Scottish Universities Environmental Research Centre

Luminescence dating of wind-blown sands from the Broo Peninsula, Shetland

Luminescence Laboratory Report

December 2012

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Summary

This report provides a temporal framework to support University of Stirling geo-archaeological investigations near Huesbreck, Broo Pennisula (Shetland), examining how the early modern population there, adapted to harsh climate conditions in the 18-19th centuries, when enhanced aeolian activity led to an influx of sand to the area, leading to adaption's in farming practices, and abandonment of several sites. Five sediment samples were submitted to the luminescence laboratories at SUERC for dating.

All samples were subjected to laboratory preparation of sand-sized quartz, and purity checked using scanning electron microscopy. Dose rates for the bulk sediment were evaluated using analyses of the uranium, thorium and potassium concentrations obtained by high resolution gamma spectrometry coupled with beta dose rate measurement using thick source beta counting, and in situ field gamma spectroscopy. Equivalent doses were determined by OSL from 32 aliquots of quartz per sample using the quartz single-aliquot-regenerative (SAR) procedure. The material exhibited good OSL sensitivity and produced acceptable SAR internal quality control performance. Radial plotting methods revealed good internal homogeneity in the dose distributions obtained for each sample.

The chronology established for the sampled sands on the site spans from the mid 16th century (AD 1540 ± 40; SUTL2441) through to the early 19th century (AD 1810 ± 25), with the dates falling within three clusters - the waning stages of the Little Ice Age, the mid 18th century (AD 1730 ± 25 to 1760 ± 25) and the early 19th century (AD 1810 ± 25). In the wider region, periods of sand movement and deposition in the mid 18th century, and early to late 18th century, are documented in sediment statigraphies sectioned at the Old Scatness Broch, Scatness.

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1. Introduction

The report is concerned with optically stimulated luminescence (OSL) investigations of five sediment samples collected from sands enclosing an early-modern structure, near Huesbreck, Broo Pennisula, Shetland. The OSL dates provide the temporal framework to support the University of Stirling's geo-archaeological investigations at the site, which are concerned with the communities resistant to harsh climatic variations in the 18th-19th centuries, associated with major sand blows, and the deposition of thick sequences of sands.

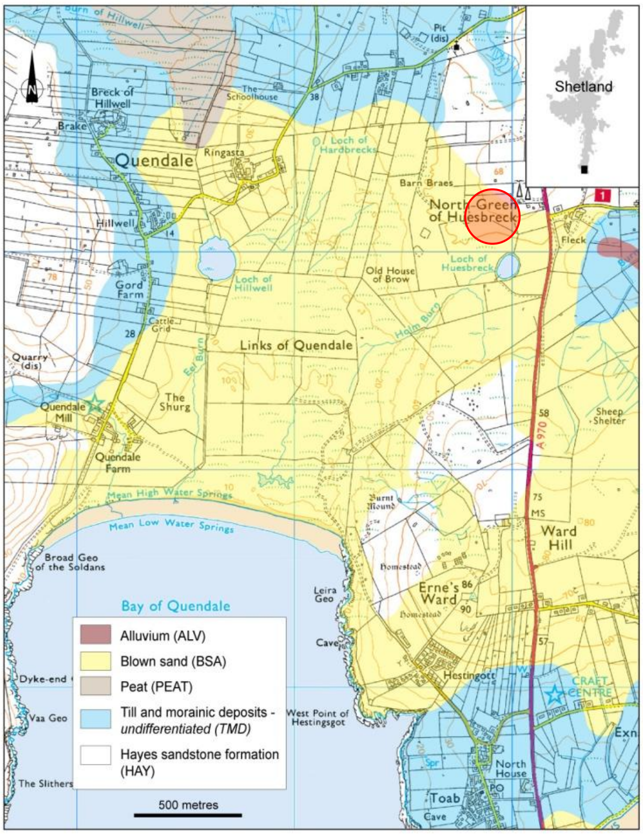

Figure 1-1: Location map, University of Stirling geo-archaeological investigations at Huesbreck, Broo Pennisula, Shetland

2. Sampling

Sampling was undertaken by Ian Simpson during the summer of 2012. Photographs of the sediment stratigraphies are reproduced in figure 2-1. Sample submission forms are reproduced in Appendix A.

Geoarchaeology trench 1 (SUTL2441 and 2442)

