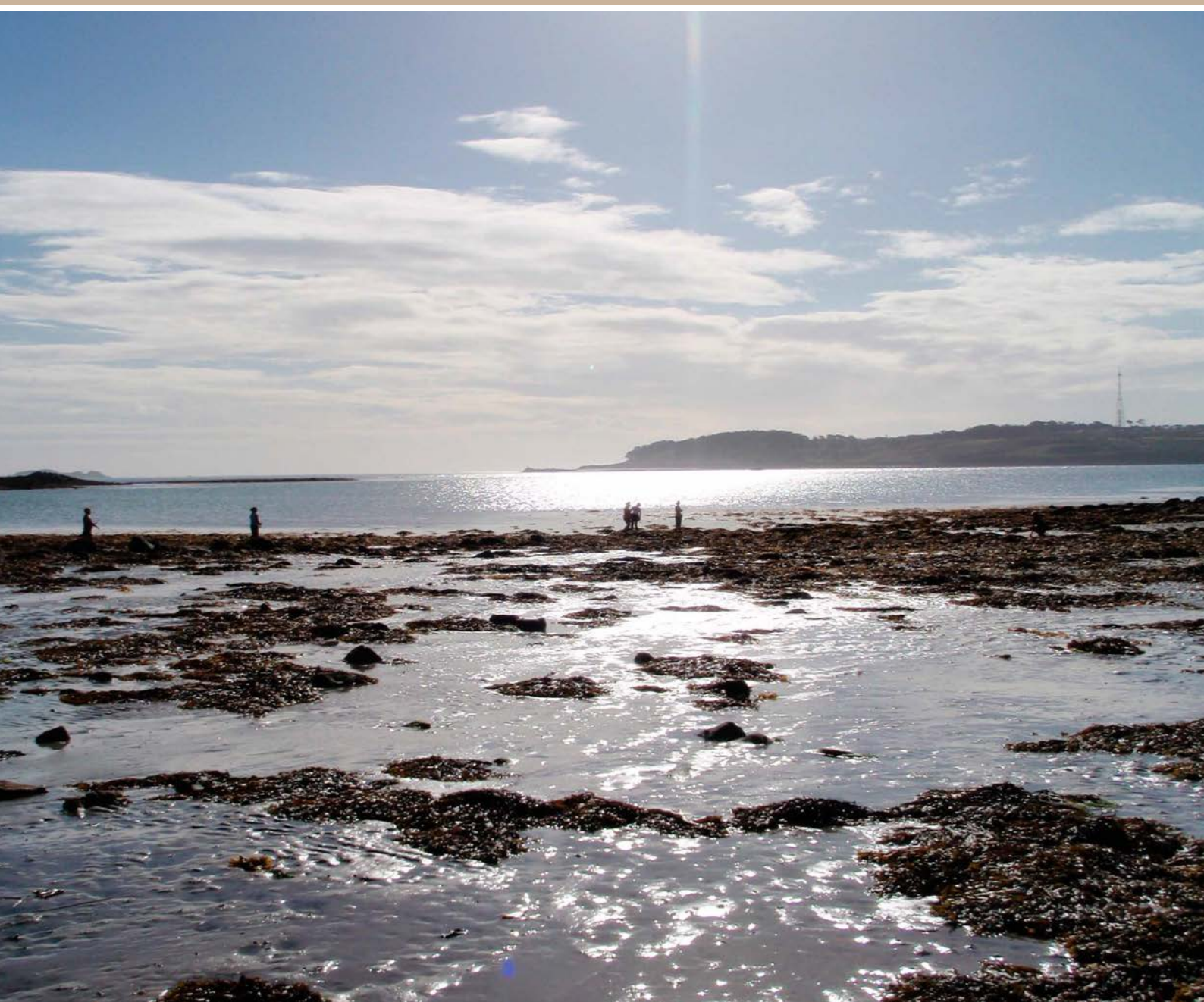


RESEARCH REPORT SERIES no. 2-2013

# ISLES OF SCILLY OPTICALLY STIMULATED LUMINESCENCE DATING OF COASTAL AND INTERTIDAL SEDIMENTS

SCIENTIFIC DATING REPORT

Helen Roberts and Peter Marshall



INTERVENTION  
AND ANALYSIS



ENGLISH HERITAGE

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Research Report Series 2-2013

## ISLES OF SCILLY

# OPTICALLY STIMULATED LUMINESCENCE DATING OF COASTAL AND INTERTIDAL SEDIMENTS

Helen M Roberts and Peter Marshall

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## **SUMMARY**

This report presents the findings of a study using optically stimulated luminescence (OSL) dating to determine the ages of coastal and intertidal sediments from the Isles of Scilly, undertaken as part of the larger Lyonesse Project commissioned by English Heritage's Historic Environment Enabling Programme (HES project number 2009029), and lead by Charles Johns, Historic Environment Projects, Cornwall Council. The Lyonesse project aimed to study the evolution of the coastal and marine environment of the Isles of Scilly during the Holocene in order to gain a greater understanding of changes in sea-level, the development of the coastal landscape, and the response of human populations to changes in the environment. Intertidal sands from sites around the islands of Tresco and St. Mary's, and fine-sands from a wetland area east of Hugh Town, St. Mary's, were dated using the OSL signal from sand-sized quartz, employing a Single Aliquot Regenerative dose (SAR) measurement protocol. Two modern analogue intertidal sediment samples were also dated, giving ages consistent with the present day, indicating that incomplete bleaching is not a problem in this setting. Where more than one OSL age was generated at any section, the ages were in chronostratigraphic order. At some sites, radiocarbon dates were also generated, and in these cases the OSL ages were in agreement with those radiocarbon dates, thus validating both methods in the environments studied. The chronologies generated using both OSL and radiocarbon methods are therefore found to be reliable, and can be used to inform the evolution of the coastal and marine environment of the Isles of Scilly.

## **CONTRIBUTORS**

Helen M Roberts

## **ACKNOWLEDGEMENTS**

Ms Hollie Wynne and Mrs Lorraine Morrison (Aberystwyth University) are acknowledged for assistance with preparation of the samples for OSL dating.

## **ARCHIVE LOCATION**

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St Mary's  
Isles of Scilly  
TR21 0JT

Archaeological Data Service (digital)  
Department of Archaeology  
The King's Manor  
University of York  
York  
YO1 7EP

## **DATE OF Research**

2009–2012

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## INTRODUCTION

This report describes the optically stimulated luminescence dating undertaken at Aberystwyth University as part of a two-year study of the evolution of the coastal and marine environment of the Isles of Scilly (Charman *et al*/2013). The Lyonesse project was commissioned by English Heritage's Historic Environment Enabling Programme (Johns *et al*/2009; HES project number 2009029), and was conducted by Historic Environment Projects, Cornwall Council (project leader, Charles Johns), in conjunction with experts from Cardiff (Jacqui Mulville and Steve Mills), Exeter (Dan Charman) and Plymouth (Ralph Fyfe and Roland Gehrels) Universities, volunteers (Rhiannon Philp and Mike Scott), local marine archaeologists and enthusiasts from the Cornwall and Isles of Scilly Maritime Archaeological Society (CISMAS) (Kevin Camidge, Innes McCartney, Luke Randall, Dave McBride, Martin Davis, Tom-Badham-Thornhill, Natasha Fellows, and Marta Pérez-Fernández), and the Islands Maritime Archaeology Group (IMAG) (Todd and Carmen Stevens, Philip Roberts and Robin Burrow).

The Scillonian archipelago (Fig 1) is located 45km south-west of Land's End, UK, and comprises around 200 islands, islets, and rocks, with large intertidal and subtidal areas currently found under shallow seas. The Lyonesse project aimed to study the evolution of the coastal and marine environment of the Isles of Scilly during the Holocene in order to improve our understanding of changes in sea-level, the changing coastal landscape, and the response of human populations to such changes (Johns *et al*/2009). The project involved the acquisition of geophysical survey data in shallow marine settings to identify submerged sediments and archaeological remains indicative of previously lower sea-levels, and also biostratigraphic analysis of coastal, intertidal, and submerged sediments at selected sites around the Isles of Scilly to reconstruct the landscape and vegetation of now-submerged land surfaces and assess their relationship to drowned archaeological structures (Charman *et al*/2013). Radiocarbon dating and optically stimulated luminescence were used to provide numerical chronologies for the sediments in this study (Charman *et al*/2013), with the aim of providing securely dated sea-level index points and hence developing a new sea-level reconstruction for the Isles of Scilly, which in turn can be used to test competing models for sea-level change for Scilly (Thomas 1985; Ratcliffe and Straker, 1996) and also models of glacial isostatic adjustment (eg Massey *et al*/2008).

In this study, optically stimulated luminescence (OSL) dating was applied to sand-sized quartz taken from sand units above or below intertidal 'peat' units for sites found on the islands of Tresco and St Mary's (Fig 1). These 'peat' units are dominated by a minerogenic component, with a relatively low organic content (eg 2–20%, Charman pers comm). Three samples were also taken from a core removed from the Lower Moors wetland site on St. Mary's (Fig 1), and furthermore, two modern samples were examined to investigate the degree of bleaching in the intertidal environment. In this report, the principles of luminescence dating are explained, the tests undertaken to establish the appropriate measurement conditions for dating are discussed, and the final luminescence ages

determined for five samples from field season 1 (September 2009), and ten samples from field season 2 (September 2010), are presented and discussed.



*Figure 1: NASA astronaut photograph ISS014-E-16597 taken of the Isles of Scilly on March 10, 2007. The key sites discussed in this OSL report are marked on this image*

## 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 ionizing radiation in the natural environment. This dating technique can be applied directly to the mineral grains that make up sediment deposits. The event being dated is the last time the mineral grains were exposed to sunlight, ie the time elapsed since deposition and burial 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; the efficacy of this 'bleaching' of any previous signal can be investigated using samples taken from a modern analogue of the depositional environment.

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 within the surrounding sediment. The luminescence signal is measured in the laboratory by stimulating small subsamples, 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 the response to known laboratory-administered radiation doses, in order to determine how much radiation the sample was exposed to during burial (termed the equivalent dose,  $D_e$ , or the ‘burial dose’). This value is divided by the radiation dose to which the sample was exposed each year since deposition and burial (termed the ‘annual dose rate’), to give the OSL age (see Equation 1). Further details on OSL methods are given in Aitken (1998), and in recent reviews by Duller (2004) and various papers within a special issue of *Boreas* (eg Wintle 2008; Roberts 2008).

#### Equation 1

$$\text{OSL age (years)} = \text{Burial dose (Grays)} / \text{Annual dose rate (Grays per year)}$$

$$(1 \text{ Gray} = 1 \text{ Joule/kg})$$

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  $>63\mu\text{m}$  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,  $D_e$ .

