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The effect of light exposure on the thermoluminescence signal from calcitic opercula

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ABSTRACT

Recent work has suggested that the thermoluminescence (TL) signal of opercula from the gastropod *Bithynia tentaculata* can be used to date the formation of calcite by the organism when it was alive. The two TL peaks of interest for dating are located at ~250 °C (Peak 2) and ~350 °C (Peak 3) when measured at a heating rate of 0.5 °C.s^{-1}. This paper assesses whether these peaks are altered by exposure to visible light, as this is important for how samples are collected in the field, and handled in the laboratory prior to measurement. Neither peak shows systematic change for exposures in a solar simulator of less than 24 h in duration. For longer exposures in the solar simulator the intensity of Peak 2 increases, possibly due to phototransfer. In contrast, the TL signal from Peak 3 is not affected by light exposure in the solar simulator for periods of up to 60 h, or by exposure to natural daylight with the UV-component removed for periods of up to ~26 d. One experiment which exposed an operculum to natural daylight for ~5.5 months led to a reduction in the TL signal from Peak 3 by 16 %, but such long exposures are unlikely in sampling and sample preparation. The lack of impact of daylight exposure on Peak 3 indicates that opercula-bearing samples can be collected and processed in normal daylight conditions, and that museum specimens are suitable for TL dating provided an associated sediment sample is available for dose rate calculations. However, as a precaution it is still recommended that light exposure is minimised where possible.

1. Introduction

The gastropod *Bithynia tentaculata* produces a calcitic operculum that acts as a trapdoor to its shell (Checa and Jiménez-Jiménez, 1998). These calcitic opercula yield a thermoluminescence (TL) signal that increases in response to ionizing radiation up to doses of 6 kGy or higher in the laboratory, meaning that they have the potential to date the last ~3 Ma (Stirling et al., 2012; Duller and Roberts, 2018). One advantage of using biogenic calcite for TL dating is that the event being dated is the formation of the calcite crystals when the organism was alive (e.g. Medlin, 1959), and unlike sediment dating using quartz or feldspar (Wintle, 2008) exposure to sunlight or heating is not required to reset the signal. However, whilst the TL signal from biogenic calcite has been investigated sporadically since the 1960s (e.g. Johnson, 1960; Johnson and Blanchard, 1967; Ninagawa et al., 1988, 1992, 1994; Carmichael et al., 1994; Duller et al., 2009; Colarossi et al., 2024), the focus has not been on the effect that light exposure has on these materials.

Previous work on optical bleaching of the TL signal from calcite has been undertaken on limestone and marble (e.g. Liritzis et al., 1996; Bruce et al., 1999; Liritzis, 2011), artificially prepared calcite crystals (e.g. Medlin, 1959; Visocekas, 1979) and speleothems (e.g. Wintle, 1978). Bleaching studies undertaken on limestone and marble reported rapid initial decay of the TL peaks observed at ~250 °C and ~350 °C, although the ~350 °C peak was observed to bleach more slowly than the ~250 °C peak, and after the rapid initial drop, the ~350 °C peak does not reduce further (Bruce et al., 1999; Liritzis et al., 1996). Furthermore, Bruce et al. (1999) bleached limestone samples with a Hönle SOL2 solar simulator through various filters and found that light between 340-400 nm was most effective at bleaching the TL signal, with little bleaching for wavelengths above 500 nm. Wintle (1978) made measurements of the 275 °C TL peak on five speleothem samples that were either: i) unbleached, ii) placed under a UV lamp (365 nm), or iii) placed under a tungsten-halogen lamp (emits across the visible light spectrum). She found three samples were unaffected by light from either lamp, while one sample showed an increase under both lamps, and another showed a decrease under both lamps. Wintle (1978) concluded that daylight exposure would not bleach the TL signal and that speleothems could be handled under normal laboratory light conditions.

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Given the disparate findings between bleaching studies undertaken on limestone and marble, and those on speleothems, it is not clear what impact light exposure may have on the TL signal from calcite in *Bithynia tentaculata* opercula. It is important to understand the effect of light exposure on these opercula to assess what precautions are necessary during sample collection and preparation, and to consider whether samples from museum collections where no special controls on light exposure were undertaken can be used for dating. The aim of this paper is to explore the effect of light exposure on the TL signal from calcitic opercula of *Bithynia tentaculata* using artificial (SOL2) and natural light sources.

