Recherches aux grottes de Sclayn, vol. 1,
LE CONTEXTE, E.R.A.U.L., 27, Liège, 1992, pp. 175-178.

THERMOLUMINESCENCE DATING OF BURNED FLINT AND STALAGMITIC CALCITE FROM GROTTES DE SCLAYN (NAMUR)

J. HUXTABLE and M. J. AITKEN*

Thermoluminescence (TL) is the emission of light when a mineral is heated. This light is additional to the ordinary red-hot glow; usually it occurs at a lower temperature. TL represents the release of energy that has been stored in the crystal lattice of the mineral. The stored energy is in the form of trapped electrons which have been excited by exposure to a weak flux of nuclear radiation. The radiation comes from the naturally occurring radioelements (potassium 40, thorium and uranium) present in samples and soil.

The basic tenet of TL dating is that at the time of the event being dated the latent TL of the sample was reset to zero by some method: then during the burial period the TL re-accummulates so that the intensity measurable today is related to the age. The erasure of geologically acquired TL at the time of the archaeological event is by heating in the case of burnt flint; a temperature of around 400°C is necessary. For stalagmitic calcite it is the actual formation of the calcite crystal which is the event being dated.

The basic age equation is

$$\text{Age} = \frac{\text{Archaeological dose}}{\text{Annual radiation dose}}$$

The Archaeological dose (A.D.) is evaluated from laboratory measurements of the TL accrued over the burial period (NTL) and the sensitivity of the sample.

$$\text{A.D.} = \frac{\text{NTL}}{\text{TL per unit dose of radiation}}$$

The annual radiation dose is determined by laboratory and on-site measurements. It is made up of two parts; the internal dose taken from the sample and the external one from the surrounding burial soil (up to a distance of 0.3 metre from the sample). For flint and calcite this latter component may be as much as 80% of the total annual dose and so the reliability of the age depends on an accurate evaluation of it.

The annual dose is influenced by the water content of sample and soil during burial. An upper limit to the effect is obtained by measuring the saturation water content, and the as-dug content can also be measured. Uncertainty about water content over the burial period is one of the chief factors limiting the accuracy attainable.

*Research Laboratory for Archaeology and the History of Art, 6 Keble Road, Oxford OX1 3QJ, UK.

Measurements (Site TL ref. n° 230)

FLINT

Only 2 flints suitable for TL dating were found on the site; these were from the cave in levels 1A (archaeological ref. n° SC. 82.113.D15, TL ref. n° a1) and 5B (archaeological ref. n° SC. 82.370.G13, TL ref. n° a2). Several burnt rocks were found but these were not suitable because their TL signals were in saturation. It is sometimes possible to use burnt stones other than flint for TL dating but on older sites this is often limited because of their higher radioactivity. This means they return to TL saturation much faster than flint. The flints were dated by the method previously described in Huxtable and Jacobi 1982.

Although measurements were made on the site using a portable gamma spectrometer and many calcium fluoride dosimeters were measured from the site after being in place for a year, the soils were also analysed in the laboratory using a high resolution gamma spectrometer (see for example Murray 1981). This was because radon emanation from some of the soils had suggested that disequilibrium effects might be present. The germanium gamma measurements do show indications of disequilibrium (see Table 1); this suggests the occurrence of geochemical leaching. The environmental doses used to evaluate the TL ages have been calculated using these analyses, on the assumption that the geochemical conditions which exist in the cave today have been the same throughout the burial period. If this assumption is not correct then the ages obtained by TL (or by ESR) may be systematically in error.

Only one dosimeter was put into layer 1A but four were inserted into layer 5B: they recorded variations of ± 30% within this layer. However if one calculates the ages using any other of the laboratory techniques used for dose rate evaluation (ie portable gamma spectrometer, calcium fluoride dosimeter, or thick source alpha counting modified to take account of radon emanation by the use of a gas cell) all the ages lie within one standard deviation of the quoted TL age. The annual internal dose was measured using thick source alpha counting and potassium analysis in the usual way. The annual cosmic dose (7 mrad) was from portable gamma spectrometer measurements.

