Chapter 14

Optically stimulated luminescence (OSL) dating

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INTRODUCTION

A small programme of OSL dating was carried out at the site. Although we had some preconceptions that the probable age of the site was *c* MIS 11, it was important not to let this prior expectation over-ride independent chronometric investigation of the date. Furthermore, it was not known until fieldwork was substantially in progress how much other vertebrate and molluscan material (including *Bithynia* opercula) that could contribute to dating was present. Therefore, since the site was rich in sand/silt horizons sedimentologically suitable for OSL dating, a series of tube-samples for OSL dating was taken throughout the sequence (Table 14.1).

Later, once fieldwork and some preliminary faunal analysis had been completed, it was clear that the most significant archaeological horizon of the site, the Phase 6 clay, which included the elephant skeleton and the flint scatter south of Trench D, was reliably dated to MIS 11 by both independent chronometric means (amino acid dating, Chapter 13) and biostratigraphy (Chapter 9). Therefore, when it came to determining the post-excavation programme, there was evidently little benefit in devoting resources to carrying out OSL dating on sediments that: (a) were already confidently dated to MIS 11 and (b) given this, were in any case at the upper limit of the viability of the method.

Nonetheless, a small programme was carried out on some of the horizons at the top of the sequence, for which there was no other dating evidence. The highest level with molluscan and/or other biological remains allowing direct dating was the Phase 6 clay. Above this, the stratigraphic superposition of deposits of Phases 7, 8 and 9 established their relative ages, although it was uncertain whether this sequence was laid down in relatively quick succession following Phase 6, or whether it included significant chronological hiatuses. It was suspected on geo-morphological grounds that the Phase 8 gravels were probably of similar age to the Lower Middle Gravel at Barnfield Pit (ie also MIS 11). However there was no other evidence to support this (apart from, perhaps, the typology of the handaxes from the gravel (see Chapter 20), although dating deposits from their artefactual content should never be relied upon). Also, there was no evidence of a major stratigraphic unconformity between the Phase 8 gravel and the overlying brickearth of Phase 9, nor was there any indication of the date of the brickearth or of the likely passage of time between its deposition and the cessation of fluvial activity associated with the underlying Phase 8 gravels.

Therefore OSL dating was initially focused on the top of the sequence, with sand-rich beds from Phases 8 and 9 being analysed (see below). When these results proved surprisingly young, it was decided to carry out a further phase of analysis that: (a) investigated a sand-rich bed at the base of the Phase 8 gravels; and (b) analysed as a control a sand-rich sample from the base of the Phase 5 gravels, well below the Phase 6 deposits that are confidently attributed to MIS 11. As discussed in more detail below, this control analysis provided a dating result significantly younger than compatible with the Phase 6 dating evidence, leading to the suspicion that some of the other OSL results might also be questionable. Nonetheless this programme of OSL work is presented in full here, and serves as a useful and thought-provoking case-study, demonstrating that, when independently verified by other chronometric, biostratigraphic and geomorphological means, potentially misleading results have been obtained that would otherwise have been unsuspected. As discussed below, it is therefore perhaps now necessary to treat OSL dating results with more caution, and to seek additional independent and stratigraphic controls when applying the technique.

METHODS AND SAMPLING LOCATIONS

OSL has been well-established over at least the last 20 years as one of the main approaches to dating sediments younger than *c* 350,000 years, which is near the effective limit of the technique as currently practiced. Developed from the thermo-luminescence (TL) method of the 1990s (Wintle 1991), current OSL methods use a single aliquot regenerative dose (SAR) protocol as described by Murray and Wintle (Murray and Wintle 2000; Wintle and Murray 2006). In the simplest terms, the technique measures the time since sand grains were last exposed to daylight. It is thus particularly suitable for Quaternary sequences, where sand-rich beds are ubiquitous, and which often lack other means of dating.

Samples are taken in the field by hammering a light-opaque sampling tube of *c* 6-8cm diameter into a freshly cleaned face, then carefully removing this and covering the ends with light-opaque caps. A small additional sample of *c* 5-10cc is also taken for analysis of moisture content; this latter sample does not need to be kept away from light, but needs to be sealed with tape and double-bagged to avoid evaporation of natural soil moisture prior to analysis. Ideally, a measurement is then taken in the sample hole of the background radiation levels using a portable gamma-ray spectrometer; however, this step is not essential and the background dose rate can also be measured in the laboratory, extrapolating from the sediment forming the tube sample.