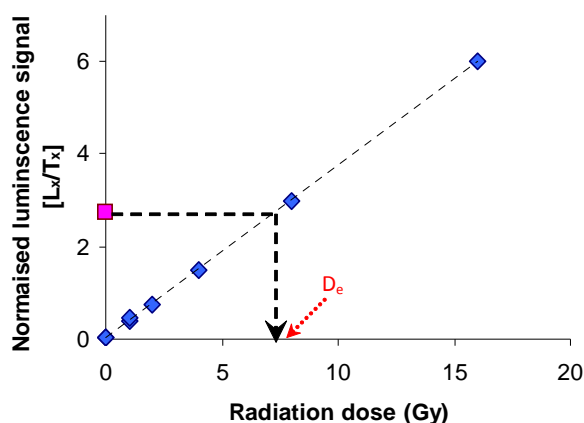


Figure 2: Dose-response or 'growth' curve (diamond symbols) generated from measurements made using the Single Aliquot Regenerative dose (SAR) measurement protocol, used in this study. The natural luminescence intensity (square symbol) of the aliquot is calibrated against the response to these known artificial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

## SAMPLE SITES AND OSL SAMPLE COLLECTION

A number of monolith samples were taken from the Isles of Scilly at the lowest tide during field season 1, September 2009. The monolith samples were wrapped to preserve the moisture content and removed to Exeter University. From these monolith tins, four samples were taken for Optically Stimulated Luminescence (OSL) dating, and one bagged surface sample was also examined, making a total of five OSL samples being examined in the first round of dating (see Table 1 for sample locations). In each case, the material sampled for dating was clean, grey coarse-sand found above or below an intertidal 'peat' unit (see descriptions in Table 1). The monolith samples were sub-sampled for OSL dating under subdued red-lighting conditions to preserve the signal used for dating; the light-exposed surface material from the monolith samples was used for dosimetry assessments, and the innermost sediments were taken for determination of the equivalent dose (' $D_e$ '; Equation 1). To check the efficacy of bleaching in the intertidal zone, two surface samples were taken, namely from Crab's Ledge and Bathinghouse Porth. These 'modern' samples represent a present-day analogue for the environment of deposition of the OSL samples, and should give an age of zero years, within errors, if the samples are well bleached at the time of deposition.

*Table 1: Location and altitude of OSL samples taken in field season 1; the OSL sample prefix for all field season 1 samples is: 'Aber-161'*

Sample No.	Site Name	Sample type	Location	Elevation of top of monolith tin* (m)
LPTR1-1	Tresco, Crab's Ledge	Clean grey sand between darker intertidal 'peat' units; monolith	89755.58 E 13808.75 N	-0.03
LPTR1-M	Tresco, Crab's Ledge	Modern sand sample taken from monolith	As above	
LPTR3-1	Tresco, Bathinghouse Porth	Clean grey sand between darker intertidal 'peat' units; monolith	89390.06 E 13601.71 N	0.83
LPT3-M	Tresco, Bathinghouse Porth	Modern sand sample collected in bag	As above	
LPPM1-1	St Mary's, Porth Mellon	Clean grey sand between darker intertidal 'peat' units; monolith	90789.27 E 10889.77 N	-1.50

\*Elevations for intertidal samples are relative to local OD St Mary's converted from GPS ellipsoid heights using Ordnance Survey's National Geoid Model OSGM02. All Eastings and Northings are British National Grid (OSGB1936) converted from GPS latitude and longitude coordinates using Ordnance Survey's National Grid Transformation OSTN02 (Mills pers comm; Charman *et al* 2013)

A further set of monolith samples were taken from the Isles of Scilly during field season 2, September 2010, taken at the lowest tide of each day; these tides were also some of the lowest tides for several years. The monolith samples were wrapped to preserve the moisture content and removed to Exeter University. From these monolith tins, seven samples were taken for Optically Stimulated Luminescence (OSL) dating, from sites on Tresco and St. Mary's. In each case, the material sampled for dating was coarse-sand found above or below an intertidal 'peat' unit (see descriptions in Table 2). Additionally, three OSL samples were taken from a 6cm-diameter core sampled in a grey-plastic core-liner from a wetland site, Lower Moors, east of Hugh Town, St. Mary's. The sediment sampled within the Lower Moors core was finer-grained than that from the intertidal (monolith) sites, being primarily silt to fine-sand sized. The field season 2 sample locations are shown in Table 2. Again, the monolith and the core-samples were sub-sampled for OSL dating under subdued red-lighting conditions to preserve the signal used for dating; the light-exposed surface material from the monolith samples was used for dosimetry assessments, and the innermost sediments were taken for determination of the equivalent dose.

*Table 2: Location and altitude of OSL samples taken in field season 2; the OSL sample prefix for all field season 2 samples is: 'Aber-184'*

Sample No.	Site Name	Sample type	Location	Elevation of top of monolith tin or of ground surface for cores* (m)
LPPM-2	St Mary's, Porth Mellon	Sandy (fine) organic-rich sediments with thin laminae, between very dark organic-rich fine sediments; monolith	90754.24 E 10908.70 N	-2.30
LPPM-3A	St Mary's, Porth Mellon	Coarse-grained, buff-coloured coarse-sand below 'peat'/organic-rich dark brown sand; monolith	90801.93 E 10859.78 N	-0.82
LPPM-3B	St Mary's, Porth Mellon	'Peat'/organic-rich dark brown sand above coarse-grained, buff-coloured coarse-sand; monolith	As above	
LPOT-1A	St Mary's, Old Town	Very coarse, buff-coloured sand between darker intertidal 'peat' units; monolith	91348.11 E 10200.26 N	2.28
LPPH-1A	St Mary's, Porth Hellick	Fine-sand, brown in colour, grading into dark, organic-rich sediments with increasing depth; found below a dark-brown organic-rich unit with a sharp contact at the base; monolith	92575.85 E 10516.97 N	-1.27
LPTR-4A	Tresco, Crab's Ledge	Light-coloured coarse-sand unit below a finer, very dark, organic-rich sand unit; monolith	89769.00 E 13849.82 N	1.84
LPTR-4B	Tresco, Crab's Ledge	Very dark, organic-rich sand unit above a coarser, light-coloured sand unit; monolith	As above	
LM10-28-161	St Mary's, Lower Moors	Laminated buff-coloured fine-sand between very dark, organic-rich silts; core	91032.57 E 10596.12 N	2.158
LM10-28-217	St Mary's, Lower Moors	Laminated buff-coloured fine-sand between very dark, organic-rich silts; core	As above	
LM10-28-277	St Mary's, Lower Moors	Laminated buff-coloured fine-sand between very dark, organic-rich silts; core	As above	

\*Elevations for intertidal samples are relative to local OD St Mary's converted from GPS ellipsoid heights using Ordnance Survey's National Geoid Model OSGM02. All Eastings and Northings are British National Grid (OSGB1936) converted from GPS latitude and longitude coordinates using Ordnance Survey's National Grid Transformation OSTN02 (Mills pers comm; Charman *et al* 2013)

## OSL SAMPLE PREPARATION

Samples were taken for preparation of coarse-grained quartz, using standard methods, outlined below.

Samples were pre-treated with a 10% v.v. dilution of concentrated (37%) hydrochloric acid (HCl) to remove carbonates and surficial coatings, then washed three times in distilled water. Samples were then treated with 20 vols hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) to remove organic material, and then washed as previously. Samples were dried and then sieved using the following mesh sizes: 355, 300, 250, 212, 180, 150, 125, 90, and 63µm diameter mesh. The 180–210µm diameter grain size was typically selected for further processing prior to OSL dating, although the range 212–355µm diameter was used for sample 184/LPOT-1A from Old Town, St. Mary's, and 63–90µm diameter grains were used for samples from Lower Moors, St. Mary's. A solution of sodium polytungstate ('heavy liquid') was used to separate out the quartz material from the feldspar and heavy mineral fractions of the selected grain size ranges, on the basis of differences in density. The quartz-rich fraction of the sediments (density between 2.62–2.70gcm<sup>-3</sup>), 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 etch procedure. The samples were rinsed a minimum of three times in distilled water, centrifuging between washings, and then dried at 50°C, prior to re-sieving. This final sieving acts as a further quartz purification step, as it removes feldspar grains that have not been totally dissolved with HF, but which have been significantly etched and therefore reduced in diameter. Any mica remaining in the sample at this point was removed by static. The final quartz grains are then ready for OSL measurements to determine the 'burial dose' or equivalent dose, D<sub>e</sub>.

The outer, light-exposed material removed from the monolith or core samples was suitable for laboratory-based measurements of water content and dosimetry, as these measurements do not require unexposed sample material. The monolith tins, cores, and sample bags had been sealed immediately on sampling to preserve their water content. The dosimetry samples were weighed prior to drying at 50°C. Drying continued until a constant mass was recorded, to establish the field water content (expressed as % dry mass sediment) at the time of sampling; these measurements of conditions at the time of sampling provide a benchmark for the water content values used in the final age calculations. After drying, the dosimetry samples were crushed to a fine powder using a ball mill, prior to thick source alpha and beta counting (discussed further below) to determine the annual dose rate to the sample.

## EQUIPMENT AND METHODS

All OSL measurements were conducted using an automated Risø TL/OSL reader, equipped with a combined high-power blue LED/ infra-red laser diode OSL unit, and a

beta source for irradiations. The combined OSL unit was employed at 80% of full diode current, providing approximately 17mW/cm<sup>2</sup> power from the blue LED unit (470nm), and 370mW/cm<sup>2</sup> from the IR laser diode (830nm). All measurements were made whilst holding the sample at 125°C, and OSL was detected using 7.5mm Hoya U-340 filters.

Measurements of OSL were made on 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.

Prepared quartz grains for each sample were presented for OSL measurements by mounting the grains in a monolayer onto 0.98mm-diameter aluminium discs, sprayed lightly with Silkospray™ silicone oil to hold the grains in place during measurement. The discs, or aliquots, can be prepared using various amounts of sample; in this study, medium-sized (5mm diameter, giving ~200 grains per aliquot for 180–210µm diameter grains) aliquots gave sufficient light for the determination of  $D_e$  values, and allowed for the possibility of identifying samples with non-homogeneous  $D_e$  values.

As part of the sequence of OSL measurements made, outlined in Table 3, a minimum of three regenerative beta doses was 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 the first regenerative dose (applied at the end of the measurement protocol) was repeated to monitor the sensitivity correction applied (this is sometimes 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. A preheat test was conducted to identify the appropriate measurement conditions for dating. This preheat test involves making measurements for a range of pre-heat temperatures between 160–300°C in 20°C step intervals (held for 10s), with three aliquots at each temperature. As a result, mid-range preheat temperatures of 200 or 220°C were identified as being appropriate for dating measurements for these samples.

*Table 3: Outline of the SAR measurement protocol. A minimum of three regenerative doses were employed in this study, designed to bracket the natural signal*

Step Number	SAR sequence description
1	Preheat: (160–300 °C), heating rate 5 °C/s, hold at temperature for 10s
2	Measure natural or regenerative dose signal ('Lx'): 100s OSL @125 °C
3	Apply a fixed test dose
4	Cut heat: 160 °C, heating rate 5 °C/s
5	Measure test dose signal ('Tx'):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 0Gy dose ('recuperation' check)
31–5	Repeat steps 1–5
36	Apply regenerative dose 1 ('recycling' test)
37–41	Repeat steps 1–5

The short exposure time between tides and the nature of the sediments being sampled (sandy sediments located within the intertidal zone, which were therefore wet and prone to collapse) precluded the use of a field gamma detector to measure directly the gamma dose-rate to the OSL sample taken. The alpha, beta, and gamma dose-rates were therefore calculated from laboratory measurements, using Daybreak detectors for thick source alpha counting and a Risø GM-25-5 beta counter for beta counting, applied to finely ground bulk sample material. The uranium and thorium determinations were derived from the pair count, whilst potassium contents were calculated by subtraction using the measured beta dose-rate and that calculated from the uranium and thorium values. The units sub-sampled for OSL dating were all within 0.2m of the uppermost surface of the 0.3m-deep monolith tin, and were bracketed by different stratigraphic units. A number of samples were therefore taken from these adjacent stratigraphic units to allow the dosimetry (Table 4) to be assessed for the gamma field (ie within a 0.3m radius of the OSL sample), and hence to allow the gamma and total dose-rate to the OSL dated unit to be calculated (shown in Tables 5–7). The gamma dose rate to the sample was calculated according to the principles outlined in Appendix H of Aitken (1985), using the multi-layer gamma model of Prof Ian Bailiff and Dr Sarah Barnett (University of Durham).

