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WATER MEADOWS, SILBURY, WILTSHIRE
OPTICAL STIMULATED LUMINESCENCE DATING

SCIENTIFIC DATING REPORT

Helen Roberts

INTERVENTION
AND ANALYSIS

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SILBURY,
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OPTICALLY STIMULATED LUMINESCENCE DATING

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SUMMARY
Five optically stimulated luminescence (OSL) samples were taken from the palaeochannel deposits located in the Water Meadows south of Silbury Hill, Wiltshire. The depositional nature of the sediments sampled requires coarse (ie sand-sized) grains to be used for OSL dating, to ensure that the D_e distribution can be examined and hence that an assessment can be made of whether the sediments were incompletely bleached at deposition. However, the palaeochannel sediments were all heavily dominated by clay. The coarsest of the five OSL samples taken was therefore selected to investigate the feasibility of isolating sufficient pure coarse-grained quartz for OSL dating from these clay-rich samples. Only 0.02% of the initial very large [~1kg] initial sample mass was found to be coarse-grained quartz of between 90–250µm diameter. Nevertheless, the luminescence characteristics of this material indicated that this coarse-grained quartz was suitable for dating using small aliquots.

The coarse-grained quartz proved sufficiently sensitive to enable well-resolved dating using the Single-Aliquot Regenerative dose (SAR) measurement protocol applied to small multiple-grain single-aliquots. The final OSL age generated for sample 183/SR6 places the time of deposition for this material at 9770 ± 580 years ago (datum 2011), during the early Holocene.

Following the completion of the pilot analysis of Water Meadows sample 183/SR6 which is the subject of the main report, the remaining four OSL samples (183/SR1–4) were prepared and analysed in an additional phase of the project. Again, using small multiple-grain aliquots of quartz, the samples studied proved sufficiently sensitive and responsive to facilitate well-resolved dating using OSL. The final OSL ages generated for the samples (reported in the Addendum) show that samples 183/SR1–4 are all significantly younger than sample 183/SR6 (discussed in the main report), giving ages that are in chronostratigraphic order (within errors) and ranging from 1200 ± 60 years ago for the lowermost clay-rich unit (sample 183/SR4) to ~740 years ago for the three uppermost clay-rich units indicating a phase of rapid deposition.

CONTRIBUTORS
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CONTENTS

1 INTRODUCTION

This report describes the measurements and findings of an optically stimulated luminescence (OSL) dating study undertaken as part of an English Heritage project to evaluate the recently revealed Romano-British settlement in the fields south of Silbury Hill, Wiltshire. The OSL aspect of the project was undertaken to determine the age of the West Overton Formation (Evans *et al* 1993) by establishing a chronology for a large palaeochannel exposed in a section through the valley. In September 2010, samples were taken for dating using optically stimulated luminescence (OSL) from Trench 7 at the 'Water Meadow' site.

Figure 1: The 'Water Meadow' site, located south of the A4 and Silbury Hill; the fences delineate Trench #7 where OSL samples were taken

2 THE PRINCIPLES OF OPTICALLY STIMULATED LUMINESCENCE DATING

Optically stimulated luminescence (OSL) dating examines the time-dependent signal that arises from the exposure of naturally occurring minerals, typically quartz and feldspar, to ionising radiation in the natural environment. This dating technique can be applied directly to the mineral grains that make up sediment deposits, and here the event being dated is the last time the mineral grains were exposed to sunlight (ie the time the sediments were deposited and buried by further sediments). The technique relies upon the principle that any pre-existing luminescence signal contained in the sediment grains is lost on exposure to sunlight during transport, prior to deposition. Once the sediments are deposited and shielded from light exposure by the deposition of further sedimentary material, the luminescence signal re-accumulates over time through exposure to cosmic radiation, and to radiation from the decay of naturally occurring radioisotopes of uranium, thorium, and potassium located within the surrounding sediment. The luminescence signal is measured in the laboratory by stimulating small sub-samples, or aliquots, of prepared mineral grains with light – hence the term 'optically stimulated luminescence' or OSL. The size or intensity of the OSL signal observed in the laboratory is related to the time elapsed since the mineral grains were last exposed to sunlight. The OSL age is determined by calibrating the intensity of the OSL signal against known laboratory-administered radiation doses in order to determine how much radiation the sample was exposed to during burial (termed the equivalent dose, D_e). This value is divided by the radiation dose to which the sample was exposed each year since deposition and burial (termed the 'annual dose'), to give the OSL age (see Equation 1). Further details on OSL methods are given in Aitken (1998), and in reviews by Stokes (1999), Duller (2004), and various papers within the special issue of Boreas (2008, vol. 37) on luminescence dating, and the English Heritage Guidelines (Duller, 2008a).

Equation 1

$$\text{OSL age (years)} = \frac{\text{Equivalent Dose, } D_e \text{ (Grays)}}{\text{Annual dose (Grays per year)}}$$

(1 Gray = 1 Joule/kg)

In this study, the D_e was obtained using the Single-Aliquot Regenerative dose (SAR) measurement protocol (Murray and Wintle 2000), applied to coarse-grained quartz (ie grains > 90µm diameter). Working with quartz offers the advantage that it is not subject to anomalous fading, unlike some feldspars (eg Spooner 1994; Huntley and Lamothe 2001). The SAR protocol uses the response to a fixed test dose to correct for any change in luminescence sensitivity occurring in the sample during laboratory measurements (eg as a result of thermal pretreatments), with all of the measurements necessary for the determination of D_e being made on a single-aliquot. By measuring several aliquots, many independent determinations of D_e can therefore be obtained. Figure 2 illustrates how D_e is obtained from the SAR measurements made. Following measurement of the Natural luminescence intensity (denoted by the square symbol on the y-axis of Fig 2), the response (L_x) to a series of artificial radiation doses is measured, and normalised to the response (T_x) to a fixed test dose. A normalised dose-response or 'growth' curve can then be constructed by plotting the ratio L_x/T_x as a function of radiation dose. This enables the Natural luminescence intensity to be calibrated to these responses to a given laboratory radiation dose, thereby determining the laboratory equivalent dose, D_e.

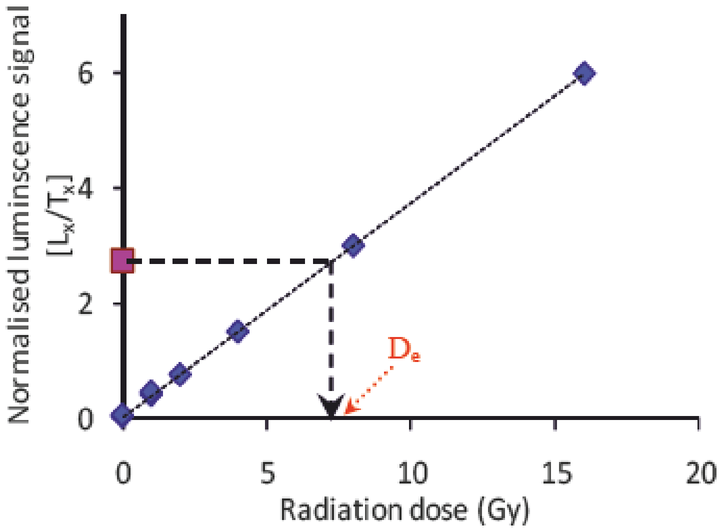

Figure 2: Normalised dose-response or 'growth' curve (diamond symbols) generated from measurements made using a Single-Aliquot Regenerative dose (SAR) measurement protocol. The Natural luminescence intensity (square symbol) of the aliquot is calibrated against the response to these known artificial irradiation doses to determine the laboratory equivalent dose, D_e

3 SAMPLE SITE AND OSL SAMPLE COLLECTION

In this project, OSL dating was to be used to date sediments from a palaeochannel running around the Water Meadows, adjacent to the Romano-British site at Silbury Hill. Fluvial sediments have been successfully dated at other sites around the world using the OSL signal from coarse-grained quartz (eg Rodnight *et al*, 2006; Rittenour, 2008), but key to this success is the ability to work on small aliquots of coarse-grained quartz which allows the question of incomplete bleaching of the OSL signal from sediments prior to deposition to be examined. Early discussions with English Heritage, and those that took place prior to the site visit for OSL sampling, emphasised the need to isolate sufficient coarse-grained (ie between 63µm and 300µm diameter) quartz for OSL dating in order to be able to investigate the equivalent dose ('D_e') distribution for the fluvial sediments studied. Although the main body of the stratigraphy was described as heavy clay texture, the English Heritage field team estimated that ~10% of the material within the silt and sand fractions was quartz, flint, and calcite.