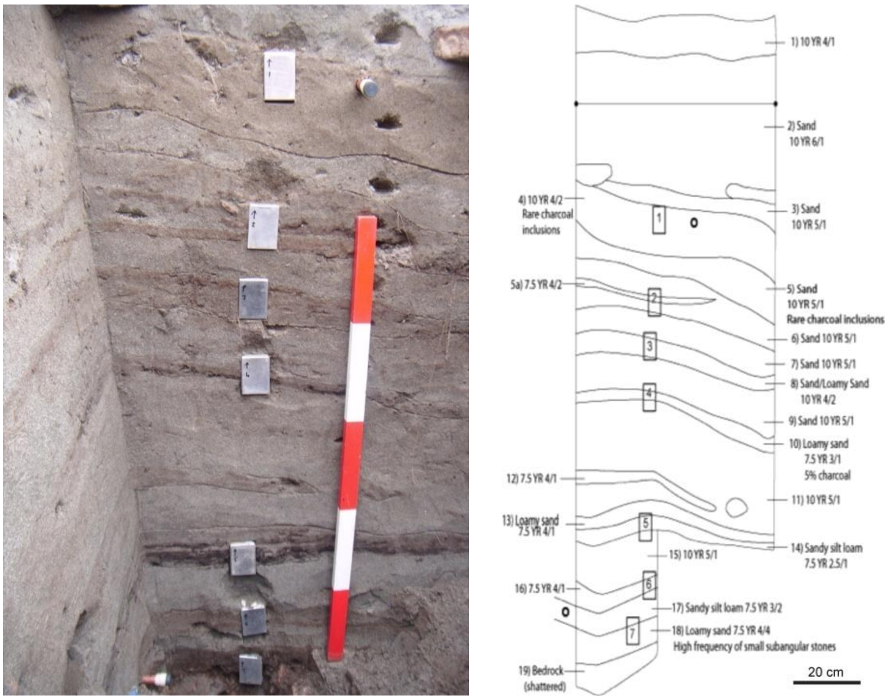

Figure 2-1: Geo-archaeological trench 1, OSL samples SUTL2441 and 2442

Samples were submitted to the luminescence laboratories at the Scottish Universities Environmental Research Centre (SUERC) for dating in two batches, in April and October of 2012. Sample numbers, contexts, and unique laboratory code (assigned on receipt) are listed in Table 2-1.

SUTL no.	Field no.	Depth (cm)	Context	Significance
2441	Section 1, OSL1	196		
2442	Section 1, OSL2	30		
2517	OSL 1, Enclosure	-	Sheet sand (wind-blown); enclosed area immediately east of the excavated Broo site	provide terminus ante quem for abandonment, and an upper constraint on the age of the soil horizon in this section
2518	OSL 2, Enclosure	-		
2519	OSL 3, Outer	-	Sheet sand (wind-blown); unenclosed area immediately south-west of the excavated Broo site	

Table 2-1: SUTL sample reference numbers

3. Quartz SAR measurements

3.1. Sample preparation

All sample handling and preparation was conducted under safelight conditions in the SUERC luminescence dating laboratories.

3.1.1. Water contents

Bulk samples were weighed, saturated with water and re-weighed. Following oven drying at 50 °C to constant weight, the actual and saturated water contents were determined as fractions of dry weight. These data were used, together with information on field conditions to determine water contents and an associated water content uncertainty for use in dose rate determination.

3.1.2. HRGS and TSBC Sample Preparation

Bulk quantities of material, weighing c. 50 g, were removed from each full dating sample for environmental dose rate determinations, including high-resolution gamma spectrometry (HRGS) and thick source beta counting (TSBC; Sanderson, 1988). This material was placed in an oven to dry to constant weight. From each of the full-dating samples, 20 g of material was temporary removed and used in TSBC. This material was then returned to the original sub-sample, placed in a HDPE pot, sealed with epoxy resin and left for 3 weeks prior to HRGS measurement to allow equilibration of ^{222}Rn daughters. In addition, 100 g samples of bulk material collected from a 30 cm radius around each full dating position, were prepared for HRGS measurement.

3.1.3. SAR Sample Preparation

Approximately 20g of material was removed for each tube and processed for luminescence measurements, to separate sand-sized quartz and feldspar grains. The sample was wet sieved to obtain the 90-150 and 150-250 µm fractions. The 150-250 µm sub-sample was treated with 1 M hydrochloric acid (HCl) for 10 minutes, 15% hydrofluoric acid (HF) for 15 minutes, and 1 M HCl for a further 10 minutes. This etched material was then centrifuged in sodium polytungstate solutions of ~2.51, 2.58, 2.62, and 2.74 g cm^{-3}, to obtain concentrates of potassium-rich feldspars (2.51-2.58 g cm^{-3}), sodium feldspars (2.58-2.62 g cm^{-}3) and quartz plus plagioclase (2.62-2.74 g cm^{-3}). The selected quartz fraction was then subjected to further HF and HCl washes (40% HF for 40mins, followed by 1M HCl for 10 mins). All materials were dried at 50°C and transferred to Eppendorf tubes. 32 aliquots were produced for each sample.

3.2. Measurements and determinations

3.2.1. Dose rate determinations

Dose rates were measured in the laboratory using HRGS and TSBC. Full sets of dose rate determinations were made for samples SUTL2508 to SUTL2509, and SUTL2511 to SUTL2513.

