M. Otte, M. Patou-Mathis et D. Bonjean (éds.). *Recherches aux grottes de Sclayn*, vol. 2,
*L'ARCHEOLOGIE, E.R.A.U.L.*, 79, Liège, 1998 : 39-43.

# THERMOLUMINESCENCE DATING OF STALAGMITIC CALCITE FROM LA GROTTE SCLADINA AT SCLAYN (NAMUR)

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The human mandible, SCLA 4A-1, which was found in La Grotte Scladina in July 1993, lay stratigraphically between two stalagmitic floors. The upper stalagmitic floor, which is referred to as CC4, was formed on the surface of Couche 4A (Sup) and is overlain by Couche 3. The lower stalagmitic floor, identified as CC14, separates Couche 4A (Sup) from Couche 4A (Inf).

Samples of *in situ* stalagmite were taken from both floors for thermoluminescence (TL) dating. Two samples (QTLS refs. SCL3 and SCL4) were removed from the upper floor, CC4, which was exposed in section H/I at square 27 (approximately 28 cm from the boundary of 26/27). The thickness of the *in situ* stalagmitic floor at this position was 15 cm, but the lower parts of this floor had an open laminated structure containing significant amounts of detrital material. The two TL samples were both situated between 1 cm and 3 cm from the upper surface of the stalagmitic floor, where the calcite was relatively compact and clean.

Two further TL samples (QTLS refs. SCL5 and SCL6) were taken from the base of a large *in situ* stalagmitic boss, which formed part of floor CC14. The boss was half-exposed in the section 30/31, and situated in square D (with its centre approximately 30 cm from the boundary of C/D). The samples were removed by drilling a 7 cm diameter core almost vertically through the stalagmitic boss. The borehole met the underlying sediment at a point beneath the top of the formation. At its base, the stalagmite was approximately 70 cm across. The calcite core was cut into sections of 2 cm lengths. The section which was 2 - 4 cm above the base of the stalagmite was identified as SCL5, while the identifier SCL6 was given to the section which was at a height of 6 - 8 cm.

## TL MEASUREMENTS

The outer surfaces of the TL samples that had been exposed to light were cut away. The remaining interior portions were crushed and two grain size fractions were collected. The larger grains, of 90 - 150 µm size, were etched in dilute acetic acid, washed, dried, and collected between 75 µm and 125 µum sieves, before being deposited onto stainless steel discs. Fine grains, of approximately 2 - 10 µm size, were collected from the crushed calcite according

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to their settling times in 0.2% acetic acid; they were washed, and then deposited from acetone suspension onto aluminium discs.

The larger grains (75 - 125 μm) were used to measure the palaeodose, which is the radiation dose received by the calcite since its formation. Palaeodose evaluation was carried out by the additive dose method, using first and second glow measurements, followed by pre-dose equalisation and fourth glow normalisation. All glows were performed with a heating rate of 5°C/s, and TL was recorded by an EMI 9235 QA photomultiplier through Corning 5-60 and Schott BG39 filters. The TL intensities at the 275°C peak were used for the analyses.

First glow measurements were extrapolated using the basic form of the second glow growth curve. Figure 1 illustrates the method of palaeodose evaluation in the case of sample SCL3. The natural TL intensity and the growth of TL with dose are shown by the first glow measurements (squares). The second glow data (crosses) show the subsequent regeneration of TL by laboratory irradiation. The non-linear TL growth seen in the second glow measurements is typical of stalagmitic calcite. The form of this growth curve has been shifted horizontally along the dose axis and scaled along the vertical axis to fit the first glow data points. The vertical scaling factor accommodates the change in TL radiation sensitivity which is evident from the two sets of measurements. The palaeodose is evaluated as the intercept of the extrapolated first glow growth curve with the dose axis. Measured palaeodoses are listed in table 1.

The discs of fine grains were used to measure the relative TL sensitivities of the samples to alpha and beta radiations. Fine grains are necessary for this measurement because large grains would not be completely traversed by the short-ranged alphas. Some of the discs were irradiated by alpha or beta radiation while others were left unirradiated. First glows were then measured using the same conditions as for the large grain discs, and the TL responses to the two types of radiation were compared. If necessary, a normalisation measurement was performed using second glows.