Samples were taken for OSL analyses from two exposed sections of the Water Meadows trench. Due to concerns over the availability of sand-sized quartz within the palaeochannel sediments, two large samples (~1kg each) were taken using 50mm diameter opaque plastic pipe at each of the five OSL sampling locations (lab codes:

183/SR1–183/SR4, and 183/SR6). An additional, sixth sample (183/SR5) was taken for dosimetry measurements to support OSL sample 183/SR1 by characterising the change in stratigraphic unit within 0.3m of OSL sample 183/SR1. OSL samples were taken as far away as possible from any change in stratigraphic unit in order to minimise potential complications from any differences in dosimetry between adjacent units. However, due to the inhomogeneous nature of the sediments, and the fact that it was often not possible to ensure that OSL samples were taken from at least 0.3m distance (ie the gamma field) from a change in stratigraphic unit, field gamma spectrometry measurements were made at the point from which the OSL sample was taken to record the *in situ* dose-rate to the sample.

Four samples were taken as shown in Figures 3a and b, from each of four clay units exposed at the narrow northwest end of the Water Meadows excavation trench #7 (Aber-183/SR1–/SR4; see Table 1 for English Heritage-assigned excavation site codes) using two 250mm lengths of 50mm diameter opaque plastic pipe driven horizontally into each unit. One OSL sample (Aber-183/6; see Table 1 for English Heritage-assigned excavation site codes) was also taken from a clay-rich unit exposed on the southern-most long side of trench #7, as shown in Figures 4a and b.

Table 1: Aberystwyth OSL sample codes and English Heritage excavation site codes

Aberystwyth OSL sample number	EH Excavation site sample code	Excavation Site Fill Number
183/SR1	57007	97011
183/SR2	57008	97012
183/SR3	57009	97013
183/SR4	57010	97014
183/SR6	57006	97007

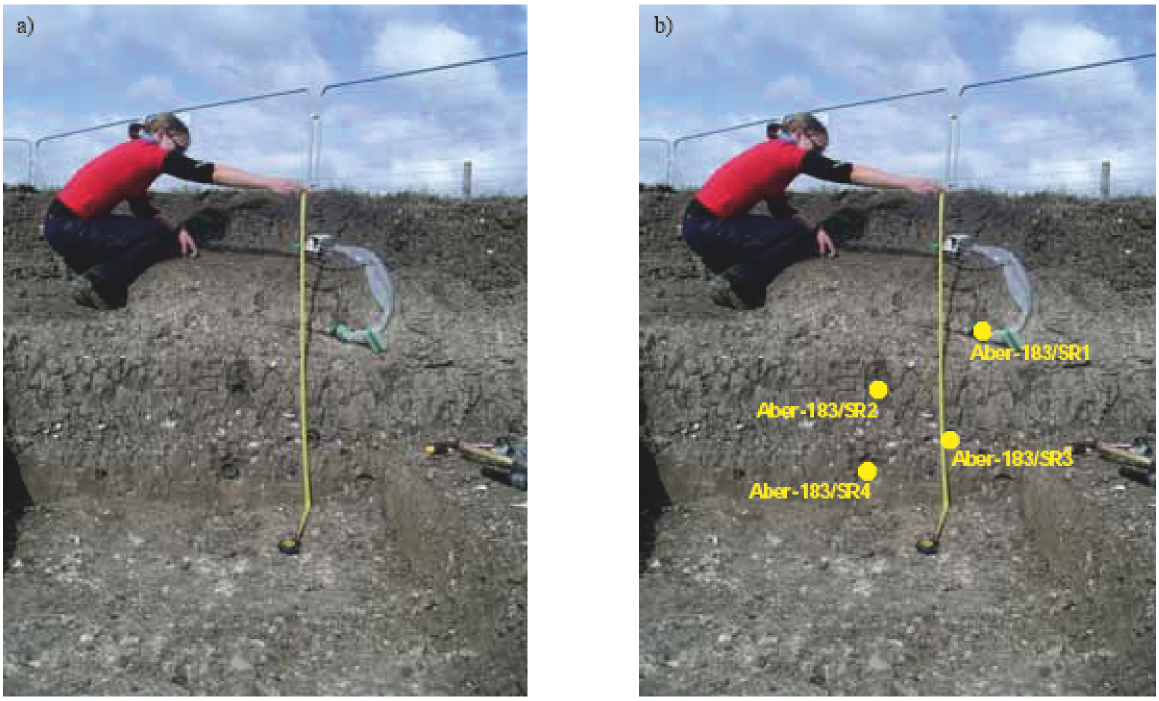

Figure 3: a) OSL sample locations marked by one 50mm diameter tube in each unit sampled in the narrow northwest end of Water Meadows Trench #7, and b) as for Fig. a but showing Aberystwyth Luminescence Research Laboratory OSL sample codes. See Table 1 for English Heritage excavation site codes

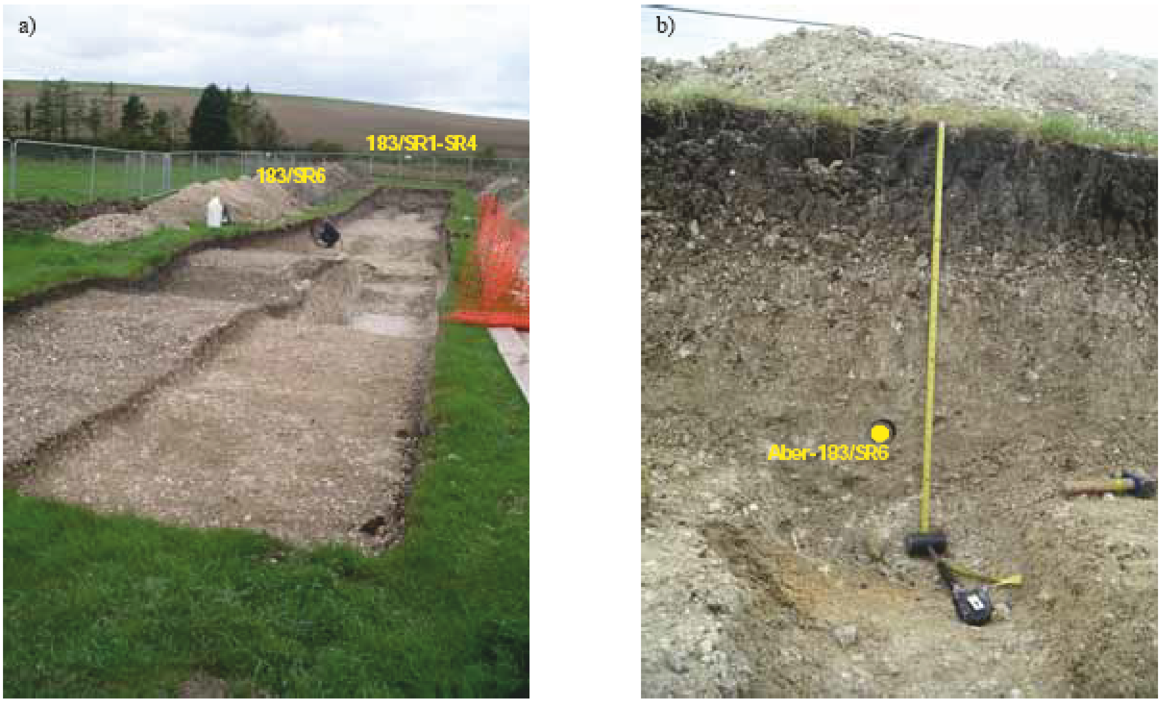

Figure 4: a) OSL sample locations within Water Meadows Trench #7, and b) the location of Aberystwyth Luminescence Research Laboratory OSL sample 183/SR6. See Table 1 for English Heritage excavation site codes

4 METHODOLOGY

4.1 Laboratory preparation and sample recoveries

Given the clay-rich nature of the sediments exposed and sampled in Water Meadows Trench #7, the decision was taken to open and process only one of the five OSL samples taken, monitoring the progress of that sample and using it as a guideline as to the likelihood of recovering sufficient pure sand-sized quartz required for an OSL study of the remaining palaeochannel samples. Sample 183/SR6 was selected for this pilot study because from field observations it appeared to contain the largest proportion of sand-sized grains. Sample material was prepared under subdued red lighting conditions in the luminescence laboratory using ~1kg of material which was excavated from one of the two plastic sample tubes taken at sample location 183/SR6. The outer 10mm of the sample at either end of the sample tube, which had been exposed to daylight during sampling and retrieval, was removed prior to the excavation of sample material for luminescence dating. Coarse-grained quartz was then prepared using the methods, outlined below.