HRGS measurements were performed using a 50% relative efficiency "n" type hyper-pure Ge detector (EG&G Ortec Gamma-X) operated in a low background lead shield with a copper liner. Gamma ray spectra were recorded over the 30 keV to 3 MeV range from each sample, interleaved with background measurements and measurements from SUERC Shap Granite standard in the same geometries. Counting times of 50-80ks per sample were used. The spectra were analysed to determine count rates from the major line emissions from ^{40}K (1461 keV), and from selected nuclides in the U decay series (^{234}Th, ^{226}Ra + ^{235}U, ^{214}Pb, ^{214}Bi and ^{210}Pb) and the Th decay series (^{228}Ac, ^{212}Pb, ^{208}Tl) and their statistical counting uncertainties. Net rates and activity concentrations for each of these nuclides were determined relative to Shap Granite by weighted combination of the individual lines for each nuclide. The internal consistency of nuclide specific estimates for U and Th decay series nuclides was assessed relative to measurement precision, and weighted combinations used to estimate mean activity concentrations (Bq kg^{-1}) and elemental concentrations (% K and ppm U, Th) for the parent activity. These data were used to determine infinite matrix dose rates for alpha, beta and gamma radiation.

Beta dose rates were also measured directly using the SUERC TSBC system (Sanderson, 1988). Sample count rates were determined with six replicate 600 s counts for each sample, bracketed by background measurements and sensitivity determinations using the Shap Granite secondary reference material. Infinite-matrix dose rates were calculated by scaling the net count rates of samples and reference material to the working beta dose rate of the Shap Granite (6.25 ± 0.03 mGy a^{-1}). The estimated errors combine counting statistics, observed variance and the uncertainty on the reference value.

The dose rate measurements were used in combination with the assumed burial water contents, to determine the overall effective dose rates for age estimation. Cosmic dose rates were evaluated by combining latitude and altitude specific dose rates (0.181 ± 0.01 mGy a^{-1}) for the site with corrections for estimated depth of overburden using the method of Prescott and Hutton (1994).

3.2.2. SAR luminescence measurements

All measurements were conducted using a Risø DA-15 automatic reader equipped with a ^{90}Sr/^{90}Y β-source for irradiation, blue LEDs emitting around 470 nm and infrared (laser) diodes emitting around 830 nm for optical stimulation, and a U340 detection filter pack to detect in the region 270-380 nm, while cutting out stimulating light (Bøtter-Jensen et al., 2000). For each sample, equivalent dose determinations were made on sets of 32 aliquots per sample, using a single aliquot regeneration (SAR) sequence (cf Murray and Wintle, 2000). According to this procedure, the OSL signal level from an individual disc is calibrated to provide an absorbed dose estimate (the equivalent dose) using an interpolated dose-response curve, constructed by regenerating OSL signals by beta irradiation in the laboratory. Sensitivity changes which may occur as a result of readout, irradiation and preheating (to remove unstable radiation-induced signals) are monitored using small test doses after each regenerative dose. Each measurement is standardised to the test dose response determined immediately after its readout, to compensate for observed changes in sensitivity during the laboratory measurement sequence. For the purposes of interpolation, the regenerative doses are chosen to encompass the likely value of the equivalent (natural) dose (determined in the initial laboratory characterisation study, see section 4). A repeat dose point is included to check the ability of the SAR procedure to correct for laboratory-induced sensitivity changes (the ‘recycling test’), a zero dose point is included late in the sequence to check for thermally induced charge transfer during the irradiation and preheating cycle (the ‘zero cycle’), and an IR response check is included to assess the magnitude of non-quartz signals. Regenerative dose response curves were constructed using doses of 1, 2.5, 5 and 10 Gy, with a test dose of 2 Gy.

3.3. Results

3.3.1. Dose rates

HRGS results are shown in Table 3-1, both as activity concentrations (i.e. disintegrations per second per kilogram) and as equivalent parent element concentrations (in % and ppm), based in the case of U and Th on combining nuclide specific data assuming decay series equilibrium. K, U and Th concentrations ranged between 1.4 and 2.1 %, 0.8 and 1.5 ppm and 6.4 to 7.8 ppm, respectively.

SUTL no.	Activity Concentration ($Bq\ kg^{-1}$)[a]			Equivalent Concentration[b]		
	^{40}K	U	Th	K (%)	U (ppm)	Th (ppm)
2441	607 ± 18	12 ± 1	26 ± 1	1.96 ± 0.06	0.98 ± 0.11	6.44 ± 0.34
2442	608 ± 19	15 ± 1	29 ± 1	1.96 ± 0.06	1.18 ± 0.12	7.10 ± 0.37
2517	611 ± 23	10 ± 2	28 ± 2	1.98 ± 0.07	0.82 ± 0.16	6.85 ± 0.53
2517B	578 ± 21	18 ± 2	29 ± 2	1.87 ± 0.07	1.46 ± 0.17	7.22 ± 0.44
2518	622 ± 22	14 ± 2	30 ± 2	2.01 ± 0.07	1.12 ± 0.17	7.30 ± 0.55
2518B	590 ± 21	13 ± 2	30 ± 2	1.91 ± 0.07	1.08 ± 0.18	7.28 ± 0.47
2519	431 ± 20	13 ± 1	26 ± 1	1.39 ± 0.06	1.09 ± 0.11	6.46 ± 0.35
2519B	643 ± 22	14 ± 2	32 ± 2	2.08 ± 0.07	1.16 ± 0.19	7.81 ± 0.47

Table 3-1: Activity and equivalent concentrations of K, U and Th determined by HRGS
[a]Shap granite reference, working values determined by David Sanderson in 1986, based on HRGS relative to CANMET and NBL standards.
[b]Activity and equivalent concentrations for U, Th and K determined by HRGS (Conversion factors based on NEA (2000) decay constants): 40K: 309.3 $Bq\ kg^{-1}\ \%K^{-1}$, 238U: 12.35 $Bq\ kg^{-1}\ ppmU^{-1}$, 232Th: 4.057 $Bq\ kg^{-1}\ ppm\ Th^{-1}$.