2. Material and methods

The opercula used in this study were collected from the Purfleet Shell Bed (Aber206/PFSB) at Purfleet, Essex, southeast England (Bridgland et al., 2013). Sample processing was carried out in the Aberystwyth Luminescence Research Laboratory under the subdued red-light conditions used for quartz and feldspar preparation. Opercula were cleaned in distilled water in an ultrasonic bath to remove any sediment grains adhering to the surface, followed by 48 h in 12 % sodium hypochlorite to remove any surface organic material.

Artificial light exposure was carried out using a Honlë SOL2 solar simulator, which produces a spectrum similar to that of natural sunlight across a wavelength range of ~340–750 nm and delivers a power density of 70 mW cm^{-2} (Winzar et al., 2025). Exposure to natural daylight was undertaken at Aberystwyth University (52° 25′ N, 4° 4' W). TL measurements were undertaken on either an automated Risø TL/OSL DA-10 reader equipped with an EMI9635 QA photomultiplier, or a DA-20 reader with an ET-9107 photomultiplier (Lapp et al., 2015). Both instruments filtered TL emissions through 4 mm of Schott BG-39 filter. Irradiation was delivered by a $^{90}Sr/^{90}Y$ beta source with a dose rate of 0.018 Gy s^{-1} or 0.079 Gy s^{-1}, respectively. Beta source calibration was completed using calcitic opercula gamma-irradiated at Risø DTU Health Tech with doses of 250 Gy and 500 Gy. Heating was undertaken in an oxygen-free nitrogen atmosphere at a rate of 0.5 °C.s^{-1} up to a maximum of 400 °C, with a second heating to 400 °C being made so that the black body radiation could be subtracted. Individual opercula were placed on steel cups provided by the manufacturer for all TL measurements. Prior to beginning the light exposure experiments, the opercula had been used to construct a dose response curve, thus no natural TL signal remained.

3. The effect of light exposure on the TL signal

The first experiment was designed to observe the impact of the SOL2 solar simulator on all the TL peaks present. A beta radiation dose of 72 Gy was given to two opercula, following which they were exposed to light in the SOL2 for periods of time from 60 s up to a maximum of 216,000 s (60 h). After each light exposure the TL signals from the opercula were measured (L_x, Table 1), followed by delivery of a test dose (43 Gy beta irradiation) and measurement of the TL signal to allow for sensitivity correction (T_x, Table 1). Additionally, measurements were also made where no light exposure in Step 2 of Table 1 was undertaken.

Fig. 1a shows TL glow curves obtained from one operculum after a range of different durations of light exposure. The TL curves were normalised to the test dose signal integrated over the 325–365 °C temperature range to account for sensitivity change across repeat measurement cycles. As previously reported (Stirling et al., 2012; Colarossi et al., 2024), three peaks are visible at ~100, 265 and 345 °C, commonly denoted as Peak 1, 2 and 3. Peak 1 appears to consist of more than a single peak, and has a thermal lifetime at room temperature of about an hour (Colarossi et al., 2024). It is therefore difficult, with this experimental design, to separate the loss of signal in Peak 1 due to detrapping at room temperature from that due to light exposure. The short lifetime of charge in this peak means that it is not of interest for dating, and so no further analysis of it is undertaken in this study. In contrast, Peaks 2 and 3 have lifetimes at 15 °C of ~75 Ma and 140 Ga (Stirling et al., 2012) and are the focus of this work. To assess the response of Peaks 2 and 3, the TL signals from 245 to 285 °C and from 325 to 365 °C have been summed for all L_x and T_x measurements (shown by the shaded pink and blue areas on Fig. 1a).

The sensitivity corrected signal (L_x/T_x) has been calculated for each peak after the different periods of SOL2 exposure. The L_x/T_x ratios have then been normalised by the average of the L_x/T_x ratios measured when no light exposure was undertaken (n = 7), so that any deviation from a value of one represents an increase or decrease in the peak intensity. The measurements with no light exposure were interspersed in between the