Sample preparation and optically stimulated luminescence measurements were performed at the Luminescence Dating Laboratory at the Research Laboratory for Archaeology and the History of Art, University of Oxford. The dating results are based on luminescence measurements of sand-sized quartz grains (180-255mm) extracted from the samples and mounted onto aluminium discs as small sized (3-4mm) multigrain aliquots and using the weighted mean of repeat measurements performed on multiple aliquots. All samples were measured in automated Risø luminescence readers (Bøtter-Jensen 1988 and 1997; Bøtter-Jensen *et al.* 2000) using a SAR post-IR blue OSL measurement protocol (Murray and Wintle 2000; Banerjee *et al.* 2001; Wintle and Murray 2006).

Dose rate calculations are based on the concentrations of radioactive elements (potassium, thorium and uranium) within the sediment. The beta dose rate was calculated from the concentrations of radioisotopes by fusion ICP-MS analyses and with the exception of sample X-1967, discussed below, the external gamma-dose rate was derived from the *in situ* radioactivity measurements. The final OSL age estimates include an additional 2% systematic error to account for uncertainties in source calibration. Dose rate calculations are based on Aitken (1985). These incorporated beta attenuation factors (Mejdahl 1979), dose rate conversion factors (Adamiec and Aitken 1998) and an absorption coefficient for the water content (Zimmerman 1971). The contribution of cosmic radiation to the total dose rate was calculated as a function of latitude, altitude, depth and average over-burden density based on data by Prescott and Hutton (1994).

INITIAL ANALYSES

The samples chosen for the initial phase of analysis were the highest three in the sampled sequence of deposits (Table 14.1). At the top of the sequence, sample <40254> (RLAHA lab code X-2060) came from the higher west end of Transect 2; one of the longitudinal strips cleared to investigate deposits to the north of the main site, where a handaxe and flint debitage scatter were found on the stripped surface of the brickearth bank (see Chapter 3). The sample came from a sand-rich bed in the upper part of the Phase 9 brickearth, close beside the location of the handaxe find (Fig. 14.1a). The second sample chosen for analysis was sample <40056> from context 40051 (RLAHA lab code X-1966), an undulating sand bed that occurred at the north end of section 40015, between the uppermost gravel bed of Phase 8 and the base of the brickearth (Fig. 14.1b). Although the upper and lower boundaries of this sand bed were sharply defined, there was no evidence that

Table 14.1 Southfleet Road OSL samples, in stratigraphic order

Phase	*Context*	*Trench*	*Section*	*Sample <>*	*Initial analysis (RLAHA lab code)*	*Further analysis (RLAHA lab code)*
9b	40087	Transect 2	Plan 40008	40254	X-2060	-
9a	40051	-	40015	40056	X-1966	-
8c	40049	-	40015	40057	X-1967	-
8b	40047	-	40015	40058	-	-
8a	40098	A	40066	40244	-	X-2056
8a	40098	A	40066	40245	-	-
8a	40045	-	40015	40243	-	-
6b	40144	-	40064	40247	-	-
6b	40070	-	40064	40246	-	-
6b	40070	-	40063	40255	-	-
5	40066	-	40016	40052	-	-
5	40066	-	40016	40053	-	X-1963
5	40066	-	40016	40054	-	-
5	40066	-	40016?	40358	-	-
5	40066	-	40016?	40359	-	-
5	40163	-	40086	40354	-	-
5	40163	-	40086	40355	-	-
5	40163	-	40086	40356	-	-
5	40163	-	40086	40357	-	-
2	40065	-	40016	40047	-	-
2	40065	-	40016	40048	-	-
2	40064	-	40016	40049	-	-
2	40060	-	40016	40051	-	-
1	40056	-	40016	40050	-	-