Infinite matrix alpha, beta and gamma dose rates from HRGS are listed for all samples in Table 3-2, together with infinite matrix beta dose rates from TSBC and in situ gamma dose rates from FGS. The environmental dose rates measured in the field range between 0.76 ± 0.07 mGy a^{-1} and 0.85 ± 0.07 mGy a^{-1} in the first section and 0.60 ± 0.05 and 0.72 ± 0.06 mGy a^{-1} in the second. Gamma dose rates, as measured on dry samples in the laboratory, ranged between 0.79 ± 0.03 to 1.04 ± 0.04 mGy a^{-1}, with a mean value of 0.95 ± 0.07 mGy a^{-1}.

Beta dose rates measured by HRGS ranged between 1.50 ± 0.06 to 2.12 ± 0.07 mGy a^{-1}, with a mean value of 1.94 ± 0.19 mGy a^{-1}. TSBC beta dose rate estimates ranged between 1.84 ± 0.07 to 2.07 ± 0.07 mGy a^{-1}, with a mean value of 1.99 ± 0.08 mGy a^{-1}. It is noted that there is a good agreement between the beta dose rates determined by HRGS and TSBC.

SUTL no.	HRGS, dry (mGy a^{-1})[a] Alpha	Beta	Gamma	TSBC, dry (mGy a^{-1})	FGS, wet (mGy a^{-1})
2441	7.50 ± 0.39	1.96 ± 0.05	0.92 ± 0.03	1.90 ± 0.05	0.76 ± 0.07
2442	8.53 ± 0.42	2.01 ± 0.05	0.97 ± 0.03	2.05 ± 0.06	0.85 ± 0.07
2517	7.35 ± 0.60	1.96 ± 0.07	0.92 ± 0.04	2.02 ± 0.07	0.65 ± 0.05
2517B	9.39 ± 0.58	1.97 ± 0.06	0.99 ± 0.03	1.96 ± 0.07	-
2518	8.50 ± 0.62	2.04 ± 0.07	0.99 ± 0.04	1.84 ± 0.07	0.72 ± 0.06
2518B	8.37 ± 0.6	1.95 ± 0.06	0.96 ± 0.04	2.03 ± 0.07	-
2519	7.80 ± 0.39	1.50 ± 0.06	0.79 ± 0.03	2.07 ± 0.07	0.60 ± 0.05
2519B	8.99 ± 0.62	2.12 ± 0.07	1.04 ± 0.04	2.03 ± 0.07	-

Table 3-2: Infinite matrix dose rates determined by HRGS and TSBC.
[a]based on dose rate conversion factors in Aikten (1983)

The water content measurements with assumed values for the average water content during burial are given in Table 3-3. The table also lists the gamma dose rate from the HRGS after application of a water content correction. Effective dose rates to the HF etched 200 µm quartz grains are given for the gamma dose rate and beta dose rate (the mean of the TSBC and HRGS data, accounting for water content and grain size).

SUTL No.	Water Content (%)			Effective Dose Rate (mGy a^{-1})		
	Fractional	Saturated	Assumed	Beta[a]	Gamma	Total[b]
2441	13.9	21.2	17 ± 4	1.45 ± 0.08	0.76 ± 0.08	2.40 ± 0.11
2442	3.9	20.1	12 ± 8	1.60 ± 0.15	0.85 ± 0.10	2.64 ± 0.18
2517	10.0	20.7	15 ± 5	1.52 ± 0.13	0.75 ± 0.03	2.45 ± 0.13
2518	7.3	21.1	14 ± 7	1.52 ± 0.15	0.79 ± 0.04	2.49 ± 0.16
2519	3.2	21.1	12 ± 9	1.52 ± 0.18	0.73 ± 0.04	2.44 ± 0.19

Table 3-3: Water contents, and effective beta and gamma dose rates following water correction.
[a] Effective beta dose rate combining water content corrections with inverse grain size attenuation factors obtained by weighting the 200 µm attenuation factors of Mejdahl (1979) for K, U, and Th by the relative beta dose contributions for each source determined by Gamma Spectrometry.

3.3.2. Single aliquot equivalent dose determinations

For equivalent dose determination, data from single aliquot regenerative dose measurements were analysed using the Risø TL/OSL Viewer programme to export integrated summary files that were analysed in MS Excel and SigmaPlot. Composite dose response curves were constructed from selected discs and for each of the four preheating groups from each sample, and used to estimate equivalent dose values for each individual disc and their combined sets. Dose response curves for each of the four preheating temperature groups and the combined data were determined using a fit to exponential function (Appendix B). The equivalent dose was then determined for each aliquot using the corresponding exponential fit parameters.