The 1kg of sample 183/SR6 was divided between several beakers and pre-treated repeatedly (~ 50 times) with 5% sodium pyrophosphate solution to disperse and remove the clays which would otherwise impede later chemical treatments. When discarding the sodium pyrophosphate washings and the large volumes of suspended clays, great care was taken not to discard any coarse-grained material. Following this exhaustive pre-treatment stage, coarse clasts and pebbles were removed by hand, prior to the sample being wet-sieved to isolate the material between 90–355μm diameter. This 90–355μm diameter material (now ~30g mass, ie only 3% of the original sample mass) was then treated with a 10% vv dilution of concentrated (37%) hydrochloric acid (HCl) to remove carbonates and surficial coatings, then washed three times in distilled water. The sample was then treated with 20 vols hydrogen peroxide (H_2O_2) to remove organic material, and then washed as previously. The sample was dried and the remaining 2.2g was sieved using 90 and 250μm diameter mesh sizes, resulting in 0.8g in the 90–250μm fraction. This fraction was then refined using a solution of sodium polytungstate ('heavy liquid') to separate out the quartz material from the feldspar and heavy mineral fractions of the sediments, on the basis of differences in density. The 0.34g quartz-rich fraction of the sediments (density between 2.62–2.70gcm^{-3}), was treated with 40% hydrofluoric acid (HF) for 45 minutes, to remove the alpha-irradiated surface of the quartz grains and to dissolve any remaining feldspar material, followed by a further 45 minutes in concentrated (37%) HCl, to dissolve any fluorides formed during the etching procedure. The sample was rinsed a minimum of three times in distilled water, centrifuging between washings, and then dried at 50ºC, giving a final mass of 0.19g. Normally, the sample is then re-sieved which acts as a further quartz purification step, because it removes feldspar grains which have not been totally dissolved with HF, but which have been significantly etched and therefore reduced in diameter. However, due to the small amount of coarse-grained quartz within the sample (0.02% of the initial very large [~1kg] initial sample mass), this final re-sieving step was omitted and instead an internal check on the purity of the fraction prepared for dating was assessed using luminescence characteristics (discussed later, in section 5.2). The final coarse-grained quartz fraction for sample 183/SR6 was then ready for OSL measurements to determine the 'burial dose' or equivalent dose, D_e.

Given the low coarse-grained quartz recovery from this, the apparently most sand-rich of the clay samples collected, and given also the large amount of labour-intensive time (6 months) taken to prepare this pilot sample, it was decided not to open any further OSL samples taken from the site. Instead, sample 183/SR6 would be examined in detail to assess the feasibility of dating other palaeochannel deposit samples in the future using coarse-grain quartz OSL dating.

The light-exposed material removed from the end of each OSL sample tube was suitable for laboratory-based measurements of water content and dosimetry as these measurements do not require unexposed sample material. The light-exposed portion of each OSL sample was weighed, prior to drying at 50ºC. Drying continued until a constant mass was recorded, to establish the field water content at the time of sampling. These measurements of conditions at the time of sampling provide a benchmark for the water content values employed in the final age calculations (shown in Table 5). After drying, the light-exposed material was then crushed to a fine powder using a ball mill, prior to alpha and beta counting (see section 4.2).

4.2 Equipment and Methods

All OSL measurements were conducted using an automated *Risø* TL/OSL reader, equipped with a combined high-power blue LED (470nm)/ infra-red laser diode (830nm) OSL unit for stimulation of multiple-grain aliquots, and a green (532nm) focussed laser for stimulation of single-grains, and a ^{90}Sr/^{90}Y beta source for irradiations. All OSL measurements were made whilst holding the sample at 125°C, following a preheat of 220°C held for 10s or a cut heat of 160°C, and OSL was detected using 7.5mm Hoya U-340 filters.

Measurements of OSL were made on 90–250μm diameter coarse-grained quartz, using the Single-Aliquot Regenerative dose (SAR) protocol of Murray and Wintle (2000). The advantage of SAR over previous measurement protocols is that it uses a measurement of the luminescence production per unit dose to monitor and correct for changes in luminescence sensitivity that have occurred as a function of time, temperature, and past-radiation exposure (Wintle and Murray 2000). The SAR procedure permits the determination of an equivalent dose (D_e), and hence potentially an OSL age, for each aliquot examined. Multiple-grain measurements were made using 'small aliquots' of coarse-grained quartz (ie grains covering the central ~1mm of a 9.8mm diameter aluminium disc). Some single-grain OSL measurements were also made.

As part of the sequence of OSL measurements made, outlined in Table 2, a minimum of four regenerative beta doses were applied to each aliquot, bracketing the expected Natural dose. Two zero beta doses were also included towards the beginning and end of the measurement cycle to monitor recuperation, and a low regenerative dose was repeated at the end of the measurement protocol to monitor the sensitivity correction applied (referred to as monitoring of the 'recycling'). Following measurement of each Natural or regenerative-dose signal, a fixed test dose was applied, with a cut-heat of 160°C, to monitor and correct for sensitivity change during the measurement procedure. As a check on purity of the prepared sample material, the OSL-IR depletion ratio of Duller (2003) was employed at the end of each SAR measurement sequence (Table 2, steps 48–53).

Dose-rates were determined using a combination of *in situ* field- and laboratory-based techniques for OSL dating sample 183/SR6, and field gamma spectrometry measurements only for 183/SR1–4. In the field, a portable MicroNomad gamma detector fitted with a 50mm crystal was used to directly assess the dose-rate at the OSL sample location (section 3 and 5.6); the field-derived uranium, thorium, and potassium contents and field-derived total dose-rates are given in Table 4 (section 5.6). In the laboratory, dosimetry measurements were made on finely ground bulk sample material using both a *Risø GM-25-5* beta counter and a *Daybreak Thick Source Alpha Counter*. Thick source alpha counting served as an internal cross-check on the laboratory dosimetry measurements, but was not used in the final calculation of dose-rates and is therefore not given in this report. The final dose-rates were calculated using thick source beta counting to determine the beta dose-rate, and field gamma spectrometry to determine the gamma dose-rate to the sample (Table 5). The cosmic ray dose was estimated from the sample burial depth (Prescott and Hutton 1994). Water contents were determined in the laboratory from sealed field samples (section 3), and the values employed in the calculation of ages are presented in Table 5 (section 6). Moisture and beta attenuation factors are given in Aitken (1985). The beta and gamma counting results, cosmic dose-rates, water content values, and the dose-rates calculated using the conversion factors of Adamiec and Aitken (1998), are given for each sample in the final age table (Table 5, section 6).