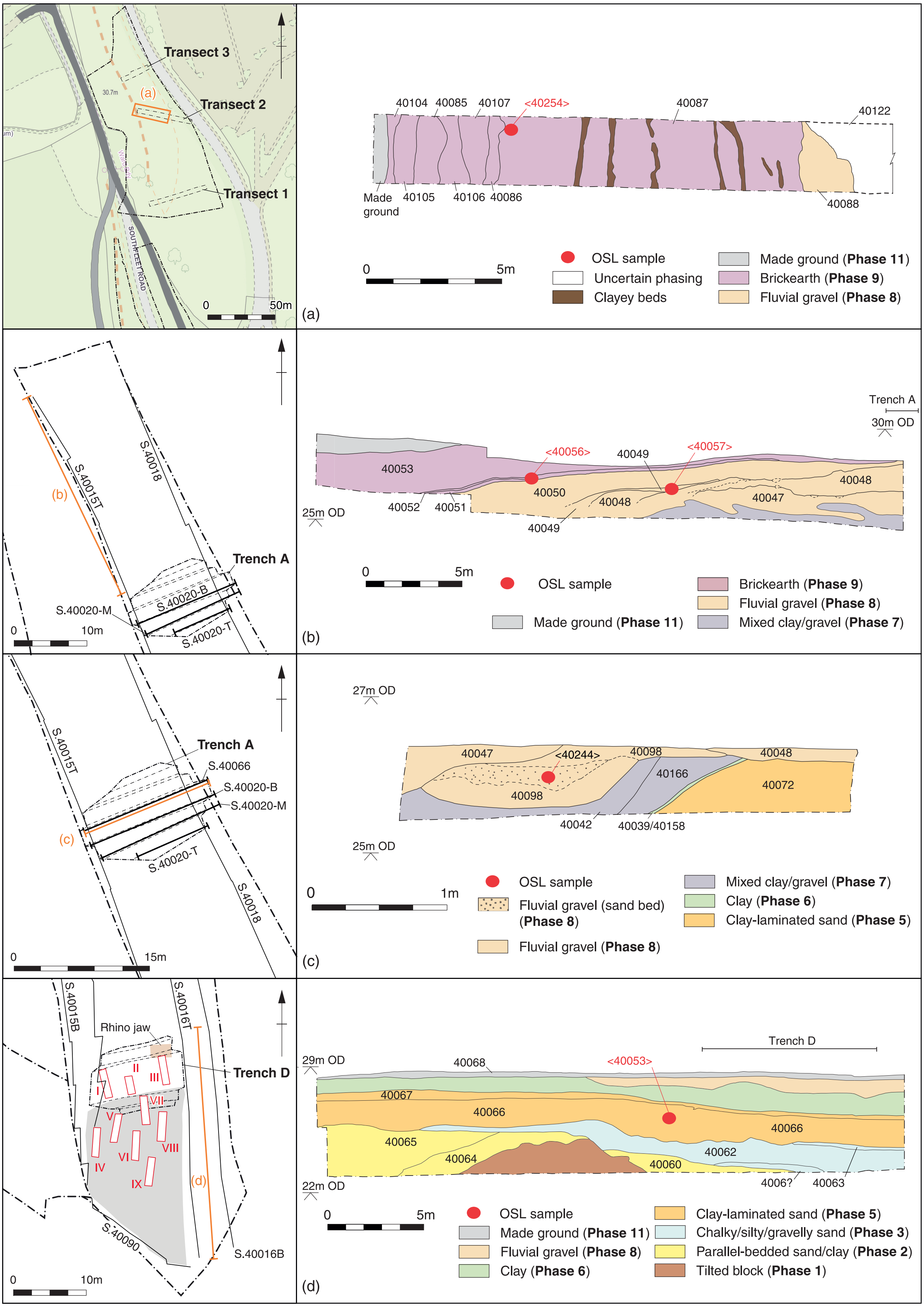

Figure 14.1 OSL sampling locations: (a) Transect 2, sample <40254>; (b) Section 40015, samples <40056> and <40057>; (c) Trench A, Section 40066, sample <40244>; (d) Section 40016, sample <40053>

either was associated with erosional truncation, so it was not initially thought that a major period of time was represented by the transition from Phase 8 to Phase 9. The third sample chosen at this stage came from slightly lower in the sequence in the same part of the site, and was sample <40057> from context 40049 (RLAHA lab code X-1967), which was a sand bed within the upper part of the Phase 8 gravels (Fig. 14.1b).

All samples were collected as described above, supported by collection of *in situ* readings of the background sedimentary radiation dose rate with a portable gamma-ray spectrometer.

The quartz OSL signal characteristics of all three samples were generally good featuring high signal intensity, negligible recuperation and good recycling ratios. Only a small number of aliquots showed a clear infrared stimulated signal (which indicates the presence of contaminant feldspar mineral grains) thereby confirming suitable sample preparation and purity of the quartz extract. The adoption of a post-IR blue OSL measurement protocol (Banerjee *et al.* 2001) in which each OSL measurement is preceded by an IR measurement in order to deplete the contribution of feldspathic minerals to the OSL signal further enabled to reduce the IR/OSL ratio to negligible levels.