The distribution in equivalent dose values was examined using radial plotting methods (Appendix B). All samples revealed some heterogeneity in their equivalent dose distributions. To check for the presence of non-uniformity (sample heterogeneity) in sample radiation dose histories we compared aliquot intensity and equivalent doses. In figure 3-1 the mean, median, robust mean and the logged and non-logged central age modelled mean of Galbraith (1999) are shown. The robust mean was calculated by two methods; by the use of an in-house excel program, which removed any data outwith 2 standard deviations in a continuous loop, so that data excluded from the last calculation was not included in the next; and by an excel add-in 'robust statistics' available from the Chemistry Society of London, which calculates a robust mean using Huber's estimate 2. In addition, the figure illustrates the large spread in estimated equivalent dose calculated using all six methods, implying that caution must be used in determining the equivalent dose to use in age calculations (see below).

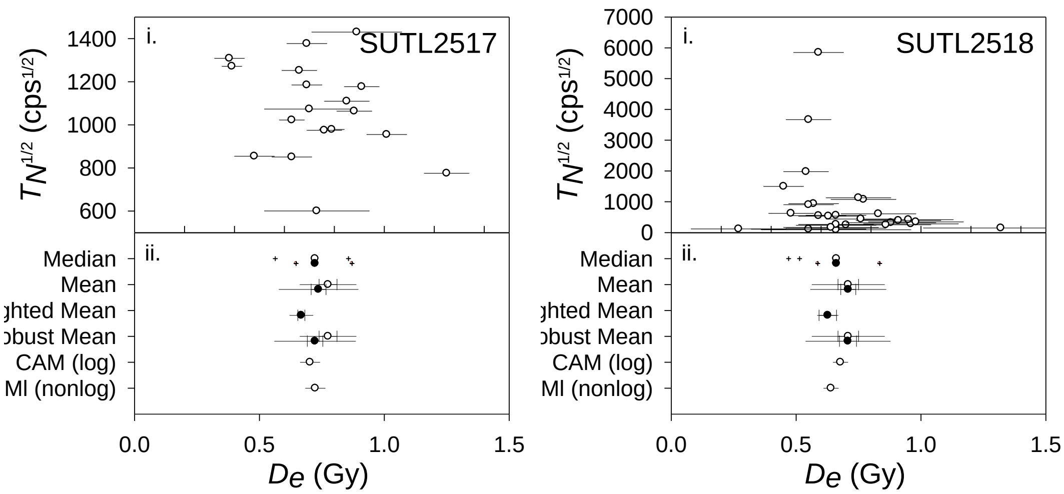

Figure 3-1: Equivalent dose distributions for samples SUTL2517-18 and 2511-13; illustrating the median, mean, weighted mean, robust mean (within 2σ) and central age modelled age values for all aliquots, and for reduced datasets containing the aliquots which statisfied the SAR criteria. In each plot, the horizontal line denotes the standard deviation on the set, and the vertical lines the standard error.

Single aliquots were rejected from further analysis based on the test dose sensitivity check, SAR criteria checks, the robust mean, feldspar contamination and radial plots. Table 3-4 summarises the quality evaluation checks on the SAR data (once filtered); the mean sensitivity of each aliquot and sensitivity change, the recycling ratio and zero dose response.

SUTL No.	Mass (mg)	Sensitivity (counts/Gy)	Sensitivity change (%)	Recycling Ratio	Zero Dose (Gy)	IRSL response (%)
2441	3.27	295 ± 96	8.43 ± 3.14	1.06 ± 0.09	0.06 ± 0.04	2.87 ± 1.95
2442	2.54	427 ± 109	5.01 ± 1.33	1.03 ± 0.02	-0.01 ± 0.03	2.88 ± 1.53
2517	3.41	1914 ± 199	17.28 ± 4.28	1.15 ± 0.03	0.03 ± 0	58.92 ± 21.7
2518	3.31	800 ± 251	7.71 ± 3.28	1.06 ± 0.03	-0.11 ± 0.19	37.67 ± 6.42
2519	3.14	832 ± 197	6.56 ± 2.49	0.98 ± 0.02	0.09 ± 0.05	62.08 ± 7.04

Table 3-4: SAR quality parameters. Standard errors given.

3.3.3. Age determinations

The total dose rate is determined from the sum of the equivalent beta and gamma dose rates, and the cosmic dose rate. Age estimates are determined by dividing the equivalent stored dose by the dose rate. Uncertainty on the age estimates is given by combination of the uncertainty on the dose rates and stored doses, with an additional 5% external error. Table 3-5 lists the total dose rate, stored dose and corresponding age of the sample.