*Table 4: Thick-source alpha and beta counting-derived potassium (K), uranium (U), and thorium (Th) contents for OSL samples and for sedimentary units located within  $\pm 0.3\text{m}$  of any OSL sample. For any given section (indicated by samples having the same prefix, eg 161/LPTR1) the samples are listed in order of increasing depth. The thickness of each unit is also shown*

Sample Name	K (%)	U (ppm)	Th (ppm)	Thickness of Unit (m)
161/LPTR1-1M (&*)	3.31 $\pm$ 0.13	2.36 $\pm$ 0.23	5.40 $\pm$ 0.75	0.02
161/LPTR1-1 Dos. A*	3.25 $\pm$ 0.14	2.55 $\pm$ 0.22	6.15 $\pm$ 0.73	0.055
161/LPTR1-1	3.74 $\pm$ 0.14	2.01 $\pm$ 0.17	3.65 $\pm$ 0.54	0.01
161/LPTR1-1 Dos. B*	2.01 $\pm$ 0.11	3.83 $\pm$ 0.28	5.52 $\pm$ 0.92	0.135
161/LPTR1-1 Dos. C*	1.37 $\pm$ 0.09	2.77 $\pm$ 0.26	5.72 $\pm$ 0.85	> 0.08
161/LPT3-M (&*)	4.18 $\pm$ 0.16	2.61 $\pm$ 0.22	4.82 $\pm$ 0.71	0.02
161/LPTR3-1 Dos. A*	4.01 $\pm$ 0.17	1.73 $\pm$ 0.34	10.19 $\pm$ 1.13	0.11
161/LPTR3-1	4.15 $\pm$ 0.16	1.98 $\pm$ 0.24	6.26 $\pm$ 0.79	0.10
161/LPTR3-1 Dos. B*	4.13 $\pm$ 0.18	3.34 $\pm$ 0.32	8.00 $\pm$ 1.04	> 0.09
161/LPPM1-1 Dos. A*	1.83 $\pm$ 0.11	2.83 $\pm$ 0.31	8.03 $\pm$ 1.02	0.09
161/LPPM1-1	3.46 $\pm$ 0.13	1.80 $\pm$ 0.11	4.63 $\pm$ 0.35	0.13
161/LPPM1-1 Dos. B*	1.76 $\pm$ 0.10	3.51 $\pm$ 0.26	5.11 $\pm$ 0.85	> 8
184/LPPM-2 Dos. A*	1.75 $\pm$ 0.10	2.89 $\pm$ 0.24	5.44 $\pm$ 0.78	0.07
184/LPPM-2	2.37 $\pm$ 0.11	2.57 $\pm$ 0.23	5.31 $\pm$ 0.76	0.06
184/LPPM-2 Dos. B*	1.54 $\pm$ 0.09	2.94 $\pm$ 0.25	6.27 $\pm$ 0.82	> 0.13
184/LPPM-3B (&*)	3.46 $\pm$ 0.13	1.99 $\pm$ 0.16	3.67 $\pm$ 0.53	0.05
184/LPPM-3A (&*)	3.52 $\pm$ 0.13	2.63 $\pm$ 0.18	3.66 $\pm$ 0.58	> 0.25
184/LPOT-1 Dos. C*	2.72 $\pm$ 0.12	2.38 $\pm$ 0.23	5.33 $\pm$ 0.74	0.10
184/LPOT-1A	4.00 $\pm$ 0.15	1.69 $\pm$ 0.17	4.13 $\pm$ 0.54	0.055
184/LPOT-1 Dos. D*	3.31 $\pm$ 0.13	2.12 $\pm$ 0.21	4.69 $\pm$ 0.67	0.055
184/LPOT-1 Dos. C*	2.72 $\pm$ 0.12	2.38 $\pm$ 0.23	5.33 $\pm$ 0.74	> 0.85
184/LPPH-1 Dos. D*	3.79 $\pm$ 0.17	3.56 $\pm$ 0.34	7.35 $\pm$ 1.10	0.02
184/LPPH-1 Dos. B*	2.33 $\pm$ 0.14	5.49 $\pm$ 0.35	5.87 $\pm$ 1.13	0.055
184/LPPH-1 Dos. C*	1.56 $\pm$ 0.14	8.27 $\pm$ 0.43	4.48 $\pm$ 1.35	0.035
184/LPPH-1A	1.47 $\pm$ 0.14	7.87 $\pm$ 0.45	4.94 $\pm$ 1.43	> 0.16
184/LPTR-4B (&*)	4.08 $\pm$ 0.14	1.29 $\pm$ 0.11	3.03 $\pm$ 0.37	0.075
184/LPTR-4 Dos. C*	2.81 $\pm$ 0.12	2.86 $\pm$ 0.17	3.85 $\pm$ 0.55	0.025
184/LPTR-4A (&*)	3.91 $\pm$ 0.15	1.62 $\pm$ 0.18	4.74 $\pm$ 0.58	> 0.20
184/LM10-28-161	1.53 $\pm$ 0.09	2.78 $\pm$ 0.22	4.68 $\pm$ 0.71	> 0.30
184/LM10-28-217	1.49 $\pm$ 0.09	3.32 $\pm$ 0.19	4.60 $\pm$ 0.60	> 0.30
184/LM10-28-277	1.26 $\pm$ 0.09	3.62 $\pm$ 0.23	4.59 $\pm$ 0.74	> 0.30

\* Indicates a sample taken for dosimetry measurements to support an OSL age with the same sample prefix.

The cosmic ray dose was estimated from the burial depth (Prescott and Hutton 1994). Water contents were determined in the laboratory from sealed field samples (above). Moisture and beta attenuation factors are given in Aitken (1985). The alpha and beta counting results, cosmic dose rates, water content values, and the dose rates were calculated using the conversion factors of Adamiec and Aitken (1998), are given for each sample in the final age tables (Tables 5–7).

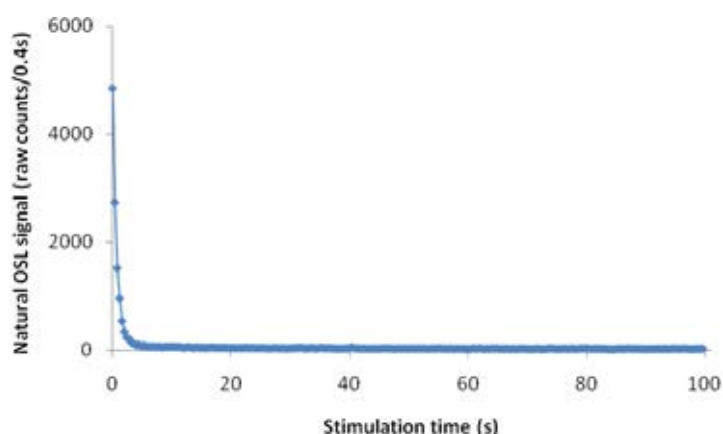


## RESULTS OF EXPERIMENTAL CHECKS

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

### 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 3, and shows a rapid decrease in signal which is characteristic of the decay of a signal from quartz. Routinely, the  $D_e$  values were calculated using the first two data channels (0.8s stimulation) and the background was taken from the end of the decay curve (channels 230–250, the final 8.4s stimulation). This maximised the contribution of the fast component of the OSL signal (Bailey *et al* 1997; Murray and Wintle 2003), and typically represented ~35% of the total OSL signal.



*Figure 3: Typical OSL signal for aliquots in this study. The example shown is from an aliquot of sample 161/LPTR1-1 which was preheated to 200°C/10s. The very rapid decrease in signal, quickly reaching a steady low background is a form which is frequently observed in the study of quartz aliquots. The signal integrated to derive the value of  $D_e$  is that from the first 0.8s of optical stimulation*

The form of the dose-response or 'growth' curve was also examined, and a minimum of three 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 4 shows a typical dose-response curve including repeat zero dose and recycled dose points; error bars are shown, calculated following Banerjee *et al* (2000) and Galbraith (2002), and generated by Analyst (written by Prof Geoff Duller, Aberystwyth University).

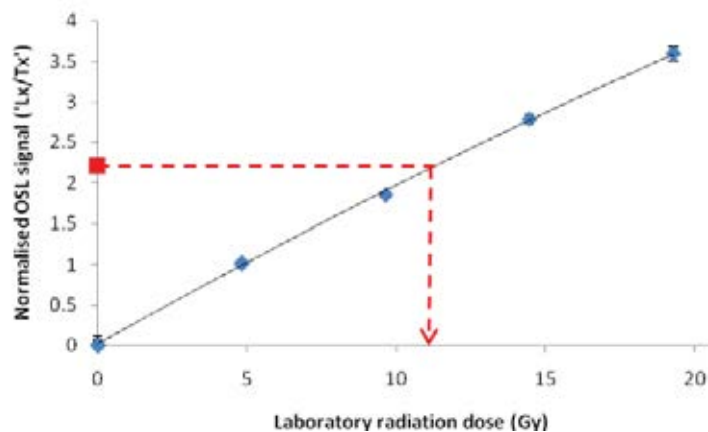


Figure 4: Typical dose-response curve constructed for aliquots in this OSL dating study (blue diamonds). The Natural OSL signal is denoted by a red square. The example shown is from the aliquot of sample 161/LPTR1-1 shown in Figure 3, which was preheated to 200°C/10s

Once the sequence of dating measurements was completed, each aliquot was irradiated and then stimulated using infra-red (IR) laser-diodes at a temperature of 50°C prior to stimulation with blue diodes to measure the regenerative dose signal ( $L_x$ ); the response to a test dose was measured ( $T_x$ ) as usual following stimulation with blue diodes only. This allows calculation of the 'OSL IR depletion ratio' (Duller 2003) which checks the purity of each aliquot (eg Fig 5). Feldspathic minerals respond to stimulation with IR, giving a rapidly decaying signal, but quartz does not appear to respond to stimulation with IR (Spooner and Questiaux 1989). The OSL IR depletion ratios were typically within 10% of unity (within errors), and hence no significant feldspar contamination was considered to be present in the quartz separates prepared for this OSL dating study. To assure the quality of all aliquots examined, the OSL IR depletion ratio was used as one of the screening criteria for dating measurements which followed later (below).