Table 2: Outline of the SAR measurement protocol applied to each aliquot in this study. A minimum of four regenerative doses were employed in this study, designed to characterise the dose-response curve and bracket the Natural signal

Step Number	SAR sequence description
1	Preheat: 220°C, heating rate 5°C/s, hold at temperature for 10s
2	Measure Natural or regenerative dose signal ('L_x'): 100s OSL @125°C
3	Apply Test Dose
4	Cut heat: 160°C, heating rate 5°C/s
5	Measure test dose signal ('T_x'):100s OSL @125°C
6	Apply 0Gy dose ('recuperation' check)
7–11	*Repeat steps 1–5*
12	Apply regenerative dose 1
13–17	*Repeat steps 1–5*
18	Apply regenerative dose 2 (larger than dose 1)
19–23	*Repeat steps 1–5*
24	Apply regenerative dose 3 (larger than dose 2)
25–9	*Repeat steps 1–5*
30	Apply regenerative dose 4 (larger than dose 3)
31–5	*Repeat steps 1–5*
36	Apply 0Gy dose ('recuperation' check)
37–41	*Repeat steps 1–5*
42	Repeat a low regenerative dose ('recycling' test)
43–7	*Repeat steps 1–5*
48	Repeat the low regenerative dose used in the 'recycling' test)
49–53	*Repeat steps 1–5, but adding 100s IRSL @ 50°C prior to step 2 ('OSL-IR depletion ratio')*

5 RESULTS

As part of the OSL measurements made in this project, a series of tests were undertaken to monitor the OSL measurement procedure, the response and behaviour of the samples, plus the choice of aliquot size. These experimental checks are discussed below.

5.1 Aliquot size

Prepared quartz grains for sample 183/SR6 were presented for OSL measurements by mounting the grains in a monolayer onto 9.8mm diameter aluminium discs, sprayed lightly with Silkospray™ silicone oil to hold the grains in place during measurement. The discs, or 'aliquots', may be prepared using varying amounts of sample. In this study, initial tests showed that signal levels were sufficiently bright to allow examination of 'small' aliquots for OSL dating; this offers the advantage that the degree of scatter in the data can be examined which is important for these palaeochannel samples where incomplete bleaching prior to deposition could lead to the determination of an erroneously old age if not detected (eg if variations in equivalent dose [D_e] values are masked by averaging the OSL signal from a large number of grains).

To confirm the typical number of grains per small multiple-grain aliquot which contributes to the luminescence signal measured, 400 single-grains of sample 183/SR6 were also examined. Typically, only 2–5% of quartz grains from any given sample examined will give a measurable OSL signal (Duller, 2008b); similar values were observed for the sample in this study, 183/SR6. Figure 5 shows the cumulative net OSL light sum versus proportion of grains for the Natural OSL signal, based on data from 400 single-grain measurements of sample 183/SR6, and demonstrates that the majority of light arises from a small number of grains (eg 1% of grains give rise to 56% of the total light sum; 2% of grains give 65% of light; 5% of grains give 77% of light); the remaining grains are very dim. Figure 6 shows the net signal arising from the OSL response to a test dose following the Natural (Tn) versus the percentage of brightest grains, and demonstrates that a maximum of 5% of grains for sample 183/SR6 give a usable OSL signal. On this basis, small (~1mm diameter) multiple-grain aliquots are found to be appropriate for dating measurements for palaeochannel sample 183/SR6 because for any given small multiple-grain aliquot the light signal is likely to arise from only 1 or 2 grains and hence such small multiple-grain measurements are essentially equivalent to single-grain data and thus capable of capturing the true D_e distribution of the sediments examined.

Small multiple-grain aliquots (~1mm diameter, containing ~20–100 grains per aliquot depending on the grain size from 250 to 90μm respectively; Duller, 2008b) were therefore examined throughout the remainder of this study.

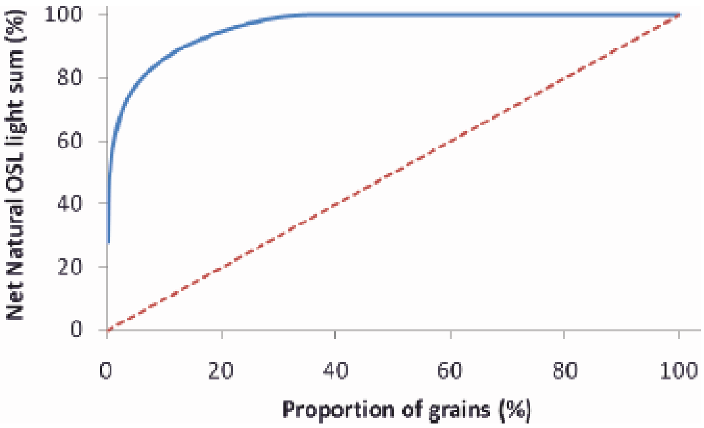

Figure 5: Cumulative net OSL light sum versus proportion of grains for the Natural OSL signal, based on data from 400 measurements of sample 183/SR6 (solid blue line). The dashed red line indicates the situation if all grains were to contribute equally to the total light sum

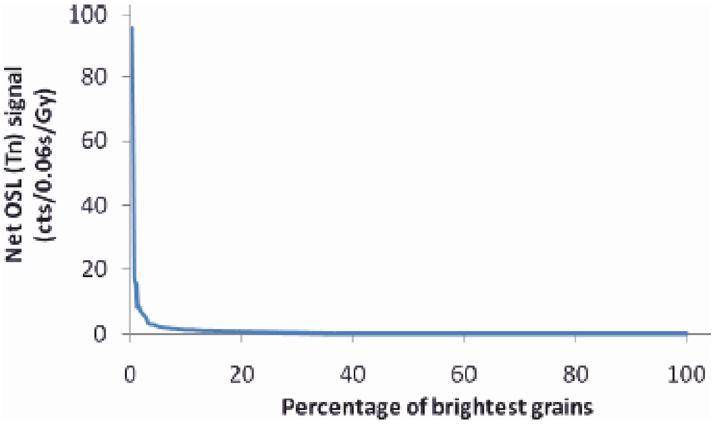

Figure 6: Net signal arising from the OSL response to a test dose following the Natural (Tn), expressed as counts per 0.06s integral per Gy, versus the percentage of brightest grains for sample 183/SR6

5.2 OSL signal checks

The OSL signal of each aliquot measured was examined visually, to check the initial signal intensity and the form of the decay curve. A typical decay curve is shown in Figure 7, and shows a rapid decrease in signal which quickly reaches a steady low background value; this is characteristic of the decay of a signal from quartz. Routinely, the D_e values were calculated using the first two data channels (0.32s stimulation) and the background was taken from the end of the decay curve (channels 230–250, the final 3.36s stimulation). This maximised the contribution of the fast component of the OSL signal (Bailey *et al* 1997; Murray and Wintle 2003), and typically represented ~15% of the total OSL signal.

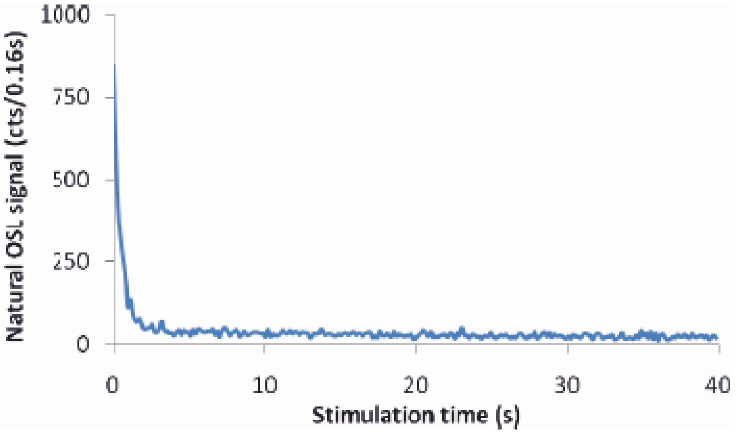

Figure 7: Typical OSL signal for small multiple-grain aliquots of sample 183/SR6

The form of the dose-response or 'growth' curve was also examined for sample 183/SR6, and a minimum of four artificial irradiation doses were used to define the growth curve for each aliquot, designed to bracket the 'Natural' signal and hence determine the value of D_e. Figure 8 shows a typical dose-response curve; error bars are shown, calculated following Banerjee *et al* (2000) and Galbraith (2002), and generated by *Analyst* (written by Prof. Geoff Duller, Aberystwyth University).