SUTL No.	submitted	Depth	Dose Rate (mGy a^{-1})	Stored Dose (Gy)	Years BP	Calendar years
2441	July	196	2.39 ± 0.11	1.12 ± 0.08	0.51 ± 0.04	AD 1540 ± 40
2442		30	2.64 ± 0.18	0.74 ± 0.04	0.30 ± 0.03	AD 1730 ± 25
2517	October		2.43 ± 0.13	0.62 ± 0.05	0.25 ± 0.03	AD 1760 ± 30
2518			2.49 ± 0.16	0.49 ± 0.06	0.25 ± 0.02	AD 1760 ± 25
2519			2.42 ± 0.19	0.50 ± 0.05	0.20 ± 0.02	AD 1810 ± 25

Table 3-5: OSL age determinations for samples SUTL2441-42 and 2517-19

4. Discussions and conclusions

Five sediment samples collected from wind-blown sands enclosing an early-modern structure near Huesbreck, Broo Peninsula (Shetland) were analysed by the OSL method to provide a temporal framework to interpret the palaeo-environmental record on site, and date the abandonment of the structure.

The chronology established for the site spans from the mid 16^{th} century (AD 1540 ± 40; SUTL2441) through to the early 19^{th} century (AD 1810 ± 25). It is notable that the latest period of sand movement and deposition recorded at the Huesbreck site, is contemporaneous with sand deposition at the Old Scatness Broch, Scatness, dated between AD 1738 ± 25 and AD 1854 ± 14 (quartz SAR ages; Burbidge et al., 2001; Rhodes et al., 2003)

5. References

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Prescott, J.R., and Hutton, J.T., 1994, Cosmic ray contributions to dose rates for luminescence and ESR dating: Large depths and long-term time variations: Radiation Measurements, v. 23, p. 497-500.

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Appendix A: Submission forms

Site Code: Site Name: **Broo, Shetland**	**Date;** 23rd June 2012	**Context No**	**Luminescence Sample No:** **Enclosed 1**
Description of sampling location :		**Sketch of surrounding area**	
Sheet sand (wind blown); enclosed area immediately east of the excavated Broo site			
		Photo No:	
Gamma Dosimetry	**Reading**	**Assoc. Sample**	**Ref No**
	-if taken		
Details:			
-dose rate estimated in field: YES (with calibration)			
Description of Sample:			
Sample collected in copper tubing; bulk sample collected for moisture determination			
Nature of Dating Problem:			
Event			

Completed By	**Checked By**	**Date**
Ian Simpson		10th December 2012

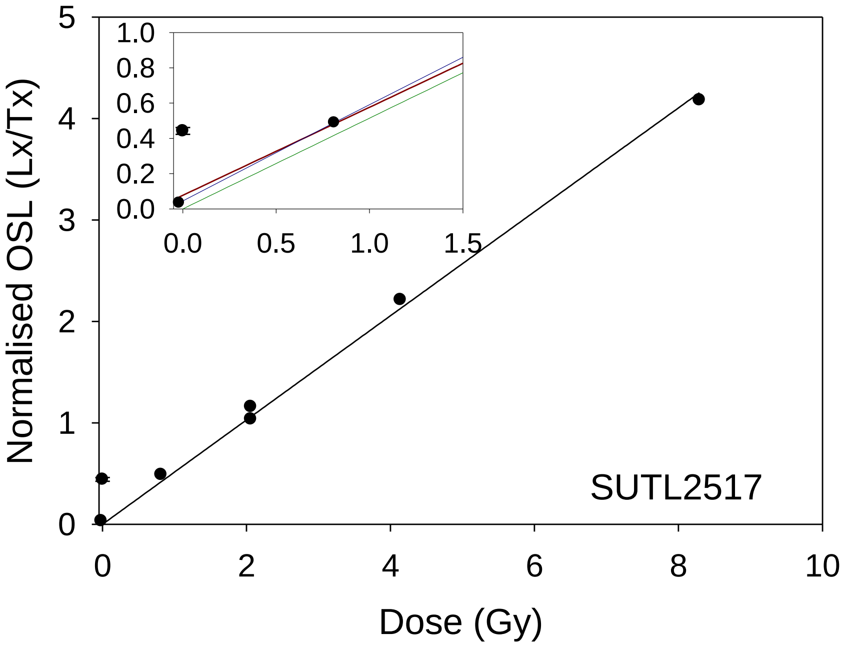
Site Code: **Site Name:** **Broo, Shetland**		**Date;** 23rd June 2012	**Context No**	**Luminescence Sample No:** **Enclosed 2**
Description of sampling location :			**Sketch of surrounding area**	
Sheet sand (wind blown); enclosed area immediately east of the excavated Broo site				
			Photo No:	
Gamma Dosimetry	**Reading**		**Assoc. Sample**	**Ref No**
	-if taken			
Details:				
-dose rate estimated in field: YES (with calibration)				
Description of Sample:				
Sample collected in copper tubing; bulk sample collected for moisture determination				
Nature of Dating Problem:				
Event				
Completed By		**Checked By**	**Date**	
Ian Simpson			10th December 2012	