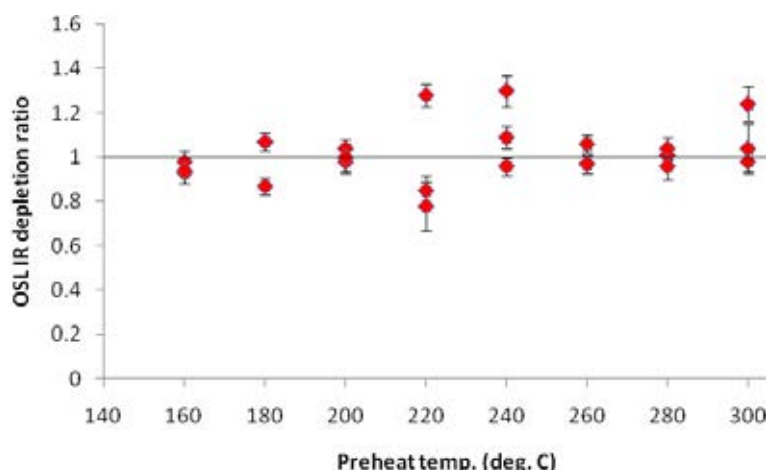


Figure 5: OSL IR depletion ratio data for sample LPTR1-1, collected at a variety of preheat temperatures

### Preheat plateau test

Thermal pretreatments are employed in order to remove any unstable trapped charge prior to measurement of either the natural or an artificially irradiated OSL signal. However, high preheat temperatures are sometimes problematic for young samples, and can lead to erroneously high  $D_e$  values being determined due to thermal transfer of trapped charge from relatively stable yet optically-insensitive traps into OSL traps during preheating (eg Bailey *et al*/2001). Given the likely young age of the samples in this study, it was therefore of particular importance to make OSL measurements using a range of preheat temperatures to try to establish a preheat plateau where common values of  $D_e$  could be identified and any erroneously high  $D_e$  values could be discounted.

A preheat plateau test was conducted for one sample in this study, to identify the appropriate measurement conditions for dating. This test involves the use of a variety of different preheat temperatures from 160–300°C, to force the maximum sensitivity change possible for the sample prior to the measurement of the equivalent dose ( $D_e$ ). The results of this preheat test are shown in Figure 6; three aliquots were measured at each preheat temperature shown. A plateau in  $D_e$  values exists across all preheat temperatures, suggesting that any of these preheat temperatures is suitable for dating. The recycling ratio for the majority of the aliquots in this preheat plateau test is also within the acceptable limits, being within errors of 10% of unity (Fig 7). On the basis of the preheat plateau and recycling ratio data, mid-range temperatures of 200 or 220°C held for 10s were selected as appropriate preheat conditions for dating measurements.

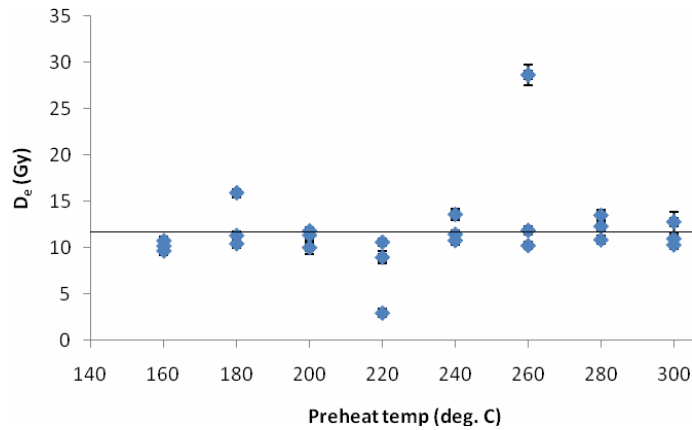


Figure 6: Preheat plateau test results for sample 161/LPTR1-1

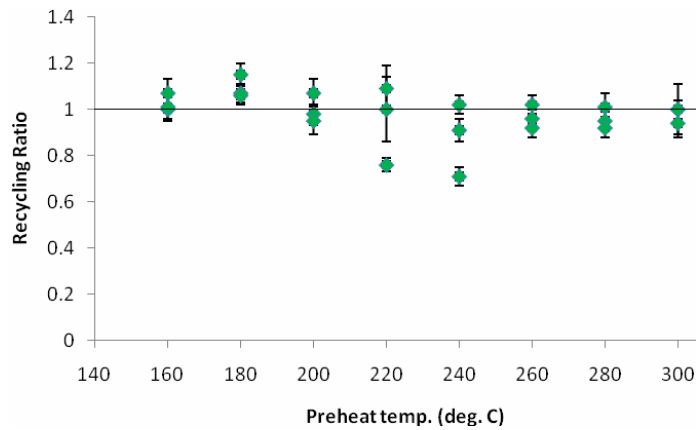


Figure 7: Recycling ratio data for sample 161/LPTR1-1 dose recovery test data

### 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 determined accurately and precisely. This is sometimes referred to as a 'dose-recovery' test and should be conducted on material which has not previously received any thermal pretreatments. This fundamental test was conducted for a total of six aliquots of sample 161/LPTR1-1, using two different thermal treatments (ie a preheat of 200 or 220°C held for 10s).

The laboratory beta dose chosen for the dose-recovery experiment was selected to be similar in magnitude to the Natural  $D_e$  value. Three aliquots were prepared in the same way as the aliquots used for dating. The natural signal was removed from each aliquot by 1000s stimulation with blue diodes at room temperature, followed by a 10,000s pause,

another 1000s stimulation with blue diodes at room temperature, and finally the laboratory radiation dose was applied. The SAR protocol was then applied using the measurement conditions identified by the preheat plateau test for use in dating measurements (ie preheat of 200 or 220°C for 10s, and a test dose cut heat of 160°C). For each aliquot in both thermal treatments, the beta dose applied was recovered to within  $\pm 5\%$ ; the threshold for acceptable recovery is  $\pm 10\%$ . The SAR measurement protocol therefore seems to be appropriate and working well for the sample material used for dating in this study.

## 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 each sample, 24 aliquots were examined to establish  $D_e$  values for use in determining an OSL age. The advantage of working with single-aliquot, rather than multiple-aliquot methods, is that each of the 24 aliquots measured gives rise to an independent assessment of  $D_e$ , and hence, potentially to an OSL age.

One of the most powerful of the in-built dating sequence tests 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 3). In the SAR measurements made in this study, a low irradiation dose was then 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 appropriate, 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 3) should fall within the range of  $1.0 \pm 0.1$  (Murray and Wintle 2000). The vast majority of aliquots passed this 'recycling test' as part of the dating sequence of measurements, 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 dose-response 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 0Gy 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). No significant net OSL signal should be observed following this 0 Gy beta dose if the sensitivity correction is working correctly. No recuperation in OSL signal was observed in the dating sequence measurements, and the dose-response or 'growth' curve generated passed through the origin (eg Fig 4), indicating that all aliquots in the dating sequence were appropriate for further use in  $D_e$  evaluation.

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

The 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, growth-curve shape, recycling ratio, recuperation, and feldspar contamination checks using IR stimulation. The vast majority of samples measured passed these screening tests and were therefore considered suitable for use in determining the final  $D_e$  value for the sample. The most common reason for rejection of aliquots was on the basis of a poor recycling ratio (ie one where the value exceeded the  $1.0 \pm 0.1$  criterion, above). In spite of this, the minimum number of acceptable aliquots combined to determine a final OSL age for any sample in this study was at least 18 (75% of aliquots) and more typically 21–24 aliquots (86–100% of aliquots) from a total of 24 measured aliquots.

For the majority of samples from field season 1 (Figs 8a, b, d, e) and all of the samples from field season 2 (Figs 9a–j) the  $D_e$  values of the aliquots accepted following screening were normally distributed. For these samples, the weighted mean of the  $D_e$  values was therefore taken for calculation of the final OSL age, and 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  $D_e$ ). For sample 161/LPPM1-1, however, the spread in  $D_e$  values for accepted aliquots was rather broad (Fig 8c); for this sample, the simple arithmetic mean of the  $D_e$  values was taken and the error calculated is the standard deviation, in order to reflect the broad distribution and hence relatively large uncertainty in the  $D_e$  value (Table 5). The  $D_e$  and error values are given for each sample in the final OSL age tables; field season 1 ages and the modern samples from field season 1 are shown in Tables 5 and 6, respectively, whilst the ages from field season 2 are given in Table 7.

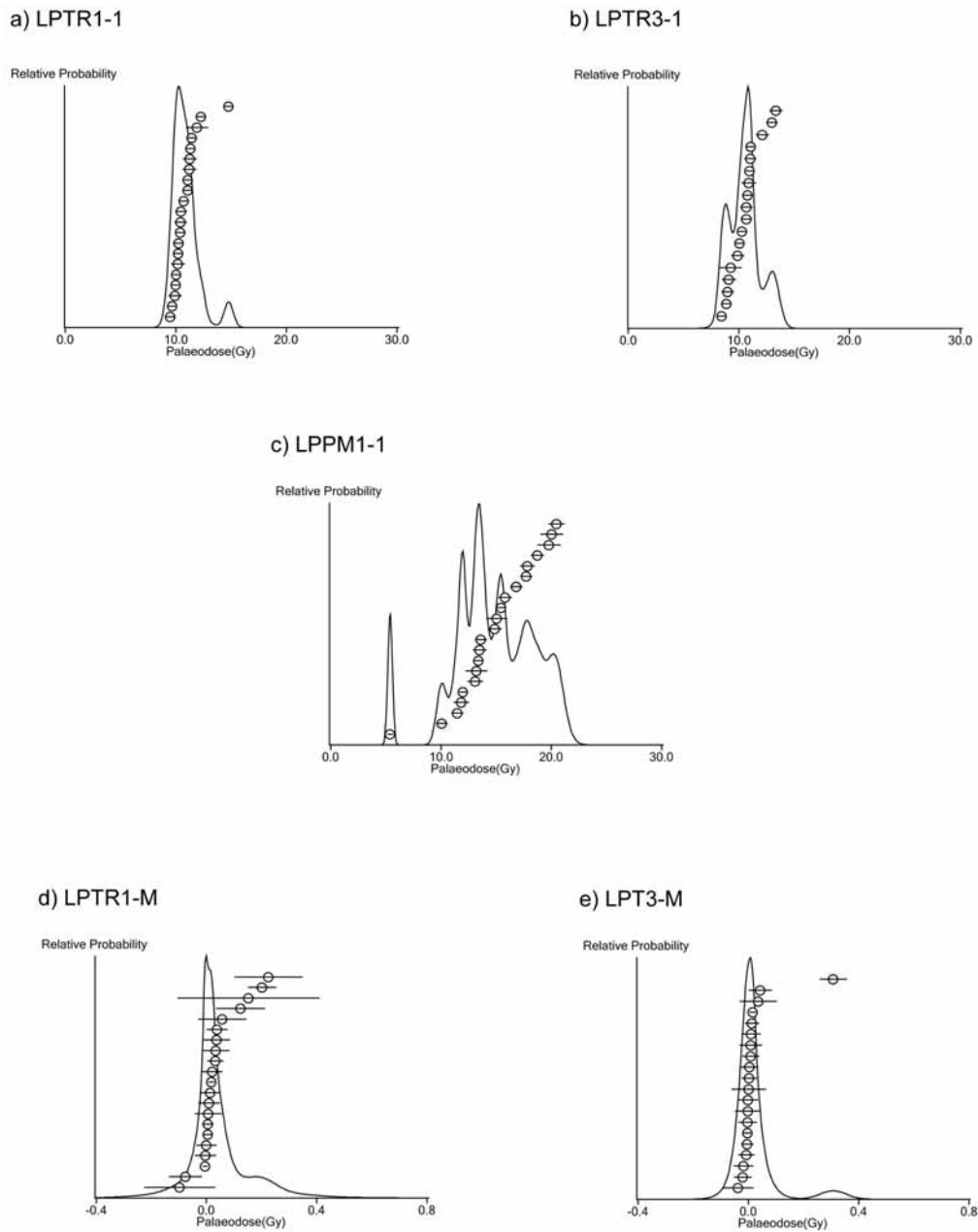
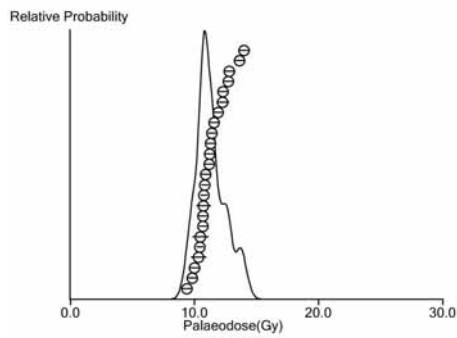
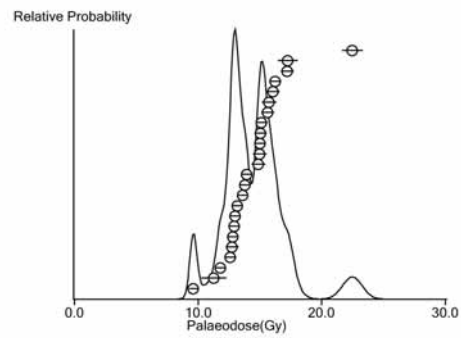