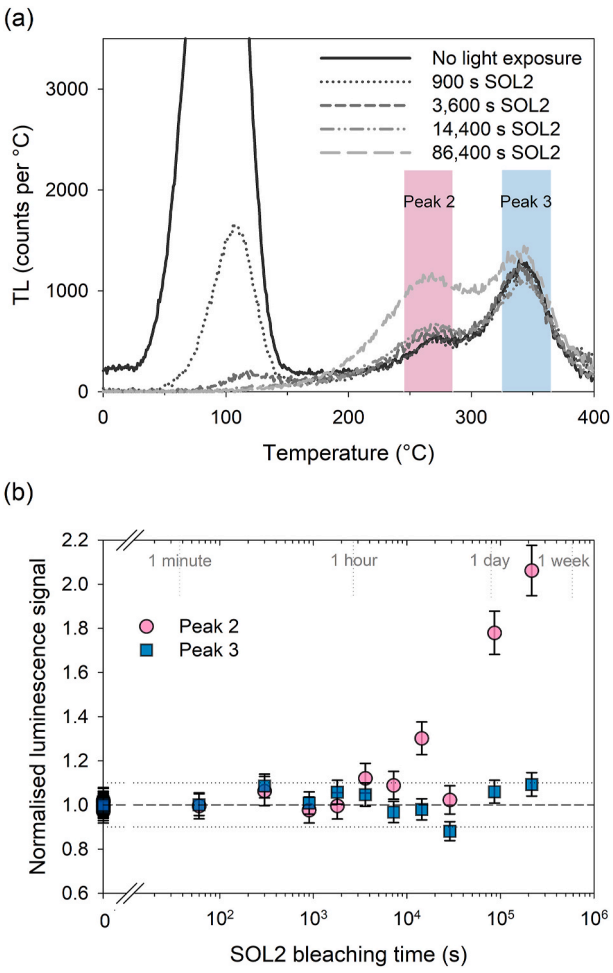

Fig. 1. (a) TL glow curves for one operculum from Purfleet. To avoid overcrowding the diagram, data are only shown for selected durations of exposure to the SOL2. (b) The sensitivity corrected (L_x/T_x) values for Peak 2 and Peak 3 after different durations of SOL2 exposure (datapoints represent the average ± SD of two opercula). The L_x/T_x ratios have been normalised to the average of the seven replicate values obtained after no light exposure. The horizontal dashed line shows unity (i.e. no change), and the dotted horizontal lines are at ratios of 1.1 and 0.9 denoting ±10% of unity.

Table 1
Experimental procedure for measuring the impact of light exposure on the TL signal from Peak 2 and Peak 3.

Step	Procedure	Signal
1	Beta irradiation (72 Gy)	
2	Light exposure for different durations (t_i)	
3	TL to 400 °C at 0.5 °C.s^{-1} with black body subtraction	L_x
4	Beta irradiation (43 Gy)	
5	TL to 400 °C at 0.5 °C.s^{-1} with black body subtraction	T_x
6	Repeat steps 1 to 5	

measurements following different SOL2 exposure times. No trend was observed in the L_x/T_x ratios of these seven measurements. Up to a period of 2000 s of SOL2 exposure, there is no discernible pattern for either Peak 2 or 3 (Fig. 1b), with normalised values remaining close to unity. For longer SOL2 exposures, up to 28,800 s (8 h), the size of Peak 2 (pink circles) starts to increase. There is some scatter in the individual datapoints, however for the longest SOL2 exposures of 86,400 s (24 h) or 216,000 s (60 h), a clear increase is seen by as much as a factor of ~2. In contrast, the signal from Peak 3 (blue squares) remains within 10% of unity for all of the SOL2 exposure periods measured, with no clear trend.

Two other measurements were made using the same opercula, and the same protocol (Table 1), but exploring the impact of different light sources. In the first experiment, the opercula were exposed to daylight behind window glass for 60 daylight hours. Peak 2 dropped to a normalised value of 0.75 ± 0.02, whilst Peak 3 had a normalised value of 0.92 ± 0.03. In the second experiment, the two opercula were placed in a glass vial and then put into the SOL2 solar simulator for a period of 14 h (50,400 s), giving normalised signals of 1.20 ± 0.04 for Peak 2 and 1.00 ± 0.05 for Peak 3. For these two exposures to light sources without any UV component, the change in Peak 2 is much less than that seen in Fig. 1b. For Peak 3 little change is seen, consistent with the pattern previously observed.