Site Code: Site Name: Broo, Shetland	Date; 24th June 2012	Context No	Luminescence Sample No: Unenclosed1
Description of sampling location :		**Sketch of surrounding area**	
Sheet sand (wind blown); unenclosed area immediately south-west of the excavated Broo site			
		Photo No:	
Gamma Dosimetry	**Reading** *-if taken*	**Assoc. Sample**	**Ref No**
Details:			
-dose rate estimated in field: YES (with calibration)			
Description of Sample:			
Sample collected in copper tubing; bulk sample collected for moisture determination			
Nature of Dating Problem:			
Event			

Completed By	Checked By	Date
Ian Simpson		10th December 2012

Appendix B: Dose Response Curves

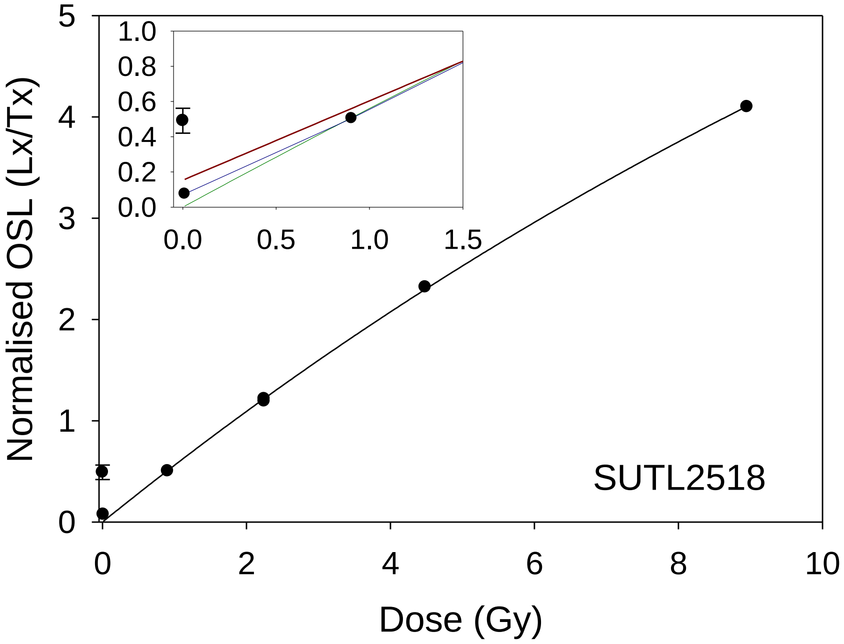
B.1 SUTL2517



Composite dose response curve for sample SUTL2508.
Lx = 0, 1, 5, 10, 30 and 5Gy;
Tx = 2 Gy

Inset shows different fits to data. Dark green = exponential fit; red = linear fit; and dark blue = best fit to lower regenerative doses (see discussion in text)

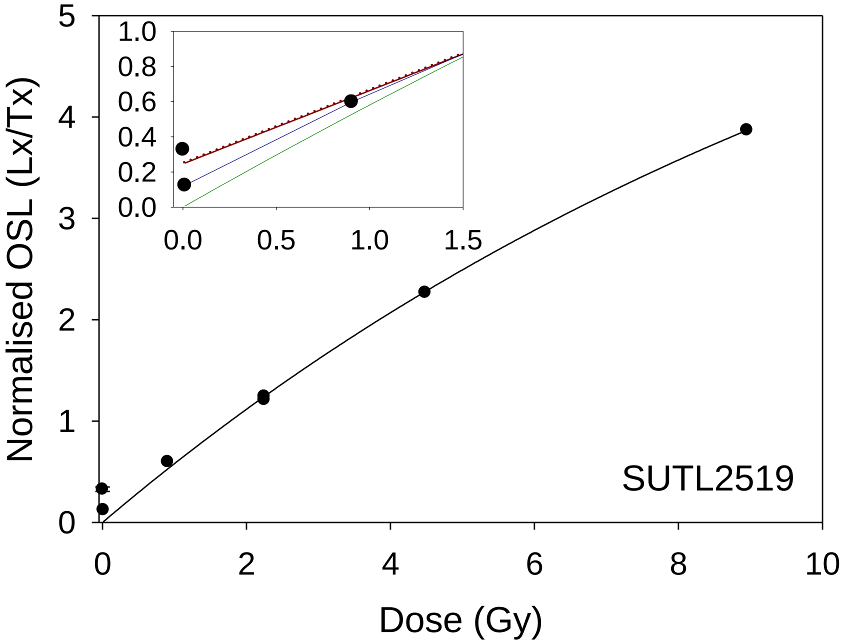
B.2 SUTL2518



Composite dose response curve for sample SUTL2509.
Lx = 0, 1, 5, 10, 30 and 5 Gy;
Tx = 2 Gy

Inset shows different fits to data. Dark green = exponential fit; red = linear fit; and dark blue = best fit to lower regenerative doses (see discussion in text)