Figure 8a–e: The distribution of  $D_e$  values for aliquots passing the screening criteria, shown for each OSL sample from field season 1. Note the different x-axis scales for the samples shown in Figs a–c versus the modern samples shown in Figures d–e

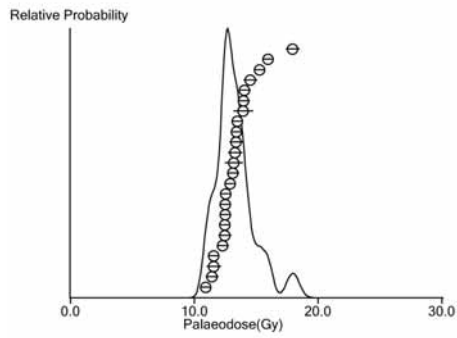
a) LPPM-2



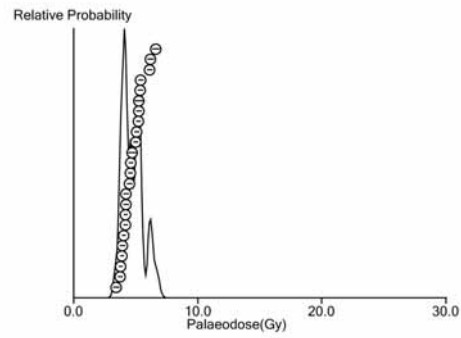
b) LPPM-3A



c) LPPM-3B



d) LPOT-1A



e) LPPH-1A

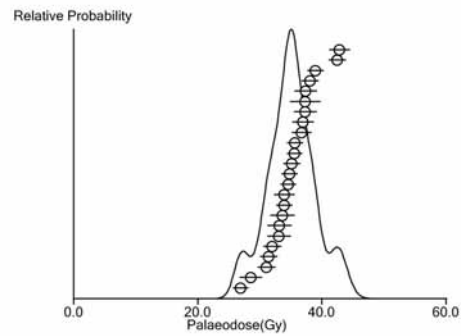
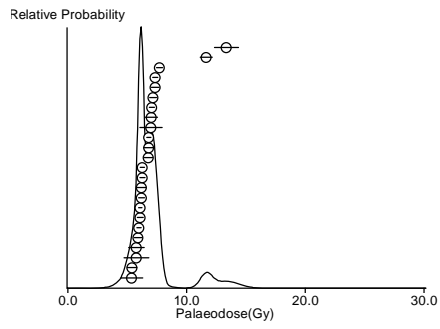


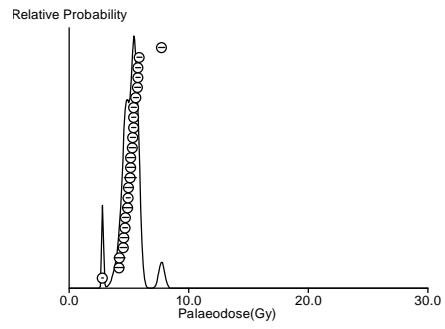
Figure 9a-j (continued overleaf): The distribution of  $D_e$  values for aliquots passing the screening criteria, shown for each OSL sample from field season 2. Note the different x-axis scale for Figure e



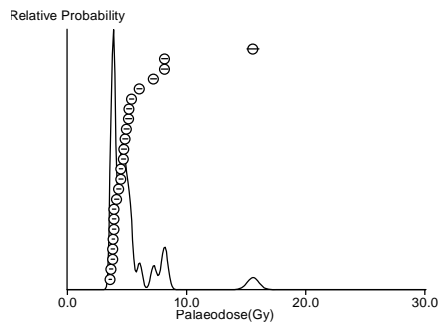
f) LPTR-4A



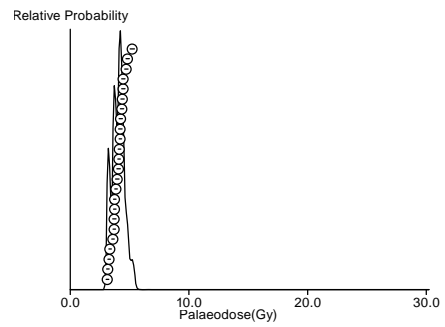
g) LPTR-4B



h) LM10-28-161



i) LM10-28-217



j) LM10-28-277

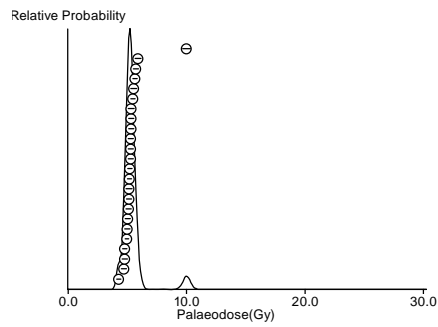


Figure 9 continued: The distribution of  $D_e$  values for aliquots passing the screening criteria, shown for each OSL sample from field season 2. Note the different x-axis scale for Figure e

## OSL age determinations

The equivalent dose ( $D_e$ ) data and the results of laboratory dosimetry measurements were combined for each sample, with corrections being made for attenuation by water and for grain size, to give an OSL age for each of the thirteen dating samples in this study, plus two modern analogue surface samples. These data, including the final age determinations, are presented in detail for each sample in Tables 5–7. For the majority of samples, the error shown for the  $D_e$  determination (Tables 5–7) is the standard error (ie the standard deviation divided by the square root of the number of independent estimates of  $D_e$ ); the standard deviation was used to calculate the error for sample, LPPM1-1 (Table 5). The percentage error on the OSL ages calculated for the majority of intertidal samples (Tables 5–7) is 6%. However, the percentage error on the OSL age for intertidal sample LPPM1-1 is very large, being 25%; this large uncertainty reflects the broad distribution in  $D_e$  values noted in Figure 8c and discussed above. The percentage error on the OSL ages for the core samples from Lower Moors, St Mary's, is between 9 and 13%, but here it is the uncertainty in the water content which is primarily responsible for these values rather than the uncertainty in  $D_e$  values (which varies from 3–6%) (Table 7).

The OSL age data for two modern analogue intertidal surface samples are given in Table 6. These modern samples were taken as a test of how well the samples in this study were likely to be bleached on deposition; if these surface samples give an OSL age of zero years, within errors, then they are considered to be well-bleached on deposition. The samples were taken from the upper ~10mm of the intertidal zone sands. The OSL data gave results equivalent to burial for  $3 \pm 2$  years for both samples (Table 6), suggesting that incomplete bleaching is not a problem for samples in this OSL dating study.

Where more than one OSL age has been determined for a section, the OSL ages generated are in chronostratigraphic order within errors (eg Porth Mellon, samples LPPM-3A and B; Tresco, samples LPTR-4A and B; Lower Moors, samples LM10-28-161 and -217 and -277) (Table 7). The OSL ages generated can also be compared to radiocarbon ages at several sections, listed in Table 8. For six of the seven sections where this comparison can be made, there is excellent agreement between the radiocarbon dates and the OSL ages, with the date ranges generated being in chronostratigraphic order within errors; these sites are Crab's Ledge, Bathinghouse Porth, and LPTR4, all on Tresco, and Porth Mellon, Old Town, and Lower Moors, on St. Mary's (Table 8). For Porth Hellick, St. Mary's, the OSL date range (11650–10270 BC, Table 8) agrees with the two underlying radiocarbon ages determined for twig fragments (9750–9280 BC and 9450–9280 BC, Table 8) within  $2\sigma$  errors (OSL age range 12340–9580 BC). This agreement between the OSL and radiocarbon dates generated validates both techniques, and suggests that both techniques are working well in the environments where the dating has been applied and that the ages generated are reliable.

*Table 5: OSL sample details, equivalent dose, dose rate data, and OSL ages – Field Season 1*

Sample Site	Crab's Ledge, Tresco	Bathinghouse Porth, Tresco	Porth Mellon, St Mary's
<b>Aberystwyth Lab. number</b>	<b>161/LPTR1-1</b>	<b>161/LPTR3-1</b>	<b>161/LPPM1-1</b>
Elevation of top of monolith tin (m OD)	-0.03	0.83	-1.5
Depth down-core (m)	0.08 ± 0.01	0.13 ± 0.01	0.16 ± 0.01
Material used for dating	Quartz		
Grain size (µm)	180–210	180–210	180–210
Preparation method	Heavy liquid separation (sodium polytungstate); 40% HF etch 45 mins		
Measurement protocol	SAR; preheat 200°C/10s, cut heat 160°C; OSL 470nm; detection filter 7.5mm Hoya U-340		
No. aliquots measured	24	24	24
No. aliquots used for D <sub>e</sub>	21	18	21
<b>Equivalent Dose, D<sub>e</sub> (Gy)*</b>	<b>10.86 ± 0.26</b>	<b>10.43 ± 0.30</b>	<b>14.78 ± 3.69</b>
Water content (% dry mass)	25 ± 7	25 ± 7	25 ± 7
Unsealed α count rate (cts/ks.cm <sup>2</sup> )	0.370 ± 0.007	0.456 ± 0.009	0.378 ± 0.004
U (ppm)	2.01 ± 0.17	1.98 ± 0.24	1.80 ± 0.11
Th (ppm)	3.65 ± 0.54	6.26 ± 0.79	4.63 ± 0.35
α count rate Sealed/Unsealed	0.99 ± 0.04	1.02 ± 0.04	1.04 ± 0.03
Infinite β dose rate (Gy/ka)	3.318 ± 0.106	3.701 ± 0.118	3.097 ± 0.099
Calculated K (%)	3.74 ± 0.14	4.15 ± 0.16	3.46 ± 0.13
Layer removed by etching (µm)	10 ± 2	10 ± 2	10 ± 2
External β dose rate 'wet' (Gy/ka)	2.221 ± 0.165	2.477 ± 0.183	2.073 ± 0.154
External β dose rate 'wet' (Gy/ka)	0.761 ± 0.038	1.230 ± 0.062	0.791 ± 0.040
Cosmic (Gy/ka)	0.267 ± 0.027	0.255 ± 0.026	0.248 ± 0.025
<b>Total dose rate (Gy/ka)</b>	<b>3.25 ± 0.17</b>	<b>3.96 ± 0.20</b>	<b>3.11 ± 0.16</b>
<b>OSL Age#* (a)</b>	<b>3340 ± 190</b>	<b>2630 ± 150</b>	<b>4750 ± 1210</b>