## DOSE RATE ASSESSMENTS

Internal alpha and beta dose rates to the stalagmitic samples were assessed by means of alpha counting, potassium analyses and water content measurements. In table 1, the calculated effective alpha dose rates have measurements errors of approximately ±17%, and the beta dose rates have errors of between ±7% and ±10%.

Environmental gamma and cosmic ray dose rates were assessed by portable gamma spectrometer. In order to measure gamma dose rates to SCL3 and SCL4, spectrometer measurements were made behind the section H/I immediately above and below the *in situ* stalagmitic floor, CC4. The variation of gamma dose rates within and around the calcite floor were computer modelled using experimentally determined attenuation factors (Debenham and Aitken 1984). The infinite matrix gamma dose rate of the stalagmitic floor was assessed from laboratory measurements of its alpha activity and potassium content. The infinite matrix gamma dose rates of Couche 3 and Couche 4A (Sup) that were deduced from the computer modelling agreed with spectrometer measurements taken within these deposits remote from stalagmitic formations. The gamma dose rate of Couche 3, which consists of stoney material, was much lower than that of Couche 4A (Sup), which is a loessic deposit.

Gamma dose rates to SCL5 and SCL6 were determined from spectrometer measurements taken inside the borehole from which the samples were taken. Measurements were made at two heights within the borehole close to the base of the stalagmite. The data agreed well with computer modelling of the gamma attenuation across the interface between the stalagmite and Couche 4a (Inf). The infinite matrix gamma dose rate of the calcite was assessed from laboratory measurements on SCL5 and SCL6. Gamma and cosmic dose rates to the TL samples are given in table 1. Uncertainties in the gamma dose rates to SCL3 and SCL4 were ±14%, while those to SCL5 and SCL6 were ±7%.

## TL DATES

When calculating the TL dates of the stalagmites, allowance has been made for temporal variations in the alpha, beta and internal gamma dose rates to the samples. These changes are caused by the long-term variations in U-234 and Th-230 activities in the stalagmite which result from the initial conditions of radioactive disequilibrium in the uranium series. The overall effect of these variations is a gradual increase through time of the dose rate experienced by the stalagmite.

The TL dates are presented in table 1. The quoted error limits include all random and systematic uncertainties, and refer to the 68% confidence level. The TL dates of samples SCL3 and SCL4 are indistinguishable from each other, and give a weighted mean date for the formation of the upper part of the stalagmitic floor, CC4, of 117.2 ± 11.2 ka BP. Likewise, the TL dates of samples SCL5 and SCL6 are not significantly different from each other. They form a weighted mean date for the lower 8 cm of the stalagmitic boss in Square D30/31 of 122.0 ± 11.7 ka BP.

While the two mean dates are not discernibly different, they are in the correct stratigraphic order. They suggest that both stalagmitic floors, CC14 and CC4, date from the early stages of the Last Interglacial, and probably coincide with oxygen isotope stage 5e or 5d. As these floors bracket the human remains, SCLA 4A, this period is also the most likely date for the phase of occupation that the remains represent.

| Sample Ref. | Palaeo-Dose (Gy) | Alpha Dose Rate (Gy/ka) | Beta Dose Rate (Gy/ka) | Gamma Dose Rate (Gy/ka) | Cosmic Dose Rate (Gy/ka) | TL Age (ka) |
|---|---|---|---|---|---|---|
| SCL3 | 41.3±1.4 | 0.170 | 0.066 | 0.175 | 0.038 | 117.7±13.1 |
| SCL4 | 40.3±1.4 | 0.159 | 0.067 | 0.174 | 0.038 | 116.7±13.2 |
| SCL5 | 43.0±3.4 | 0.143 | 0.090 | 0.212 | 0.032 | 112.8±11.5 |
| SCL6 | 35.7±1.8 | 0.124 | 0.052 | 0.132 | 0.032 | 132.5±12.5 |