4. Can preheating before the TL measurement isolate a signal unaffected by light exposure?

The single aliquot regenerative dose (SAR) protocol used by Duller et al. (2015) for equivalent dose determination using opercula included a preheat at 320 °C to remove Peak 1 and 2, leaving only Peak 3. A second set of optical bleaching experiments were conducted to investigate the impact of light on Peak 3 following such a preheat, and to further explore the response to both broad-spectrum artificial light (SOL2) and natural light. The impact of an additional artificial light source was also tested in this experiment, utilising the sample camera mounted on the Risø TL/OSL reader, which uses a ring of white LEDs positioned around the camera to capture an image during the measurement protocol. During natural light exposure, the opercula were placed on an interior windowsill which ensured the removal of the UV component from the natural light when passing through the glass pane. The TL response from Peak 2 was subsequently removed by including a preheat treatment directly before the TL measurement (L_x and T_x, in Table 2). The preheat measurement to 320 °C was made using a heating rate of 5 °C.s^{-1} to force as much sensitivity change in the material as possible prior to measurement of the signal used for dating (Duller et al., 2015). The TL signals were integrated between 270 to 380 °C for all L_x and T_x measurements. To assess the impact of light exposure, the L_x/T_x ratios were normalised by the L_x/T_x value determined when no light exposure was undertaken; the ten measurements with no light exposure were made before (n = 4), during (n = 3) and after (n = 3) the experiment.

The TL glow curves obtained from a typical operculum after a range of different exposure durations to both artificial light sources and natural light filtered through a glass windowpane to remove the UV-component, are shown in Fig. 2a. In each case, the TL signals shown have been normalised to the test dose signal integrated over the 270–380 °C temperature range to account for sensitivity change across repeat measurement cycles. The TL signal from Peak 3 shows no change after exposure to the sample camera, or to 26 days behind the window glass. The change after 24 h (86,400 s) SOL2 exposure is limited, and it is only after 163 days exposure behind window glass that some reduction is seen.

Fig. 2b shows the normalised response of Peak 3 to different duration exposures using both the SOL2 (green triangles) and windowsill (pink squares), as well as to the instrument-mounted sample camera (orange inverted triangles). As observed in the previous experiment, the signal from Peak 3 remains within 10 % of unity for all of the SOL2 and windowsill exposure periods measured up to a period of 26 d (2,246,400 s), with no clear trend. It is only following the longest exposure of 163 d (14,079,600 s) that a clear decrease in Peak 3 is observed, equivalent to a 16 % reduction in the normalised TL signal intensity (Fig. 2b).

Table 2
Experimental procedure for measuring the impact of light exposure on the TL signal from Peak 3 only.

Step	Protocol	Signal
1	Beta irradiation (158 Gy)	
2	Light exposure for different durations (t_i)	
3	Preheat to 320 °C at 5 °C.s^{-1}	
4	TL to 400 °C at 0.5 °C.s^{-1} with black body subtraction	L_x
5	Beta irradiation (134 Gy)	
6	Preheat to 320 °C at 5 °C.s^{-1}	
7	TL to 400 °C at 0.5 °C.s^{-1} with black body subtraction	T_x
8	Repeat steps 1 to 7	