Repeat measurements on the same sample showed that in the case of samples X-1966 and X-1967 there is a wide degree of scatter between aliquots with some clear outliers and this directly accounts for the rather large error of ~55ka obtained for sample X-1966. This variability may be attributed to problems of incomplete bleaching which commonly affect mineral grains from fluvial sediments. For this reason obvious high outliers were omitted from these calculations.

Although, this study only includes a limited set of three samples, the luminescence measurements seemed to provide a stratigraphically consistent set of results (Table 14.2). There is good agreement between samples X-1966 and X-1967 which provided OSL age estimates of respectively *c* 281 and 287 ka [ka = thousands of years ago]. If accepted as correct, these dates would place the gravel and overlying sand in MIS 8. The date calculated for X-2060 is substantially younger (*c* 58 ka) as a result of a reduced palaeodose and a higher environmental dose rate. This date would place the upper part of the brickearth in the later part of the Devensian, at the end of MIS 4 or the start of MIS 3.

Although the close agreement of samples X-1966 and X-1967 corresponds with the sedimentological observations suggesting no major depositional hiatus between the sampled deposits, the actual date suggested was substantially younger than the expected date of *c* 380,000-360,000 BP, even allowing for the margin of error accompanying the dating result.

Even more striking, was the dating result for X-2060 from the upper part of the brickearth, which was very much younger than anticipated. The dated sand-rich bed was definitely within the main body of the brickearth, which contained no evident sedimentary boundaries in the exposure seen, right down to its basal junction with gravel deposits that were thought to be equivalent to the top of the Phase 8 gravels.

The accuracy of this result should not, however, be ruled out. It is becoming increasingly well-established from numerous OSL investigations carried out recently in the Ebbsfleet Valley (Wenban-Smith and Bates 2011a; Wenban-Smith *et al.* forthcoming) and the Dartford area (Wessex Archaeology 2008c; Wenban-Smith *et al.* 2010; Wenban-Smith and Bates 2011b) that the Devensian glacial (taken as starting with the post-Ipswichian climatic deterioration of MIS 5d, *c* 115,000 BP) was a period of major colluvial/slopewash activity in north-west Kent, with repeated evidence of massive slope movement and redeposition of substantial bodies of sand/silt and brickearth. Even if the precision of this result is questionable, it still suggests that there is a major, unsuspected depositional hiatus between context 40051 (Phase 9a) and the main body of the overlying brickearth (Phase 9b). And it also seems possible that this brickearth may well be associated, at least in its upper part, with the Devensian, rather than MIS 11 or 10, as previously suspected.

FURTHER ANALYSES

Following from the results of the first phase of OSL analysis, two further samples were dated (Table 14.1). The first of these, sample <40244> (RLAHA lab code X-2056) came from the base of the Phase 8 gravels, from a substantial sand bed in context 40098 at the bottom of the south-facing section 40066 of Trench A (Fig. 14.1c). Even though the dating result from the upper part of the gravel was considered to be too young, the possibility that it was correct was not ruled out, and it was therefore decided to investigate further down the gravel sequence to see if a similar, or slightly earlier, result was obtained.

The second additional sample analysed, sample <40053> (RLAHA lab code X-1963), came from the Phase 5 clay-laminated sands in the southern part of the site in the main east-facing section 40016 (Fig. 14.1d).

Table 14.2 Southfleet Road OSL results: initial analyses

Phase	*Context*	*Sample* <>	*Lab code*	*Palaeodose(Gy)*	*Dose rate(Gy/ka)*	*Age estimate*(ka)
9b	40087	<40254>	X-2060	125.5 ± 23.2	2.15 ± 0.11	58.5 ± 11.3
9a	40051	<40056>	X-1966	304.7 ± 56.9	1.09 ± 0.07	281 ± 56
8c	40049	<40057>	X-1967	298.5 ± 26.5	1.04 ± 0.07	287 ± 33

This sand was stratigraphically sealed beneath the Phase 6 clay, which was securely dated to MIS 11 by amino acid racemization (Chapter 13) and on biostratigraphic grounds (Chapter 9). This sample therefore serves as a control for the application of OSL as a dating technique at the site, to establish the apparent dating result for a deposit that is firmly believed to be associated with early MIS 11, *c* 400,000 BP in age.

The quartz OSL signal characteristics of both samples were generally good, featuring high signal intensity, negligible recuperation and good recycling ratios. Some aliquots showed a small but clear infrared stimulated signal (>5%), indicating the presence of contaminant feldspar mineral grains. The adoption of a post-IR blue OSL measurement protocol (Banerjee *et al.* 2001) in which each OSL measurement is preceded by an IR measurement in order to deplete the contribution of feldspathic minerals to the OSL signal enabled reduction of the IR/OSL ratio to negligible levels (<3%).