The TL age of level 1A was found to be 44,000 years B.P. (± 5,500; ref. n° OxTL 230a1). The total annual dose was 140 mrad, 63% of this being due to the environment. This TL age is considered to be in good agreement with a C14 age of 38,600 years B.P. (± 1,500 ref. n° Lv 1377) for a bone fragment from this level. The TL age of level 5B was determined as 130,000 years B.P. (± 20,000; ref. n° OxTL 230a2). The total annual dose was 102 mrad, 83% of this being due to the environment.

Soil moisture content was assumed to be (0.75 ± 0.25) of saturation throughout the burial period, and the error limits quoted represent the total error at the 68% level of confidence.

This TL age is supported by a TL measurement done on the sediment from layer V section 10/11 above the flint layer by Dr N. C. Debenham when working in this laboratory. He obtained an apparent age of 80,000 years B.P. but because of the presumed fading effect in loess (Debenham 1985) he concluded that layer V was "older than 100,000 years".

CALCITE

The application of TL dating to calcite has been fully described in Debenham and Aitken 1984. Two samples of calcite were dated (laboratory reference C(i) and C(ii)). They came from a small block of calcite in square G16 at the top of level 4. Calcium fluoride dosimeters were inserted into holes drilled into the calcite block. The block was left in situ for a year and then removed. The TL samples used for dating were from the calcite immediately surrounding the dosimeters.

The archaeological doses of C(i) and C(ii) were 14.8 and 15.4 krad and their ages were 99,000 and 104,000 years B.P. Half the annual dose was from the calcite and the rest was from the environment.

The average TL age of this level is 100,000 years B.P. (± 15,000; ref. n° Ox TL 230c).

ACKNOWLEDGEMENTS

Our thanks are due to Dr R. Davie for the germanium gamma spectrometer analyses.

REFERENCES

DEBENHAM, N.C., 1985. Use of UV emission in TL dating of sediments. *Nuclear Tracks and Radiation Measurements*, 10, pp. 717-724.

DEBENHAM, N.C. and AITKEN, M.J., 1984. Thermoluminescence dating of stalagmitic calcite. *Archaeometry*, 26, pp.155-170.

HUXTABLE, J. and JACOBI, R.M., 1982. Thermoluminescence dating of burned flints from a British Mesolithic site : Longmoor Inclosure, East Hampshire. *Archaeometry*, 24, pp.164-169.

MURRAY, A.S., 1981. *Environmental radioactivity studies relevant to thermoluminescence dating*. Unpublished D. Phil. thesis, Oxford University.

Isotope used to determine the activity	Soil 1A	Soil VB	Soil V
U-235, U-234	47.2 (3.5)	45.2 (3.2)	46.1 (3.1)
Th-230	21.7 (23.8)	31.4 (22.3)	25.8 (21.3)
Ra-226, Pb-214 Bi-214	46.4 (1.2)	33.6 (1.0)	38.8 (1.0)
Pb-210	38.9 (3.5)	34.1 (3.2)	43.4 (3.2)
Average Activity for U chain	45.7 (1.1)	34.6 (0.9)	39.8 (0.9)
Ac-228	42.6 (3.1)	21.5 (2.7)	52.8 (3.0)
Ra-224,Pb-212 Bi-214, Tl-208	40.0 (1.2)	20.0 (0.8)	50.8 (1.2)
Average Activity for Th chain	40.4 (1.1)	20.1 (0.8)	51.1 (1.1)
K-40	365.9 (34.9)	198.7 (28.4)	481.3 (32.3)
Saturation water content (% of dry weight)	30	20	26

Table I : Gamma spectrometer activity measurements (Bq/kg) for soils used in the TL dating.

Note : The figures in parenthesis represent, in Bq/kg, the ± error limits at the 68% level of confidence.