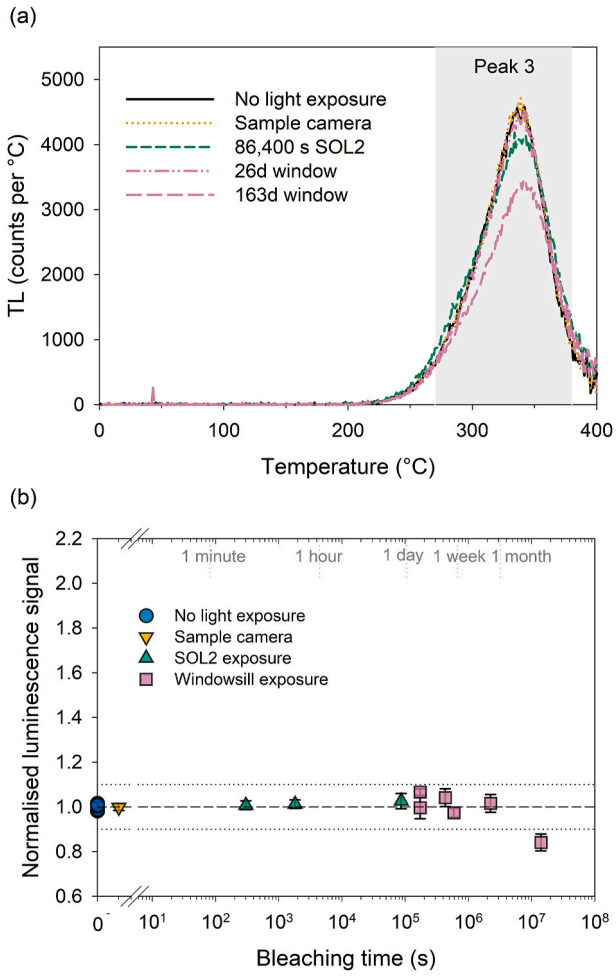

Fig. 2. (a) TL glow curves for one operculum from Purfleet measured after a 320 °C preheat. To avoid overcrowding the diagram, data are only shown for selected durations of exposure to the SOL2 and on the windowsill. (b) The sensitivity corrected (L_x/T_x) values for Peak 3 (following a preheat) after different durations of artificial (SOL2 and camera) and natural light exposure. Datapoints represent the average ± SD from 3 opercula. The L_x/T_x ratios have been normalised to the average of ten replicate values obtained after no light exposure. The horizontal dashed line shows unity (i.e. no change in signal following bleaching), and the dotted horizontal lines are at ratios of 1.1 and 0.9 denoting ±10% of unity.

5. Discussion

Previous studies have explored the impact of UV light on the TL signal of calcite, providing a useful comparison for this dataset. Working on crushed calcite crystals, Lima et al. (1990) concluded that UV irradiation altered the TL glow curve due to retrapping of charge by the same trap types, or phototransfer from deeper traps into shallower traps. Phototransfer resulting from light sources with a UV component has been reported previously for Peak 2 by Liritzis et al. (1996) and Bruce et al. (1999), albeit for different calcitic materials. It is possible that the rise in Peak 2 that we observe in opercula where long SOL2 exposures are used (Fig. 1b) may result from phototransfer, even though, like sunlight, the SOL2 has very little emission below ~340 nm. In contrast, Peak 3 (~350 °C) shows little detectable change for periods of SOL2 exposure of up to 60 h (Fig. 1b). The effect of phototransfer on Peak 2, and the lack thereof on Peak 3, can be seen clearly in the data from the 60 h (216,000 s) exposure measurement, with normalised luminescence ratios of 2.06 ± 0.11 and 1.09 ± 0.05 for Peak 2 and Peak 3 respectively (Fig. 1b). Based on this experimental data, it seems that the traps responsible for Peak 3 are not the source of the signal being transferred into Peak 2 following light exposure. This implies that there may be deeper traps contributing to the TL signal from calcite. In recent work Chithambo (2023) observed phototransfer in both Peak 2 and Peak 3 from calcite crystals after illumination with either a 405 nm UV laser or with 470 nm blue LEDs. However, that work with optical stimulation used a U-340 filter to isolate a UV emission from calcite, very different to the signal that is observed here. For the TL signal from opercula in this study, Peak 3 appears to offer significant advantages compared to Peak 2, being much less susceptible to light exposure and phototransfer than Peak 2.

In the second experiment in the current paper we included a preheat step to isolate Peak 3 directly prior to measuring the TL signal, but after the light exposure step. Inclusion of a preheat had no discernible impact upon the response of Peak 3 to light exposure (cf Figs. 1b and 2b); ratios of the normalised luminescence remaining from the 24 h (86,400 s) exposure excluding and including the preheat are the same within uncertainties, being 1.06 ± 0.05 and 1.04 ± 0.03, respectively. Exposure periods longer than 2 d were investigated using natural light behind window glass to remove the UV component from the light source. The response of Peak 3 remained negligible for exposure periods ranging from 2 d (172,800 s) to 26 d (2,246,400 s). Following 163 d (14,079,600 s) of exposure, the TL signal from Peak 3 was observed to have decreased by 16 %. This duration of exposure is equivalent to ~37 d in the SOL2 (assuming the intensity of the SOL2 is approximately 4.4 times that of sunlight), and the sensitivity to natural light or SOL2 exposure is minimal compared to the optically stimulated luminescence (OSL) signals that are more commonly used for luminescence dating (Fig. 3). For example, to achieve the same level of signal reduction for the OSL signal from sand-sized quartz grains would require ~1 s based on data from Colarossi et al. (2015). In contrast, for feldspar grains the post-infrared infrared stimulated luminescence signal measured at 225 °C ($pIRIR_{225}$), which is considered to be a slow bleaching luminescence signal, would be reduced by 15 % in less than 20 s in the SOL2 (Fig. 3).