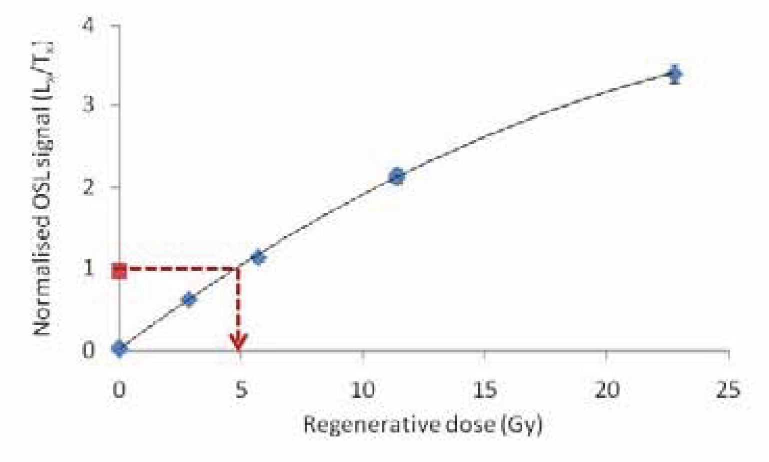

Figure 8: Typical dose-response curve constructed for aliquots of 183/SR6 (blue diamond symbols), and the Natural normalised OSL signal (red square symbol) used to determine the laboratory equivalent dose (D_e, Gy). Recycling points, repeat recuperation points, and errors bars are shown; where they cannot be seen, these lie within the symbols shown

Once the sequence of dating measurements was completed, each aliquot was irradiated and then stimulated using infrared (IR) laser-diodes at a temperature of 125°C to check the purity of each aliquot (steps 48–53, Table 2). Stimulation with IR was proposed as a check on the purity of prepared quartz material by Stokes (1992). Feldspathic minerals respond to simulation with IR, giving a rapidly decaying signal, however, quartz does not appear to respond to stimulation with IR (Spooner and Questiaux 1989). There was no evidence of any response above background signal levels to stimulation with IR for any aliquot in this study (a typical IR stimulated luminescence signal response is shown in Fig 9). The OSL-IR depletion ratio (Duller, 2003) supported this observation, giving values within ± 5% of unity. No feldspar contamination was therefore considered to be present in the quartz separate prepared for sample 183/SR6.

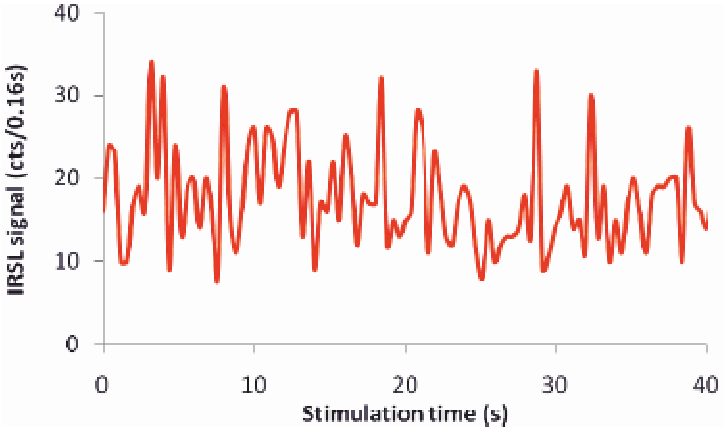

Figure 9: Typical response of sample 183/SR6 to stimulation with infra-red (IR). The IRSL signal level is very low, being approximately at background levels, thereby suggesting that no feldspar is present in the quartz fraction prepared for OSL dating

5.3 Recovery of a known laboratory irradiation dose

An important test of any luminescence dating protocol employed is whether the value of a previously delivered laboratory irradiation dose can be accurately and precisely determined. This is sometimes referred to as a 'dose-recovery' test and should be conducted on material which has not previously received any thermal pre-treatments. This fundamental test was conducted for sample 183/SR6 using two small multiple-grain aliquots of unheated material.

The laboratory beta dose chosen for the dose-recovery experiment was 4.74 Gy, which is of a similar magnitude to the Natural D_e value. The Natural OSL signal was removed from both aliquots by 2 × 1000s stimulation with blue diodes at room temperature, with a 10,000s pause between each stimulation; the 4.74 Gy beta dose was then applied and treated as an unknown dose. The SAR protocol was then applied using regeneration and test dose values of the same size as used in the dating measurement sequences, and applying a preheat of between 220ºC for 10s, and a cut heat of 160°C as used for dating measurements.

The beta dose recovered for both aliquots of sample 183/SR6 is shown numerically in Table 3. In both cases, the ratio of the beta dose recovered to the dose given is within ±5% of unity. The SAR measurement protocol and preheat conditions used for dating (Table 2) are therefore appropriate and working well for the sample material 183/SR6.

Table 3: Recovery of a known beta dose for two aliquots prepared from sample 183/SR6

Sample 183/SR6	Dose given (Gy)	Dose recovered (Gy)	Dose recovered Dose given
Aliquot 1	4.74	4.72 ± 0.16	1.00 ± 0.03
Aliquot 2	4.74	4.74 ± 0.20	1.00 ± 0.04

5.4 OSL dating measurements and checks

The SAR measurement sequence employed in this study has several checks built into it to monitor the behaviour of the sample and the efficacy of the sensitivity correction. For sample 183/SR6, 48 multiple-grain single-aliquots were measured to establish D_e values for use in determining an OSL age. The advantage of working with the single-aliquot methods used in this study, rather than the multiple-aliquot methods available, is that each of the 48 aliquots measured gives rise to an independent assessment of D_e, and hence, potentially to an OSL age.

One of the most powerful tests used to evaluate the behaviour and reliability of the aliquots used for dating arises from the use of the SAR protocol for the OSL dating measurements. In this measurement procedure, the Natural luminescence signal is measured, followed by the response to a series of artificial laboratory beta doses of increasing magnitude designed to bracket the intensity of the Natural signal (Table 2). In the SAR measurements made in this study, a low irradiation dose was repeated, or recycled, and applied at the end of the measurement cycle for all aliquots to test how well the sensitivity correction procedure is working. If the sensitivity correction is adequate, then the ratio of the signal arising from this repeated regenerative dose at the end of the measurement sequence to that of its earlier regeneration dose (eg Table 2) should fall within the range of 1.0 ± 0.1 (Murray and Wintle 2000). All 48 aliquots examined for OSL dating passed this 'recycling test', indicating that the sensitivity correction in the SAR measurement procedure is working well for these samples in monitoring and correcting for changes in luminescence sensitivity that may have occurred as a function of time, temperature, and past-radiation exposure.

A further test of the reliability of the sensitivity corrected growth curve generated using the SAR measurement protocol is a check on the 'recuperation' of signal (Murray and Wintle 2000) following the application of a regeneration dose of 0 Gy at both the beginning (following measurement of the Natural signal) and towards the end of the measurement cycle (following the largest regeneration dose and prior to the application of the recycling regeneration dose) (Table 2). No significant net OSL signal should be observed following this 0 Gy beta dose if the sensitivity correction is working correctly. For sample 183/SR6, only 1 of the 48 multiple-grain single-aliquots examined showed any recuperation in OSL signal; this aliquot was therefore omitted from the final age determination. The other 47 aliquots measured for dating do not exhibit signs of recuperation of signal, suggesting that thermal transfer of charge from optically insensitive traps into OSL traps is not a factor in this study.

5.5 Determination of the equivalent dose for use in the final OSL age calculation

The small, multiple-grain single-aliquots on which OSL dating measurements were conducted were screened for their suitability for use in the final age equation using the series of tests described and discussed above. These checks included examination of signal intensity levels, decay curve shape, dose-response curve shape, recycling ratio, recuperation, and feldspar contamination checks using IR stimulation. One of the 48 small aliquots measured for dating was rejected on the basis of recuperation of signal (discussed in section 5.4), and 12 small aliquots were rejected on the basis of low signal levels, causing errors on the test dose and D_e to exceed 10%. The number of acceptable aliquots combined to determine a final OSL age for sample 183/SR6 was 35.