# Ages are expressed as years before AD 2010, rounded to the nearest 10 years

\* The error shown following the D<sub>e</sub> value is the standard error on the mean for all samples, except 161/LPPM1-1 which is the standard deviation

*Table 6: OSL sample details, equivalent dose, dose rate data, and OSL ages for modern samples – Field Season 1*

Sample Site	Crab's Ledge, Tresco	Bathinghouse Porth, Tresco
<b>Aberystwyth Lab. number</b>	<b>161/LPTR1-M</b>	<b>161/LPT3-M</b>
Elevation of top of monolith tin (m OD)	-0.03	0.83
Depth down-core (m)	0.005 ± 0.005	0.005 ± 0.005
Material used for dating	Quartz	
Grain size (µm)	180–210	180–210
Preparation method	Heavy liquid separation (sodium polytungstate); 40% HF etch 45 mins	
Measurement protocol	SAR; preheat 200°C/10s, cut heat 160°C; OSL 470nm; detection filter 7.5mm Hoya U-340	
No. aliquots measured	24	24
No. aliquots used for D <sub>e</sub>	21	20
<b>Equivalent Dose, D<sub>e</sub> (Gy)*</b>	<b>0.007 ± 0.006</b>	<b>0.009 ± 0.008</b>
Water content (% dry mass)	25 ± 7	25 ± 7
Unsealed α count rate (cts/ks.cm <sup>2</sup> )	0.473 ± 0.009	0.483 ± 0.009
U (ppm)	2.36 ± 0.23	2.61 ± 0.22
Th (ppm)	5.40 ± 0.75	4.82 ± 0.71
α count rate Sealed/Unsealed	1.00 ± 0.03	1.06 ± 0.04
Infinite β dose rate (Gy/ka)	3.083 ± 0.096	3.779 ± 0.120
Calculated K (%)	3.31 ± 0.13	4.18 ± 0.16
Layer removed by etching (µm)	10 ± 2	10 ± 2
External β dose rate 'wet' (Gy/ka)	2.063 ± 0.152	2.529 ± 0.187
External β dose rate 'wet' (Gy/ka)	0.498 ± 0.025	0.758 ± 0.038
Cosmic (Gy/ka)	0.289 ± 0.029	0.289 ± 0.029
<b>Total dose rate (Gy/ka)</b>	<b>2.85 ± 0.16</b>	<b>3.58 ± 0.19</b>
<b>OSL Age#* (a)</b>	<b>3 ± 2</b>	<b>3 ± 2</b>

# Ages are expressed as years before AD 2010

\* The error shown following the D<sub>e</sub> value is the standard error on the mean

*Table 7: OSL sample details, equivalent dose, dose rate data, and OSL ages – Field Season 2*

Sample Site	Porth Mellon, St Mary's	Porth Mellon, St Mary's	Porth Mellon, St Mary's
<b>Aberystwyth Lab. number</b>	<b>184/LPPM-2</b>	<b>184/LPPM-3A</b>	<b>184/LPPM-3B</b>
Elevation of top of monolith tin (m OD)	-2.30	-0.82	-0.82
Depth down-core (m)	0.11 ± 0.02	0.17 ± 0.02	0.03 ± 0.01
Material used for dating	Quartz		
Grain size (µm)	180–210	180–210	180–210
Preparation method	Heavy liquid separation (sodium polytungstate); 40% HF etch 45 mins		
Measurement protocol	SAR; preheat 220°C/10s, cut heat 160°C; OSL 470nm; detection filter 7.5mm Hoya U-340		
No. aliquots measured	24	24	24
No. aliquots used for D <sub>e</sub>	24	24	24
<b>Equivalent Dose, D<sub>e</sub> (Gy)*</b>	<b>11.27 ± 0.22</b>	<b>13.81 ± 0.44</b>	<b>13.17 ± 0.31</b>
Water content (% dry mass)	25 ± 7	25 ± 7	25 ± 7
Unsealed α count rate (cts/ks.cm <sup>2</sup> )	0.495 ± 0.009	0.445 ± 0.008	0.367 ± 0.007
U (ppm)	2.57 ± 0.23	2.63 ± 0.18	1.99 ± 0.16
Th (ppm)	5.31 ± 0.76	3.66 ± 0.58	3.67 ± 0.53
α count rate Sealed/Unsealed	1.03 ± 0.03	1.02 ± 0.04	1.15 ± 0.03
Infinite β dose rate (Gy/ka)	2.372 ± 0.074	3.240 ± 0.100	3.099 ± 0.100
Calculated K (%)	2.37 ± 0.11	3.52 ± 0.13	3.46 ± 0.13
Layer removed by etching (µm)	10 ± 2	10 ± 2	10 ± 2
External β dose rate 'wet' (Gy/ka)	1.588 ± 0.117	2.169 ± 0.160	2.074 ± 0.154
External β dose rate 'wet' (Gy/ka)	0.586 ± 0.029	0.935 ± 0.047	0.708 ± 0.035
Cosmic (Gy/ka)	0.259 ± 0.026	0.247 ± 0.025	0.285 ± 0.029
<b>Total dose rate (Gy/ka)</b>	<b>2.43 ± 0.12</b>	<b>3.35 ± 0.17</b>	<b>3.07 ± 0.16</b>
<b>OSL Age#* (a)</b>	<b>4630 ± 250</b>	<b>4120 ± 250</b>	<b>4290 ± 250</b>

# Ages are expressed as years before AD 2010, rounded to the nearest 10 years

\* The error shown following the D<sub>e</sub> value is the standard error on the mean for all samples

*Table 7 cont: OSL sample details, equivalent dose, dose rate data, and OSL ages – Field Season 2*

Sample Site	Old Town, St Mary's	Porth Hellick, St Mary's
<b>Aberystwyth Lab. number</b>	<b>184/LPOT-1A</b>	<b>184/LPPH-1A</b>
Elevation of top of monolith tin (m OD)	2.28	-1.27
Depth down-core (m)	0.15 ± 0.01	0.16 ± 0.01
Material used for dating	Quartz	
Grain size (µm)	212-355	180–210
Preparation method	Heavy liquid separation (sodium polytungstate); 40% HF etch 45 mins	
Measurement protocol	SAR; preheat 220°C/10s, cut heat 160°C; OSL 470nm; detection filter 7.5mm Hoya U-340	
No. aliquots measured	24	24
No. aliquots used for D <sub>e</sub>	24	24
<b>Equivalent Dose, D<sub>e</sub> (Gy)*</b>	<b>4.57 ± 0.15</b>	<b>34.90 ± 0.80</b>
Water content (% dry mass)	25 ± 7	25 ± 7
Unsealed α count rate (cts/ks.cm <sup>2</sup> )	0.348 ± 0.006	1.12 ± 0.02
U (ppm)	1.69 ± 0.17	7.87 ± 0.45
Th (ppm)	4.13 ± 0.54	4.94 ± 1.43
α count rate Sealed/Unsealed	1.03 ± 0.03	1.04 ± 0.03
Infinite β dose rate (Gy/ka)	3.490 ± 0.112	2.429 ± 0.079
Calculated K (%)	4.00 ± 0.15	1.47 ± 0.14
Layer removed by etching (µm)	10 ± 2	10 ± 2
External β dose rate 'wet' (Gy/ka)	2.250 ± 0.174	1.626 ± 0.121
External β dose rate 'wet' (Gy/ka)	0.770 ± 0.038	0.816 ± 0.041
Cosmic (Gy/ka)	0.250 ± 0.025	0.248 ± 0.025
<b>Total dose rate (Gy/ka)</b>	<b>3.27 ± 0.18</b>	<b>2.69 ± 0.13</b>
<b>OSL Age#* (a)</b>	<b>1400 ± 90</b>	<b>12970 ± 690</b>

# Ages are expressed as years before AD 2010, rounded to the nearest 10 years

\* The error shown following the D<sub>e</sub> value is the standard error on the mean for all samples

*Table 7 continued: OSL sample details, equivalent dose, dose rate data, and OSL ages – Field Season 2*

Sample Site	Tresco	Tresco
<b>Aberystwyth Lab. number</b>	<b>184/LPTR-4A</b>	<b>184/LPTR-4B</b>
Elevation of top of monolith tin (m OD)	1.84	1.84
Depth down-core (m)	0.19 ± 0.01	0.05 ± 0.01
Material used for dating	Quartz	
Grain size (µm)	180–210	180–210
Preparation method	Heavy liquid separation (sodium polytungstate); 40% HF etch 45 mins	
Measurement protocol	SAR; preheat 220°C/10s, cut heat 160°C; OSL 470nm; detection filter 7.5mm Hoya U-340	
No. aliquots measured	24	24
No. aliquots used for D <sub>e</sub>	24	24
<b>Equivalent Dose, D<sub>e</sub> (Gy)*</b>	<b>6.61 ± 0.19</b>	<b>4.57 ± 0.27</b>
Water content (% dry mass)	25 ± 7	25 ± 7
Unsealed α count rate (cts/ks.cm <sup>2</sup> )	0.361 ± 0.006	0.261 ± 0.004
U (ppm)	1.62 ± 0.18	1.29 ± 0.11
Th (ppm)	4.74 ± 0.58	3.03 ± 0.37
α count rate Sealed/Unsealed	0.98 ± 0.03	1.03 ± 0.04
Infinite β dose rate (Gy/ka)	3.420 ± 0.109	3.463 ± 0.111
Calculated K (%)	3.91 ± 0.15	4.08 ± 0.14
Layer removed by etching (µm)	10 ± 2	10 ± 2
External β dose rate 'wet' (Gy/ka)	2.289 ± 0.170	2.318 ± 0.172
External β dose rate 'wet' (Gy/ka)	0.956 ± 0.048	0.722 ± 0.036
Cosmic (Gy/ka)	0.243 ± 0.024	0.282 ± 0.028
<b>Total dose rate (Gy/ka)</b>	<b>3.49 ± 0.18</b>	<b>3.32 ± 0.18</b>
<b>OSL Age#* (a)</b>	<b>1890 ± 110</b>	<b>1380 ± 110</b>

# Ages are expressed as years before AD 2010, rounded to the nearest 10 years

\* The error shown following the D<sub>e</sub> value is the standard error on the mean for all samples

Table 7 continued: OSL sample details, equivalent dose, dose rate data, and OSL ages – Field Season 2