Repeat measurements on the same sample revealed a wide degree of scatter between aliquots (especially for sample X-2056, from the base of the Phase 8 gravel) which can lead to rather large errors on the final dates. The presence of high outliers is often encountered in rapidly deposited fluvial sediments such as this, and this is attributed to incomplete bleaching of the OSL signal at the time of deposition. The same issue affected some of the samples in the first phase of analysis and clear outliers were omitted from the age calculations presented here (Table 14.3).

The results of both these additional analyses were incompatible with both site stratigraphy and the independent dating of Phase 6 to MIS 11. For sample X-2056 from the base of the Phase 8 gravel, the apparent age of *c* 211 ka is substantially younger than the previous result from towards the top of the gravel. Taking account of the (albeit substantial) error margins of these dates, the stratigraphically higher date has a range of 320-254 ka, and the stratigraphically lower one a range of 255-167 ka. If the gravel was very rapidly deposited, one could interpret the combined results as suggesting a date of *c* 255 ka, towards the end of MIS 8, based on the overlap of the error margin range. This date would still, however, the substantially younger than the MIS 11 date of *c* 380,000 BP expected on geological grounds.

For sample X-40053, the result of *c* 142 ka was completely at odds with all other dating information, and has worrying implications for the widespread and uncritical application of OSL dating, particularly in conjunction with the relatively narrow error margin of ± 18 years, suggesting a relatively accurate and precise result. The sample was taken in the field under optimum conditions, with *in situ* measurement of the sedimentary background radiation dose rate using a portable gamma ray spectrometer, and there was nothing in the laboratory analysis to indicate that the result was problematic. However, the apparent result would not only place the deposit in MIS 6, despite being securely sealed beneath the Phase 6 deposits that are securely dated to MIS 11, but also directly contradicts the other OSL dating results from the site (Table 14.2; Table 14.3). These (with the exception of X-2060 from the very top of the site sequence) have all provided older dates for stratigraphically higher deposits.

DISCUSSION AND CONCLUSIONS

The incompatibility of the additional results with: (a) previous results from higher in the stratigraphic sequence, (b) site stratigraphy and (c) independent dating evidence, demonstrates that many of the OSL dating results must be wrong. Since there is no means of judging which ones are least unreliable, they must therefore all be regarded as suspect – the only possible exception being the Devensian date of *c* 58 ka for X-2060, from the top of the Phase 9 brickearth, the highest natural deposit in the site sequence. This result not only matches the stratigraphic sequence, but it is also compatible with a substantial body of evidence from the region in and around Swanscombe: the Darent Valley (Wenban-Smith *et al.* 2010; Wenban-Smith and Bates 2011b); the Ebbsfleet Valley (Wenban-Smith and Bates 2011a; Wenban-Smith *et al.* forthcoming) and the Dartford area (Wessex Archaeology 2008c), for slope instability and substantial colluvial/slopewash redeposition throughout the Devensian from *c* 115,000 to 10,000 BP (MIS 5d-2). The quantity and consistency of these results lend them credence. They also tally with the likely depositional processes associated with this predominantly cold period and with available lithostratigraphic data, in that the proposed Devensian colluvial sediments are always near the top of the Quaternary sequence and never buried by deposits for which independent dating suggests an earlier age.

Besides this one Devensian date, the rest of the OSL results reported here are regarded as unreliable, and the other evidence of geomorphological position and lithostratigraphic relationships with deposits dated by other means is given greater credence when considering the age of the deposits for which OSL dating was attempted. The results suggest that, although we should always keep

Table 14.3 Southfleet Road OSL results: further analyses

Phase	*Context*	*Sample* <>	*Lab code*	*Palaeodose(Gy)*	*Dose rate(Gy/ka)*	*Age estimate*(ka)
8a	40098	<40244>	X-2056	271.06 ± 54.06	1.28 ± 0.07	211 ± 44
5	40066	<40053>	X-1963	221.75 ± 25.03	1.56 ± 0.07	142 ± 18

an open mind that our preconceptions may not be right, the Quaternary community in general should be cautious about uncritically accepting OSL dating results that are not independently supported and that significantly conflict with prior expectation. It should also be a priority for the OSL dating community to develop the technique further, perhaps with increased application of approaches such as single grain dating. Also, crucially, with further analytical controls which, especially when a date cannot be supported by independent means, provide better indications of when a particular dating result is reliable.