The D_e values of the aliquots accepted following screening are shown in Figure 10. The probability density plot demonstrates that the D_e values are essentially normally distributed with a slight tail to larger D_e values. The aliquots which were accepted following all the screening tests (Fig 10) are also shown in Figure 11 replotted as radial plots (Galbraith 1990), with the D_e of each aliquot being shown as a single point on the plot. These plots are presented as a visual aid to the data only, and displaying the data on radial plots offers the advantage of showing the precision to which each data point is known. The precision is displayed on the x-axis, with data of high precision being plotted towards the right hand side of the plot. The y-axis shows the number of standard deviations away from a central value for each D_e value, whilst the radial scale displays the D_e value. The horizontal line extending from 0 on the y-axis denotes the weighted mean D_e calculated for the sample. The grey shaded bar extending from the y-axis to the radial scale in Figure 11 is placed at two standard deviations, and any points falling within these limits (indicated by filled circles) therefore lie within two standard deviations of the weighted mean D_e value. Ideally, the data for all aliquots will fall within this band indicated on the diagrams, indicating one population of D_e values. The over-dispersion value is 22%. The data show very little scatter in the distribution of D_e values obtained following screening, suggesting only one population of D_e values for sample 183/SR6. The weighted mean of these D_e values was therefore taken for calculation of the final OSL age. The error on each determination of D_e was calculated using the standard error (ie the standard deviation divided by the square root of the number of estimates of D_e). The D_e and standard error are given in the final OSL age table (Table 5).

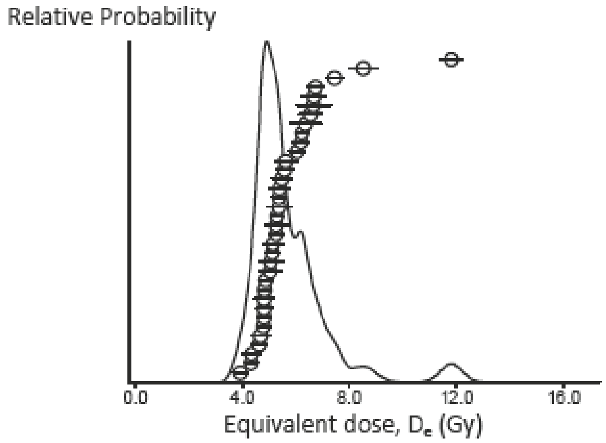

Figure 10: The distribution of D_e values obtained for sample Aber-83/SR6. Each of the 35 points shown is from an individual aliquot, which is plotted with the associated error

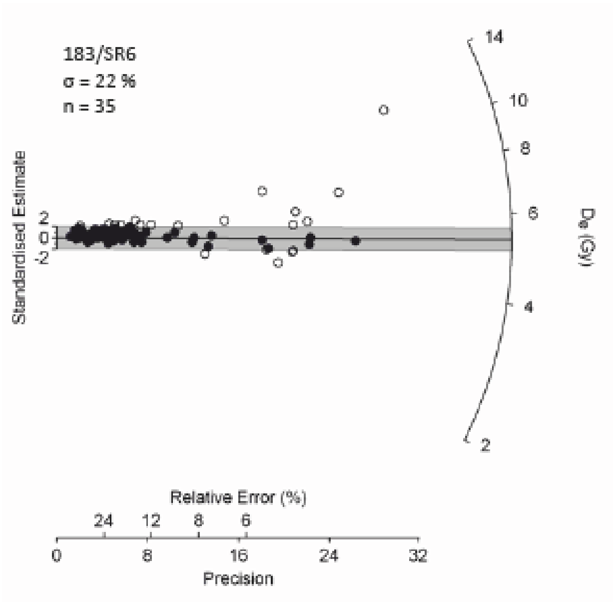

Figure 11: Radial plot to show the distribution of equivalent dose (D_e) values used for the determination of OSL ages for sample Aber-183/SR6. Each data point represents one aliquot which has passed the screening tests; closed symbols indicate those data points falling within 2σ error of the weighted mean D_e value, whilst open symbols are used for data points beyond 2σ error

5.6 Dosimetry data

The uranium, thorium, and potassium contents and total dose-rates based on *in situ* field gamma spectrometry measurements at the site of OSL samples 183/SR1–4 and 183/SR6 are given in Table 4. Thick source alpha counting served as a cross-check on the laboratory dosimetry measurements. The final dose-rates used to determine the age of sample 183/SR6 were calculated using thick source beta counting to determine the beta dose-rate, and field gamma spectrometry to determine the gamma dose-rate to the sample, and are shown in Table 5.

Table 4: Field gamma spectrometry (FGS)-derived potassium (K), uranium (U), and thorium (Th) contents and FGS-derived total dose-rates

Sample Name	K (%)	U (ppm)	Th (ppm)	Total FGS-derived dose-rate (Gy/ka)
183/SR1	0.52 ± 0.02	0.98 ± 0.04	4.63 ± 0.20	1.33 ± 0.02
183/SR2	0.65 ± 0.03	1.11 ± 0.04	5.27 ± 0.21	1.43 ± 0.03
183/SR3	0.53 ± 0.03	0.93 ± 0.05	4.46 ± 0.23	1.24 ± 0.03
183/SR4	0.41 ± 0.02	0.94 ± 0.04	3.51 ± 0.16	1.04 ± 0.02
183/SR6	0.17 ± 0.01	0.49 ± 0.04	1.24 ± 0.09	0.53 ± 0.01

6 OSL AGE DETERMINATIONS

The equivalent dose (D_e) data (discussed in section 5.5) and the results of laboratory dosimetry measurements (discussed in section 5.6) were combined for each sample, with corrections being made for attenuation by water and for grain size, to give an OSL age for sample 183/SR6 of 9770 ± 580 years (datum 2011, see Table 5). These data, including the final age determinations, are presented in detail in Table 5. The error shown for the D_e determination (Table 5) is the standard error (see section 5.5) (ie the standard deviation divided by the square root of the number of independent estimates of D_e). The percentage error on the OSL age is relatively small, being 6%.

Table 5: OSL sample details, equivalent dose and dose-rate data, and OSL ages

'Later Silbury', Trench #7 'Water Meadows'	
Aberystwyth Lab. number	183/SR6
EH excavation codes	*Sample 57006 in fill 97007*
Depth down-section (m)	0.80 ± 0.02
Material used for dating	quartz
Grain size (µm)	90-250
Preparation method	Heavy liquid separation (sodium polytungstate); 40% HF etch 45 mins
Measurement protocol	SAR; small multiple-grain aliquots; OSL 470nm; detection filter 7.5mm Hoya U-340
Aliquots measured	48
Aliquots used for D_e	35
Equivalent Dose, De (Gy)*	5.39 ± 0.20
Water content (% dry mass)	20 ± 5
U (ppm)	0.49 ± 0.04
Th (ppm)	1.24 ± 0.09
K (%)	0.17 ± 0.01
Layer removed by etching (µm)	10 ± 2
Infinite β dose-rate (Gy/ka)	0.263 ± 0.011
External β dose-rate 'wet' (Gy/ka)	0.210 ± 0.014
External β dose-rate 'wet' (Gy/ka)	0.151 ± 0.010
Cosmic (Gy/ka)	0.190 ± 0.019
Total dose-rate (Gy/ka)	0.55 ± 0.03
OSL Age (a)#	9770 ± 580

Ages are expressed as years before AD 2011, rounded to the nearest 10 years. All calculations were performed before rounding.

* The error shown is the standard error on the mean.

7 SUMMARY AND CONCLUSIONS

Five OSL samples were taken from the palaeochannel deposits exposed within Trench #7, located in the Water Meadows south of Silbury Hill (samples 183/SR1–4, and 183/SR6). The depositional nature of the sediments sampled requires coarse (ie sand-sized) grains to be used for OSL dating, to ensure that the D_e distribution can be examined and hence that an assessment of incomplete bleaching of the sediments prior to deposition can be made. However, the palaeochannel sediments were all dominated by heavy clay. Large samples were therefore taken for this OSL study, and the coarsest sample (183/SR6) was selected to explore the potential for isolating sufficient coarse-grain quartz for OSL dating. The luminescence characteristics of this material were assessed and found to be suitable for OSL dating. Through single-grain measurements, an assessment of the number of grains giving light was made and found to be in keeping with other typical sediments from around the world, with the majority of light arising from only 2-5% of the grains measured. For this reason, small (~1mm diameter) multiple-grain aliquots of quartz were found to be the most appropriate scale for dating using a Single-Aliquot Regenerative (SAR) dose protocol, as only 1–2 grains per aliquot were likely to give light.