B.3 SUTL2519

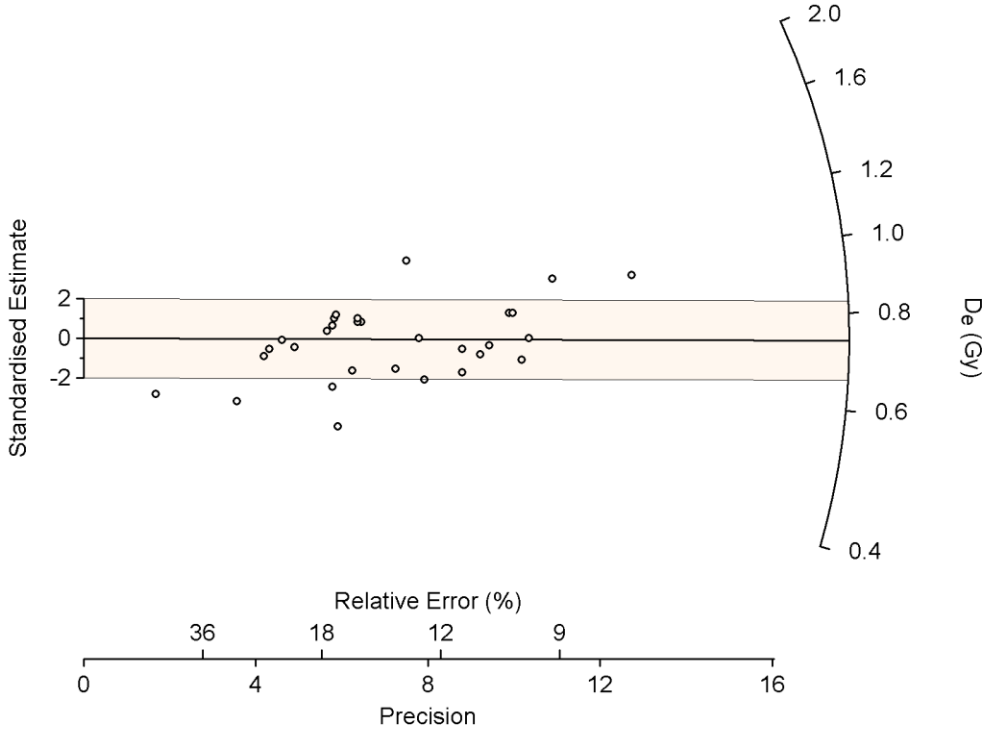


Composite dose response curve for sample SUTL2519.
Lx = 0, 1, 2.5, 5, and 10 Gy;
Tx = 2 Gy

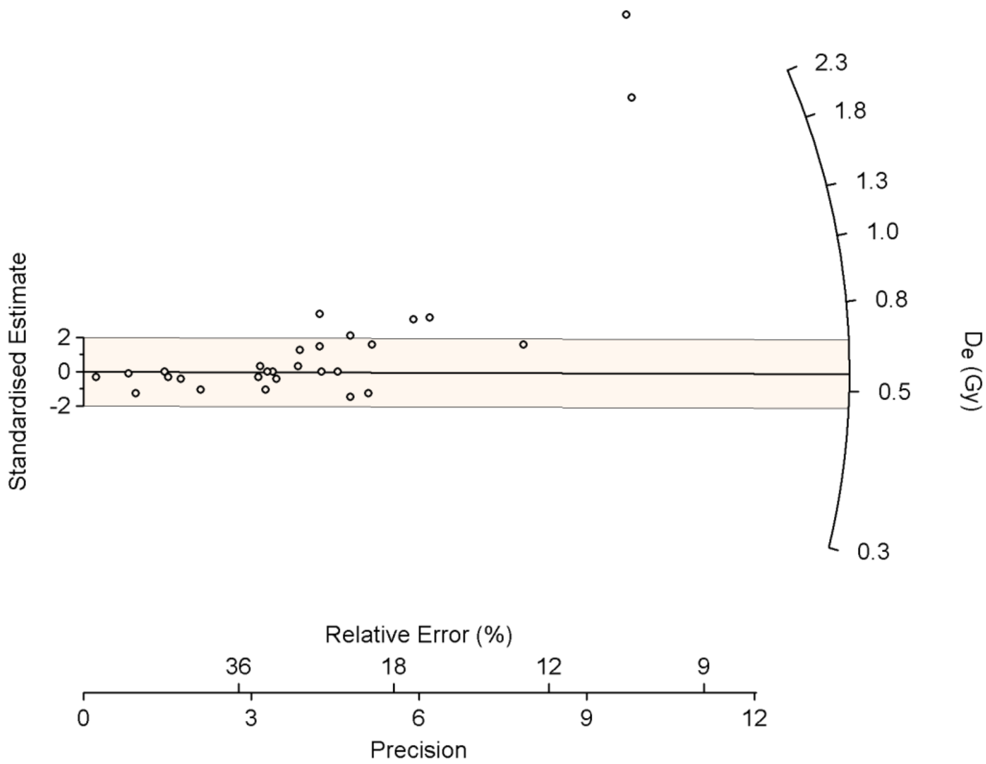
Inset shows different fits to data. Dark green = exponential fit; red = linear fit; and dark blue = best fit to lower regenerative doses (see discussion in text)

Appendix C: Radial plots

C.1 Radial plot for SUTL2517



C.2 Radial plot for SUTL2518



C.3 Radial plot for SUTL2519