**Table 1**

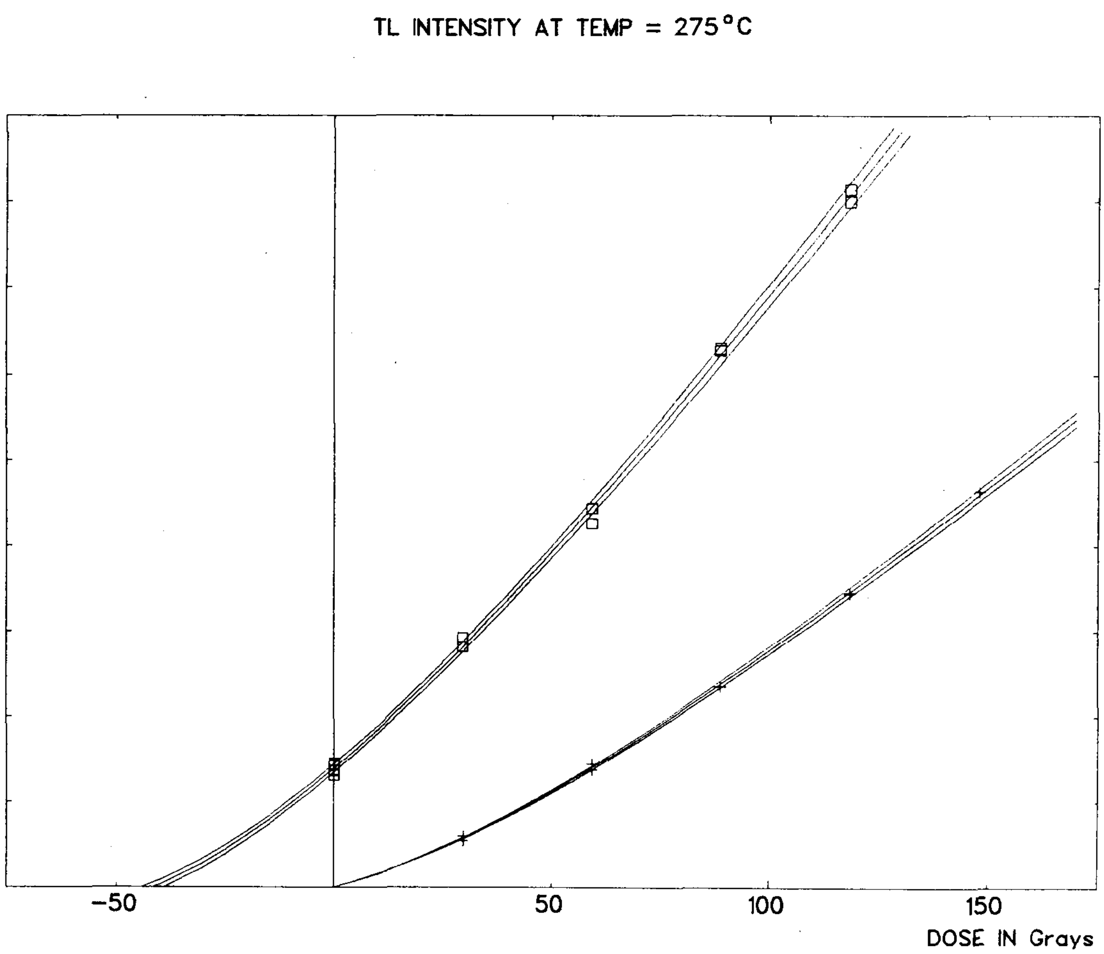


**Fig. 1**

## FIGURE CAPTION

Figure 1. Growth curves of TL *versus* radiation dose. Squares show TL first glow measurements, which include the natural TL intensity (at zero dose) and TL induced by four different additive beta doses. Crosses show the non-linear growth of second glow TL intensity with increasing radiation dose. The curve which has been fitted to the first glow measurements has the same basic form as that fitted to the second glow data, and its intersection with the dose axis evaluates the palaeodose.

## REFERENCE

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