Several checks and screening criteria were applied to the small multiple-grain aliquots used for OSL dating, and also to additional aliquots prepared from the sample, to ensure that the data included in the final age calculation were of the highest quality. The SAR measurement protocol was appropriate for the sample studied, and the sensitivity correction worked well. Using small multiple-grain aliquots, the sample studied proved sufficiently sensitive and responsive to facilitate well-resolved dating using OSL. The final OSL age generated for sample 183/SR6 places the time of deposition for this material at 9770 ± 580 years ago (datum 2011), during the early Holocene.

Sample 183/SR6 was assessed in the field as being the coarsest of the five OSL samples taken, however coarse-grained quartz of between 90–250µm diameter still accounts for only 0.02% of the very large [~1kg] initial sample mass and sample preparation was therefore extremely time-consuming. Nevertheless, the luminescence characteristics of sample 183/SR6 indicate that the quartz is suitable for dating using small aliquots prepared from these coarse-grains; this suggests that the remaining four OSL samples are also potentially suitable for dating using OSL provided that sufficient pure sand-sized quartz can be isolated.

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ADDENDUM

A1. Second phase of OSL analysis

Following the completion of the pilot analysis of Water Meadows sample 183/SR6 (see main report), English Heritage decided to proceed with the preparation and analysis of the four remaining OSL samples, 183/SR1–4. These samples were prepared, measured, and analysed in the same manner as sample 183/SR6, described in the main report, with the only difference being that unlike sample 183/SR6, the mass of prepared quartz for samples 183/SR1-4 was sufficiently large to permit re-sieving of the quartz grains after HF etching. Between 1.3–2.1 g of material was available for samples 183/SR1–4 after re-sieving, prepared from ~750g initial sample mass (in contrast to 0.19g of material prepared from ~1kg initial mass for sample 183/SR6). Where possible, such re-sieving is preferred because it acts as an additional purification step by removing partially etched feldspars; however, examination of the OSL-IR depletion ratio and of the raw IRSL decay curves demonstrated that there was no significant feldspar contamination of any of the sample material prepared in this dating study, regardless of whether it could be re-sieved after etching (183/SR1–4) or not (183/SR6).

A2. Recovery of a known laboratory irradiation dose

A dose recovery test was conducted for sample 183/SR2, as conducted for sample 183/SR6 and described in section 5.3 of the main report. Sample material was not restricted (unlike the situation for sample 183/SR6), therefore 24 small aliquots of sample 183/SR2 were used for this dose recovery test. As was also found for sample 183/SR6, for sample 183/SR2 the ratio of the beta dose recovered to the dose given (1.13 Gy) is within 5% of unity (mean ratio dose recovered to dose delivered = 0.99 ± 0.1; see Figure A1 for data from individual aliquots). The SAR measurement protocol and preheat conditions used for dating (given in Table 2 of the main report) are therefore appropriate and working well for the sample material from this site.

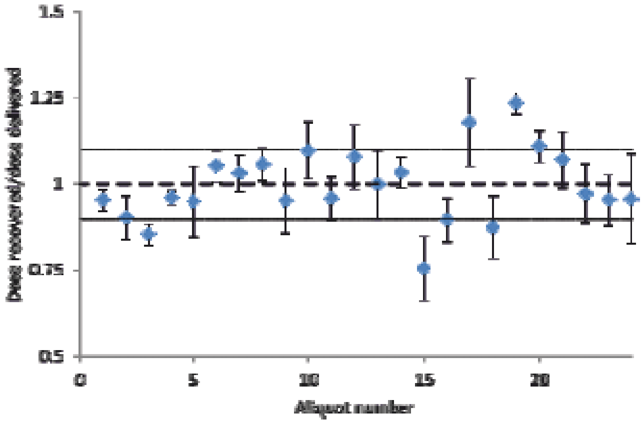

Figure A1: Recovery of a known beta dose for 24 aliquots prepared from sample 183/SR2. The dashed line indicates unity, and the dotted lines indicate ± 10% of unity

A3. Determination of the equivalent dose for use in the final OSL age calculation

The equivalent dose was determined for samples 183/SR1-4, as described in the main report for sample 183/SR6 (section 5.4–5.5), using small aliquots of prepared quartz material. The aliquots were screened for their suitability in the final age equation using the series of tests described in the main report. The equivalent dose was determined for each sample based on a minimum of 40 aliquots for each sample (see Figure A2 and Table A1); a total of 48 small aliquots were measured for each sample, with the most common reason for rejection of aliquots being low signal levels leading to poor recycling ratios. The data for aliquots that were accepted after screening are shown plotted as radial plots in Figure A2 (see section 5.5 of the main report for a description of radial plots). In each case, the over dispersion value (σ) is low (Figure A2), and hence the weighted mean of the D_e values was used for the calculation of the final OSL ages. The error on each determination of D_e was calculated using the standard error. The D_e and standard error for samples 183/SR1-4 are given in the OSL age table (Table A1).