In previous published studies which focused on dating speleothems and limestones, the TL peak at ~250 °C (Peak 2) was typically used for equivalent dose determination. This was due in large part to the linear dose response up to ~1 kGy reported by Aitken (1985, p.204). However, supralinear behaviour of Peak 2 was reported by Debenham and Aitken (1984), Berger and Marshall (1984) and Guibert et al. (2015). Whilst Peak 3 is considered to have a non-linear dose response in speleothems (Aitken, 1985, p.204), Stirling et al. (2012) showed Peak 3 to be more reliable for dating opercula in terms of the dose response to beta irradiation, as well as the ability to successfully recover a laboratory-given beta dose. Furthermore, Peak 3 also has a greater lifetime than Peak 2 (e.g. Wintle, 1974; Stirling et al., 2012), suggesting that Peak 3 would offer the greater maximum age for dating. The lack of response of Peak 3 to light exposure observed in this study offers another important advantage, supporting the recommendation of Stirling et al. (2012) to use the TL peak at ~350 °C (Peak 3) for dating opercula.

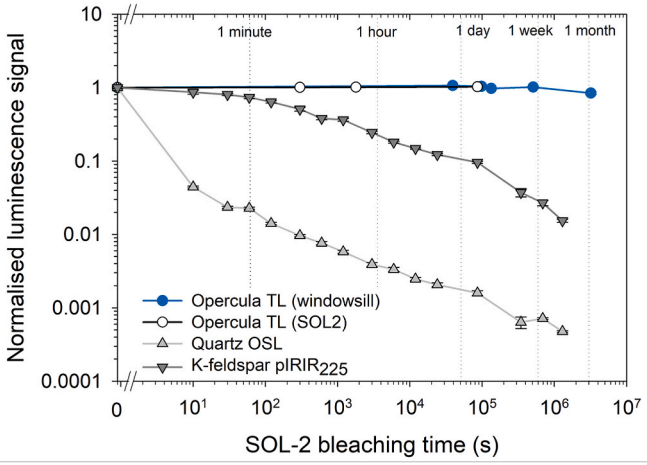

Fig. 3. The rate of optical bleaching of the TL signal from opercula due to artificial and natural light exposure (from this study) compared to the quartz OSL signal and K-feldspar $pIRIR_{225}$ signal (data from Colarossi et al., 2015). Each TL datapoint represents the average ± SD from 3 opercula, whilst each OSL and pIRIR datapoint represents the average ± SD of 3 coarse-grained aliquots. The exposure time to natural daylight has been scaled to account for the more powerful SOL2 light source using a factor of 4.4 as reported in Winzar et al. (2025). Where error bars on a datapoint are not visible they are obscured by the symbols.

6. Conclusions

The lack of impact of daylight exposure for periods of up to ~24 h means that collecting samples under natural daylight conditions would be expected to have no impact on equivalent doses whether they were measured using TL from Peak 2 or Peak 3. Peak 3 is insensitive to light for much longer times than Peak 2, showing little or no change in intensity up to at least 26 d exposure, and its relative insensitivity to natural light offers advantages for sample collection in the field, and for sieving and picking macrofossils in preparation for TL dating. Bulk sediment samples that may contain opercula but were collected previously without any precautions to exclude light or which have been stored in clear plastic bags should also be suitable for TL dating using Peak 3, provided that during storage there has not been prolonged periods of exposure (in excess of ~5 months) to direct sunlight. Similarly, museum specimens that have been stored in cupboards or drawers should be suitable for TL dating. Although it is possible to collect and prepare opercula samples in white light, excessively long exposure to UV should be avoided, and samples should be stored under dark conditions when possible as a precautionary measure. These findings support the possibility of using museum specimens for TL dating, provided that an associated sediment sample exists for the dosimetry calculations.

CRediT authorship contribution statement

D. Colarossi: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Conceptualization. **G.A.T. Duller:** Writing – review & editing, Writing – original draft, Visualization, Methodology, Funding acquisition, Formal analysis, Conceptualization. **H.M. Roberts:** Writing – review & editing, Methodology, Funding acquisition, Conceptualization. **R.J. Stirling:** Methodology, Investigation, Formal analysis. **K.E.H. Penkman:** Writing – review & editing, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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