limit that should be used in making comparison with dates based on other techniques. Thus at the 68% level of confidence the age can be said to lie within the range 250-290 ka, and at the 95% level within the range 225-315 ka.

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TABLE 1: FLINT TL MEASUREMENTS AND RESULTANT AGES

Sample	*K4*	*K5*	*K6*	*K11*	*K12*	*K13*	*K14*
Palaeodose (krad)							
fine grain	33±3	24.7±1.5	28±1.5	31.3±1.5	29.4±1.4	28±1.8	23.5±1.5
90-125 micron grains	32.2±1.3	27.6±1.3	27±1.3	31.5±2.3	31.9±2.3	27.5±1.8	25.2±1.5
$\alpha_0(Ks^{-1})$	1.26	0.35	1.60	0.93	1.59	0.94	0.43
K_20 (%)	0.115	0.059	0.076	0.178	0.076	0.062	0.197
a value	0.072	0.098	0.11	0.112	0.115	0.100	0.151
Dose-rates (mrad/a)							
D'_α	8.2	2.8	16.2	9.4	16.8	8.5	6.2
D_β	16.5	6.1	16.4	7.6	16.3	10.6	16.4
$D_{\gamma+c}$	85	85	85	85	85	85	85
Total	110	94	118	102	118	104	107
Age (ka)	300±32	263±27	238±20	307±28	250±21.5	269±26	219±20

1. TL ages have been calculated using the palaeodose for fine grains. Error limits on the ages are at the 68% level of confidence, calculated on the Aitken and Alldred (1972) system.
2. α_0 is the unsealed thick-source count-rate (per ks) for a 42 mm diameter zinc sulphide screen with the electronic threshold setting such that for a thorium only sample the fraction of pulses above the threshold would be 0.85.
3. The gamma dose-rate is derived from the gamma spectrometer measurements made near the find spot of K 4, 5 and 6, i.e. in layer K of Site C. The measured moisture content in the soil at that time was 0.16 of the dry weight. It is assumed that during antiquity the moisture content was 0.16 ± 0.05. The saturation water content was 0.21.
4. The value for the cosmic-ray dose rate has been taken as 13 ± 3 mrad/a, corresponding to an average overburden during burial of 2 metres.
5. D'_α has been taken as (0.75±0.25) of the effective alpha dose-rate assuming homogeneity. The reduction is to allow for the possibility of some anti-correlation between alpha activity and TL sensitivity.

TABLE 2. IN-SITU GAMMA SPECTROMETER EVALUATIONS AT BELVÉDÈRE

	U		*Th*		*K_2O*		*Water content as % of dry weight WC*
	ppm		*ppm*		*%*		*%*
712a	2.3 ±0.3	(2.7±0.4)	8.2±0.4	(9.6±0.5)	1.73±0.09	(2.03±0.11)	15
c	2.6 ±0.3		8.2±0.4		1.92±0.09		
d	2.4 ±0.2	(2.7±0.2)	7.7±0.4	(8.8±0.5)	1.55±0.07	(1.76±0.08)	12
e	2.4 ±0.2		8.2±0.4		1.56±0.07		
f	3.1 ±0.2		6.3±0.3		1.45±0.07		
g	2.02±0.14	(2.34±0.16)	8.4±0.2	(9.7±0.2)	1.62±0.06	(1.88±0.07)	14
h	1.83±0.18		9.7±0.3		1.65±0.08		
i	2.14±0.26		8.6±0.4		1.49±0.08		
j	1.59±0.26		7.2±0.4		1.19±0.07		16
k	1.62±0.16		5.8±0.2		1.06±0.05		13

The values quoted are the result of in situ measurement with a gamma spectrometer (sodium iodide). They have not been corrected for water content and hence the values appropriate to dried sediment will be higher by about 15% (on the basis of the water content that has been measured for 5 of the layers, as indicated).