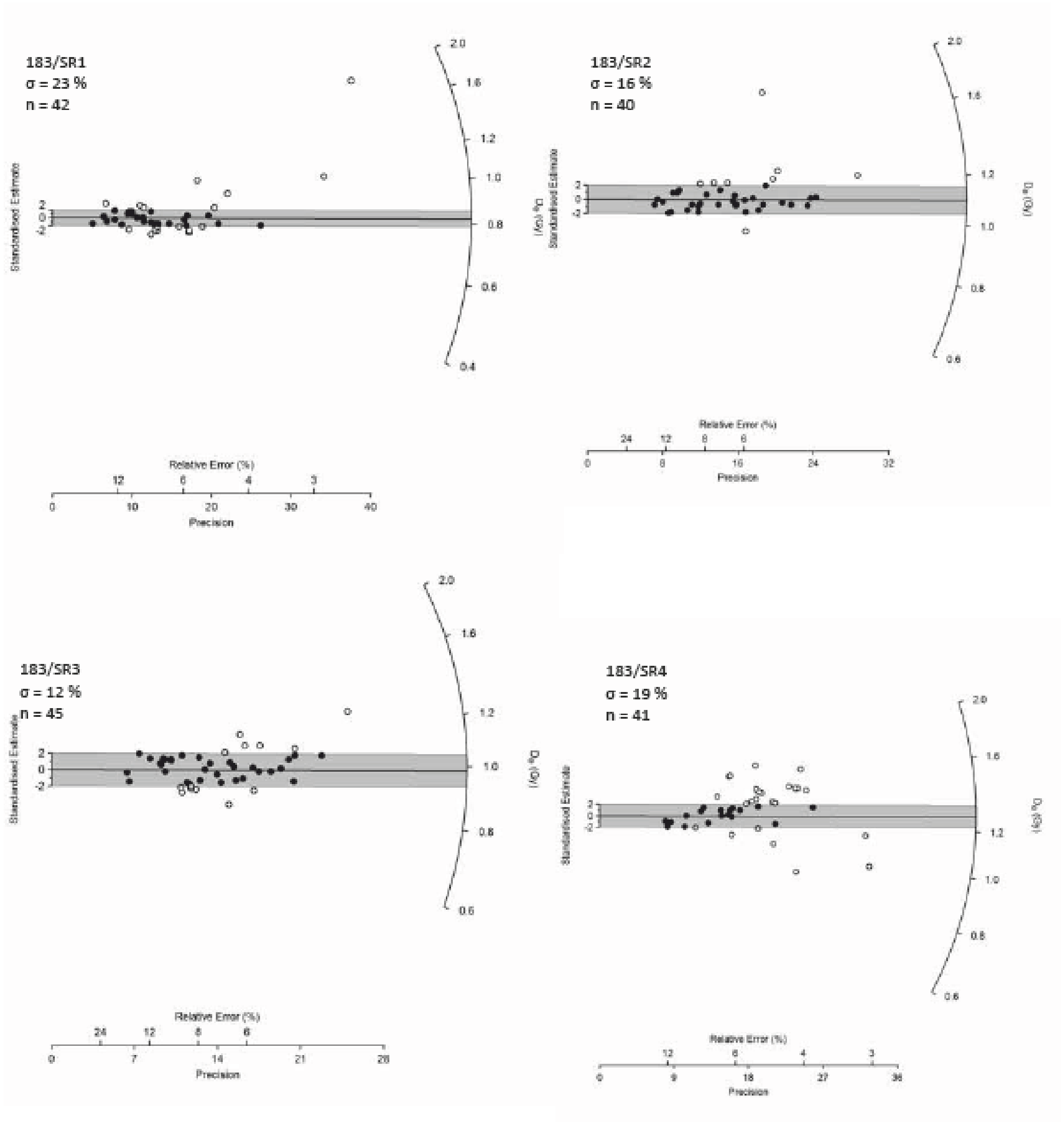

Figure A2: Radial plots to show the distribution of equivalent dose (D_e) values used for the determination of OSL ages for samples Aber-183/SR1–4. Each data point represents one aliquot which has passed the screening tests; closed symbols indicate those data points falling within 2σ error of the weighted mean D_e value, whilst open symbols are used for data points beyond 2σ error

A4. Dosimetry data

The dose-rates for samples 183/SR1–4 were assessed and calculated as described in section 5.6 of the main report. The field gamma spectrometry data are shown in Table 4 of the main report, and the dose-rates used in the determination of the OSL ages are given for each sample in Table A1.

A5. OSL age determinations

The equivalent dose (D_e) data (discussed in section A3) and the results of laboratory dosimetry measurements (discussed in section A4) were combined for each sample, with corrections being made for attenuation by water and for grain size, to give OSL age for each sample. These data, including the final age determinations, are presented in detail in Table A1. The datum is 2011, and the error shown for the D_e determination (Table A1) is the standard error (see sections A3 and 5.5) (ie the standard deviation divided by the square root of the number of independent estimates of D_e). The percentage error on each OSL age is relatively small, and in keeping with that of sample 183/SR6, being < 6%.

A6. Summary and conclusions

Following a pilot OSL study of sample 183/SR6, taken from a clay-rich unit exposed on the southern-most long side of the Water Meadows excavation trench #7 (see Figures 4a and b and discussions within the main report), four further OSL samples were investigated and are discussed in this Addendum to the main report. These four additional samples (Aber-183/SR1–/SR4; see Table 1 for English Heritage-assigned excavation site codes) were taken from each of four clay units exposed at the narrow northwest end of trench #7 (see Figures 3a and b of the main report).

Using small multiple-grain aliquots of quartz, the samples studied proved sufficiently sensitive and responsive to facilitate well-resolved dating using OSL. Checks and screening criteria were applied to the aliquots used for OSL dating, and also to additional aliquots prepared from the samples, to ensure that the data included in the final age calculation were of the highest quality. The SAR measurement protocol was appropriate for the samples studied, and the sensitivity correction worked well.

The final OSL ages generated for the samples reported in this Addendum show that samples 183/SR1–4 are all significantly younger than sample 183/SR6 (discussed in the main report) which was early Holocene in age (9770 ± 580 years). Samples 183/SR1–4 give ages that are in chronostratigraphic order (within errors). The lowermost clay-rich unit was deposited 1200 ± 60 years ago (sample 183/SR4), whilst the three uppermost clay-rich units were deposited in rapid succession ~740 years ago (samples 183/SR1-3;

see Table A1). Although the percentage error on each OSL age is low (<6%), further resolution of the time of deposition of these three uppermost clay-rich units is not possible due to the rapid accumulation of these sedimentary units.

Table A1: OSL sample details, equivalent dose and dose-rate data, and OSL ages

'Later Silbury', Trench #7 'Water Meadows'		
Aberystwyth Lab. number	183/SR1	183/SR2
EH excavation codes	*Sample 57007 in fill 97011*	*Sample 57008 in fill 97012*
Depth down-section (m)	0.68 ± 0.02	0.88 ± 0.02
Material used for dating	quartz	Quartz
Grain size (µm)	90-250	90-250
Preparation method	Heavy liquid separation (sodium polytungstate); 40% HF etch 45 mins	Heavy liquid separation (sodium polytungstate); 40% HF etch 45 mins
Measurement protocol	SAR; small multiple-grain aliquots; OSL 470nm; detection filter 7.5mm Hoya U-340	SAR; small multiple-grain aliquots; OSL 470nm; detection filter 7.5mm Hoya U-340
Aliquots measured	48	48
Aliquots used for D_e	42	40
Equivalent Dose, D_e (Gy)*	0.83 ± 0.04	1.10 ± 0.03
Water content (% dry mass)	20 ± 5	30 ± 5
U (ppm)	0.98 ± 0.04	1.11 ± 0.04
Th (ppm)	4.63 ± 0.02	5.27 ± 0.21
K (%)	0.52 ± 0.02	0.65 ± 0.03
Layer removed by etching (µm)	10 ± 2	10 ± 2
Infinite β dose-rate (Gy/ka)	0.621 ± 0.026	0.984 ± 0.042
External β dose-rate 'wet' (Gy/ka)	0.496 ± 0.032	0.715 ± 0.045
External β dose-rate 'wet' (Gy/ka)	0.456 ± 0.024	0.539 ± 0.026
Cosmic (Gy/ka)	0.201 ± 0.020	0.191 ± 0.019
Total dose-rate (Gy/ka)	1.15 ± 0.05	1.45 ± 0.06
OSL Age (a)#	720 ± 40	760 ± 40

[#] Ages are expressed as years before AD 2011, rounded to the nearest 10 years. All calculations were performed before rounding.

[*] The error shown is the standard error on the mean.

‘Later Silbury’, Trench #7 ‘Water Meadows’		
Aberystwyth Lab. number	183/SR3	183/SR4
EH excavation codes	*Sample 57009 in fill 97013*	*Sample 57010 in fill 97014*
Depth down-section (m)	1.09 ± 0.02	1.22 ± 0.02
Material used for dating	quartz	quartz
Grain size (µm)	90-250	90-250
Preparation method	Heavy liquid separation (sodium polytungstate); 40% HF etch 45 mins	Heavy liquid separation (sodium polytungstate); 40% HF etch 45 mins
Measurement protocol	SAR; small multiple-grain aliquots; OSL 470nm; detection filter 7.5mm Hoya U-340	SAR; small multiple-grain aliquots; OSL 470nm; detection filter 7.5mm Hoya U-340
Aliquots measured	48	48
Aliquots used for D_e	45	41
Equivalent Dose, D_e (Gy)*	0.99 ± 0.02	1.28 ± 0.05
Water content (% dry mass)	30 ± 5	30 ± 5
U (ppm)	0.93 ± 0.05	0.94 ± 0.04
Th (ppm)	4.46 ± 0.23	3.51 ± 0.16
K (%)	0.53 ± 0.03	0.41 ± 0.02
Layer removed by etching (µm)	10 ± 2	10 ± 2
Infinite β dose-rate (Gy/ka)	0.995 ± 0.043	0.701 ± 0.031
External β dose-rate ‘wet’ (Gy/ka)	0.724 ± 0.045	0.510 ± 0.032
External β dose-rate ‘wet’ (Gy/ka)	0.457 ± 0.024	0.376 ± 0.019
Cosmic (Gy/ka)	0.187 ± 0.019	0.184 ± 0.018
Total dose-rate (Gy/ka)	1.37 ± 0.06	1.07 ± 0.04
OSL Age (a)#	720 ± 30	1200 ± 60

[#] Ages are expressed as years before AD 2011, rounded to the nearest 10 years. All calculations were performed before rounding.

* The error shown is the standard error on the mean.

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