Sample Site	Lower Moors, St Mary's	Lower Moors, St Mary's	Lower Moors, St Mary's
<b>Aberystwyth Lab. number</b>	<b>184/LM10-28-161</b>	<b>184/LM10-28-217</b>	<b>184/LM10-28-277</b>
Elevation of top of monolith tin (m OD)	2.16	2.16	2.16
Depth down-core (m)	1.63 ± 0.02	2.19 ± 0.02	2.74 ± 0.03
Material used for dating	Quartz		
Grain size (µm)	63-90	63-90	63-90
Preparation method	Heavy liquid separation (sodium polytungstate); 40% HF etch 45 mins		
Measurement protocol	SAR; preheat 220°C/10s, cut heat 160°C; OSL 470nm; detection filter 7.5mm Hoya U-340		
No. aliquots measured	24	24	24
No. aliquots used for D <sub>e</sub>	24	24	24
<b>Equivalent Dose, D<sub>e</sub> (Gy)*</b>	<b>4.48 ± 0.26</b>	<b>3.89 ± 0.11</b>	<b>5.25 ± 0.15</b>
Water content (% dry mass)	75 ± 25	110 ± 25	50 ± 25
Unsealed α count rate (cts/ks.cm <sup>2</sup> )	0.499 ± 0.009	0.560 ± 0.008	0.596 ± 0.010
U (ppm)	2.78 ± 0.22	3.32 ± 0.19	3.62 ± 0.23
Th (ppm)	4.68 ± 0.71	4.60 ± 0.60	4.59 ± 0.74
α count rate Sealed/Unsealed	0.95 ± 0.03	0.97 ± 0.03	0.96 ± 0.03
Infinite β dose rate (Gy/ka)	1.732 ± 0.058	1.776 ± 0.059	1.634 ± 0.055
Calculated K (%)	1.53 ± 0.09	1.49 ± 0.09	1.26 ± 0.09
Layer removed by etching (µm)	10 ± 2	10 ± 2	10 ± 2
External β dose rate 'wet' (Gy/ka)	0.830 ± 0.137	0.694 ± 0.094	0.934 ± 0.182
External β dose rate 'wet' (Gy/ka)	0.490 ± 0.079	0.424 ± 0.057	0.594 ± 0.112
Cosmic (Gy/ka)	0.170 ± 0.017	0.159 ± 0.016	0.148 ± 0.015
<b>Total dose rate (Gy/ka)</b>	<b>1.49 ± 0.16</b>	<b>1.28 ± 0.11</b>	<b>1.68 ± 0.22</b>
<b>OSL Age#* (a)</b>	<b>3000 ± 370</b>	<b>3050 ± 280</b>	<b>3130 ± 410</b>

# Ages are expressed as years before AD 2010, rounded to the nearest 10 years

\* The error shown following the D<sub>e</sub> value is the standard error on the mean for all samples



*Table 8: Comparison of radiocarbon and OSL ages for individual sections; samples are listed in depth order for each monolith or core shown. The radiocarbon ages are reproduced from Marshall et al (2013)*

Laboratory code	Sample name and depth	Identification	$\delta^{13}\text{C}$ (‰)	Radiocarbon age (BP)	$^{14}\text{C}$ fM	OSL age (years before 2010)	Date range*	Notes
Crab's Ledge, Tresco								
OxA-22578	LPTR1: 1–2cm	bulk unidentified plant macrofossils	-23.6	2896 ±26		-	1200–1000 cal BC	Uppermost sample with high saltmarsh pollen content
OxA-22579	LPTR1: 5–6cm	bulk unidentified plant macrofossils	-25.6	2824 ±29		-	1060–900 cal BC	Above OSL sample
161/LPTR1-1	LPTR1-1: 7–9cm	180–210µm quartz	-	-		3340 ±190	1520–1140 BC	
OxA-22580	LPTR1: 7–8cm	bulk unidentified plant macrofossils	-24.9	2953 ±39		-	1310–1020 cal BC	Below OSL sample
SUERC-28999	LPTR1: 13–14cm	bulk organic sediment (humic acid fraction#)	-24.7	3090 ±30		-	1430–1270 cal BC	Lowermost sample with high saltmarsh pollen content
Bathinghouse Porth, Tresco								
SUERC-29000	LPTR3: 2–3cm	bulk organic sediment (humic acid fraction#)	-23.2	2435 ±30		-	760–400 cal BC	Uppermost sample with high saltmarsh pollen
SUERC-29001	LPTR3: 10–11cm	bulk organic sediment (humic acid fraction#)	-23.5	2465 ±30		-	770–410 cal BC	Above OSL sample
161/LPTR3-1	LPTR3-1: 12–14cm	180–210µm quartz	-	-		2630 ±150	770–470 BC	
SUERC-29002	LPTR3: 22–23cm	bulk organic sediment (humic acid fraction#)	-20.4	2620 ±30		-	830–770 cal BC	Lowermost sample with high saltmarsh pollen

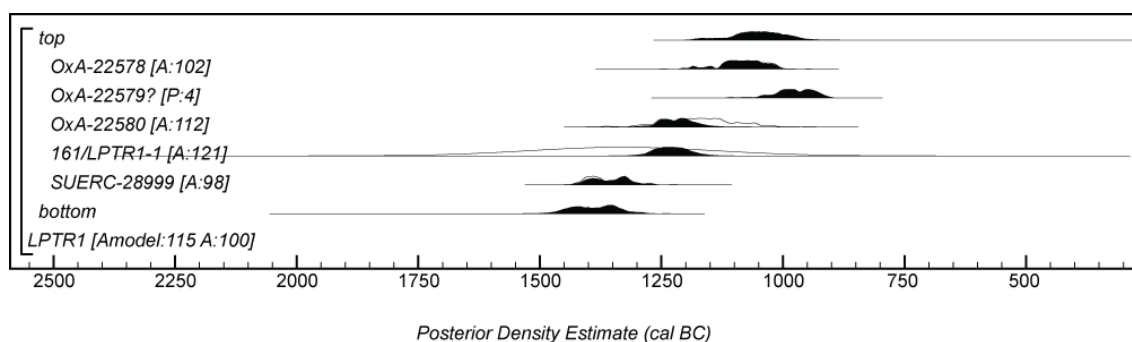
Laboratory code	Sample name and depth	Identification	$\delta^{13}\text{C}$ (‰)	Radiocarbon age (BP)	$^{14}\text{C}$ fM	OSL age (years before 2010)	Date range*	Notes
Porth Mellon, St. Mary's								
184/LPPM-2	LPPM-2: 9–13cm	180–210 $\mu\text{m}$ quartz	-	-		4630 $\pm$ 250	2870–2370 BC	
OxA-23859	LPPM2: 23cm	plant fragment: Monocot stem	-24.7	4269 $\pm$ 38		-	2920–2870 cal BC	
Old Town, St. Mary's								
OxA-23827	LPOT1: 4–5cm	bulk: mix of herbaceous and Monocot stems (humin fraction)	-25.9	1469 $\pm$ 25		-	cal AD 540–650	
OxA-23862	LPOT1: 4–5cm	bulk: mix of herbaceous and Monocot stems (humic acid fraction)	-27.6	1632 $\pm$ 28		-	cal AD 350–540	
184/LPOT-1A	LPOT-1A: 14–16cm	180–210 $\mu\text{m}$ quartz		1400 $\pm$ 90		AD 520–700		
SUERC-32920	LPOT1: 22–23cm	bulk: organic sediment including Phragmites stem and ?Monocot stem (humic acid fraction)	-28.9	1980 $\pm$ 30		-	50 cal BC–cal AD 80	
SUERC-32921	LPOT1: 22–23cm	bulk: organic sediment including Phragmites stem and ?Monocot stem (humin fraction)	-28.7	2065 $\pm$ 30		-	180 cal BC–cal AD 10	
Porth Hellick, St. Mary's								
184/LPPH-1A	LPPH-1A: 15–17cm	180–210 $\mu\text{m}$ quartz		12970 $\pm$ 690		11650–10270 BC		
OxA-23858	LPPH1: 19cm A	wood: single twig fragment	-28.5	9945 $\pm$ 60		-	9750–9280 cal BC	
SUERC-32915	LPPH1: 24cm	wood: unidentified twig	-30.1	9915 $\pm$ 35		-	9450–9280 cal BC	
LPTR4, Tresco								
184/LPTR-4B	LPTR-4B: 4–6cm	180–210 $\mu\text{m}$ quartz		1380 $\pm$ 110		AD 520–740		
SUERC-32927	LPTR4: 8.5–9.5cm	bulk: organic sediment with ?Monocot roots (humic acid fraction)	-26.5	1390 $\pm$ 30		-	cal AD 600–670	
SUERC-32993	LPTR4: 8.5–9.5cm	bulk: organic sediment with ?Monocot roots (humin fraction)	-25.0	1595 $\pm$ 30		-	cal AD 400–550	
184/LPTR-4A	LPTR-4A: 18–20cm	180–210 $\mu\text{m}$ quartz		1890 $\pm$ 110		AD 10-230		

Laboratory code	Sample name and depth	Identification	$\delta^{13}\text{C}$ (‰)	Radiocarbon age (BP)	$^{14}\text{C}$ fM	OSL age (years before 2010)	Date range*	Notes
Lower Moors, St. Mary's								
SUERC-39449	LM10/28- 25–26cm	peat, humic fraction	-29.6	-	102.47 ±0.48	-	cal AD 1955–1957	
SUERC-39452	LM10/28- 39–40cm	peat, humic fraction	-28.3	689 ±38		-	cal AD 1260–1390	
Beta-351909	LM10/28- 120–121cm	organic sediment, humin fraction	-26.1	730±30		-	cal AD 1250–1300	
184/LM10-28-161	LM10/28- 161–165cm	63–90µm quartz	-	-		3000 ±370	1360–620 BC	
SUERC-39453	LM10/28- 162–163cm	organic silt, humic fraction	-28.3	2481 ±38		-	790–410 cal BC	
184/LM10-28-217	LM10/28- 217–221cm	63–90 µm quartz	-	-		3050 ±280	1320–760 BC	
SUERC-39454	LM10/28- 218–219cm	organic silt, humic fraction		2560 ±38		-	810–550 cal BC	
Beta-301603	LM10/28- 257–259cm	silt with fine sand, humin fraction	-25.1	2720 ±30		-	930–800 cal BC	
184/LM10-28-277	LM10/28- 271–277cm	63–90µm quartz	-	-		3130 ±410	1530–710 BC	

\*Calibrated radiocarbon dates (cal AD/cal BC) are quoted at 95% confidence limit. OSL dates (AD/BC) are shown with  $1\sigma$  error derived as shown in Tables 5–7.

## Bayesian age-depth modelling

The OSL ages generated can also be incorporated with radiocarbon dates in Bayesian age-depth models (Bronk Ramsey 2008, Parnell *et al* 2011) at several sections, listed in Table 8. For six of the seven sections with OSL ages and radiocarbon dates, there is good agreement between the radiocarbon dates, the OSL ages and their stratigraphic position (Figs 10–15); these sites are Crab's Ledge, Bathinghouse Porth, and LPTR4, all on Tresco, and Porth Mellon, Old Town, and Lower Moors, on St. Mary's. For Porth Hellick, St. Mary's, the OSL age is not in agreement with its stratigraphic position (Fig 16) – if the two underlying radiocarbon dates accurately date their positions in the sequence. Overall the excellent agreement between the OSL and radiocarbon dates generated validates both techniques, and suggests that both techniques are working well in the environments where the dating has been applied and that the ages generated are reliable.



*Figure 10: Bayesian age-depth model of the chronology of the sediment sequence at Crab's Ledge, Tresco, LPTR1 (P\_Sequence model ( $k=0.01-100$ ); Bronk Ramsey 2008). For each of the radiocarbon dates two distributions have been plotted, one in outline, which is the result of simple calibration, and a solid one, which is based on the chronological model used. For each OSL result there are also two distributions, one in outline – the age, and a solid one which is also based on the chronological model. Figures in brackets after the laboratory numbers are the individual indices of agreement which provide an indication of the consistency of the radiocarbon and OSL dates with the prior information included in the model (Bronk Ramsey 1995)*

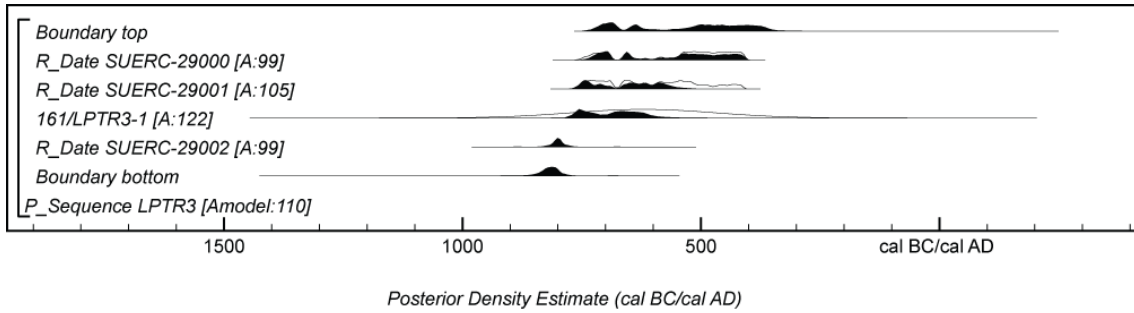


Figure 11: Bayesian age-depth model of the chronology of the sediment sequence at Bathinghouse Porth, Tresco, LPTR3 ( $P_{\text{Sequence}}$  model ( $k=0.01-100$ ); Bronk Ramsey 2008). The format is identical to Figure 10

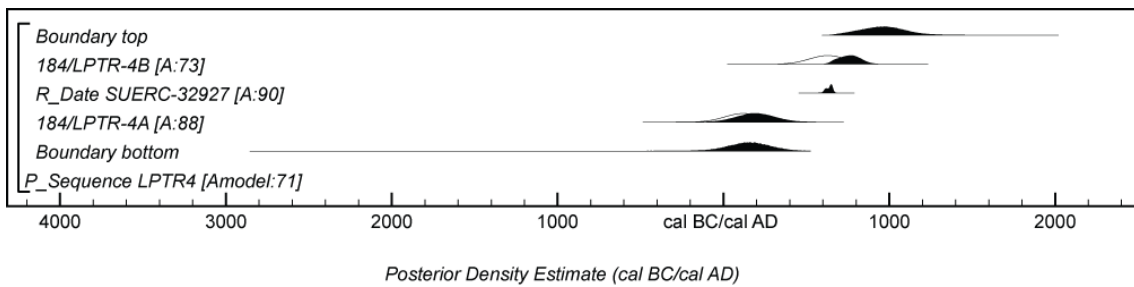


Figure 12: Bayesian age-depth model of the chronology of the sediment sequence at Tresco, LPTR4 ( $P_{\text{Sequence}}$  model ( $k=0.01-100$ ); Bronk Ramsey 2008). The format is identical to Figure 10

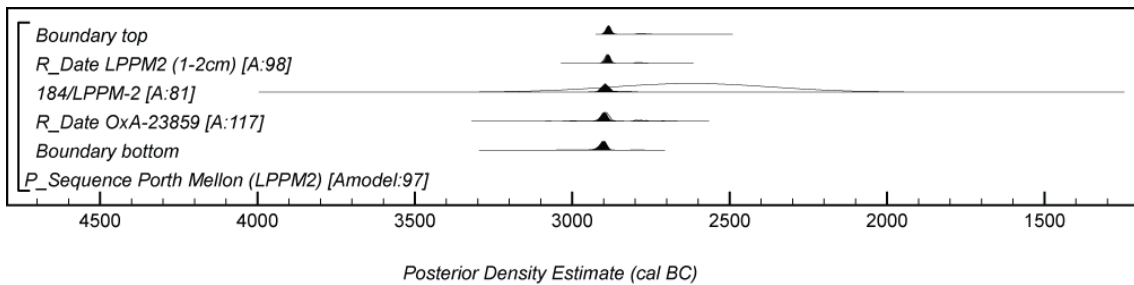


Figure 13: Bayesian age-depth model of the chronology of the sediment sequence at Porth Mellon, LPPM2 ( $P_{\text{Sequence}}$  model ( $k=0.01-100$ ); Bronk Ramsey 2008). The format is identical to Figure 10

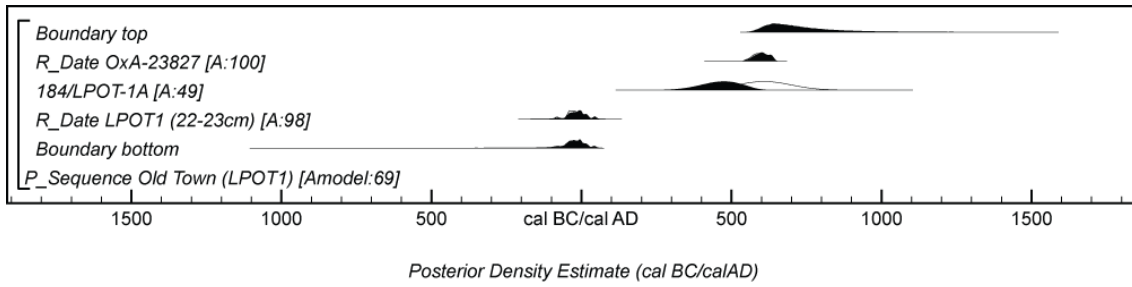


Figure 14: Bayesian age-depth model of the chronology of the sediment sequence at Old Town, LPOT1 ( $P_{\text{Sequence}}$  model ( $k=0.01-100$ ); Bronk Ramsey 2008). The format is identical to Figure 10

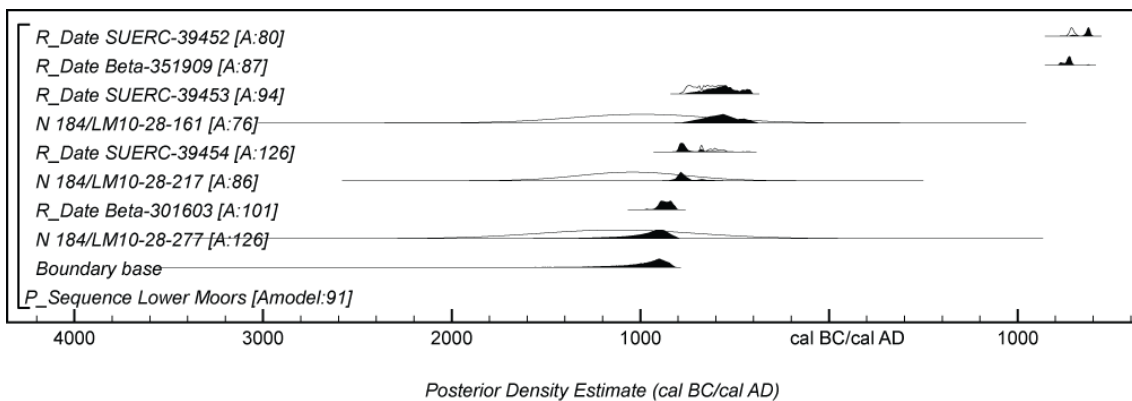


Figure 15: Bayesian age-depth model of the chronology of the sediment sequence at Lower Moors, St Mary's ( $P_{\text{Sequence}}$  model ( $k=0.01-100$ ); Bronk Ramsey 2008). The format is identical to Figure 10. The 'modern' date (SUERC-39449) from 25cm has been excluded

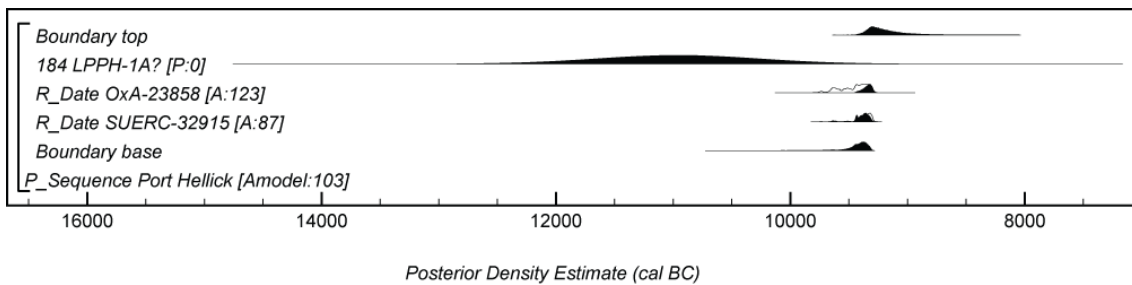


Figure 16 Bayesian age-depth model of the chronology of the sediment sequence at Port Hellick, LPPH, ( $P_{\text{Sequence}}$  model ( $k=0.01-100$ ); Bronk Ramsey 2008). The format is identical to Figure 10 apart from the measurement that has been excluded from the analysis that is shown with a ?

## SUMMARY AND CONCLUSIONS

Intertidal sand units from St Mary's and Tresco were dated using OSL applied to coarse-grained quartz. A total of five samples were taken for the first stage of OSL dating following field season 1; three samples were grey sand units found within intertidal 'peat' units, and two modern intertidal sand samples were also examined. In field season 2, a further ten samples were taken for OSL dating; seven from monolith tins removed from intertidal settings on St. Mary's and Tresco, and three from a core taken from Lower Moors, St. Mary's. The OSL measurement procedure employed was the Single Aliquot Regenerative dose (SAR) protocol which corrects for sensitivity change. The SAR measurement protocol identified as suitable for dating used a mid-range preheat temperature of 200 or 220°C held for 10s and a test dose cut-heat temperature of 160°C with immediate cooling. Several checks and screening criteria were applied to the OSL dating aliquots 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 OSL data and quality control checks suggest that the final OSL ages generated for the intertidal sediments are typically both accurate and of relatively high precision (6%), with the exception being sample LPPM1-1, which showed a broad  $D_e$  distribution apparent in the relatively large uncertainty (25%) on the final age determination. The two modern samples gave ages of  $3 \pm 2$  years, indicating that the intertidal sands should typically be well-bleached on deposition. The percentage error on the OSL ages for the core samples from Lower Moors, St Mary's, is between 9 and 13%, but here it is the uncertainty in the water content that is primarily responsible for these values rather than the uncertainty in  $D_e$  values (which varies from 3–6%).

The OSL ages generated are in agreement (within  $1\sigma$  error) with other OSL or radiocarbon ages, giving sequences of dates that are in chronostratigraphic order; the only exception to this is Porth Hellick, St. Mary's, site LPPH1 where the OSL date range (11650–10270 BC) agrees with the two underlying radiocarbon dates determined for twig fragments (9750–9280 BC and 9450–9280 BC) within  $2\sigma$  errors (ie OSL range 12340–9580 BC). The agreement between the OSL and radiocarbon dates validates both methods, indicating that both methods are working well in the depositional settings and for the materials examined in this study, and generating reliable ages suitable for informing the evolution of the coastal and marine environment of the Isles of Scilly.

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