Feldspar SARA IRSL dating of very low dose rate aeolian sediments from Sandwick South, Unst, Shetland

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Abstract

Young sediments, with low sensitivity and low dose rates, are challenging for luminescence dating. Here, we present work on the site of Sandwick South, a Norse settlement, in which these challenges were present. Field gamma dose rates below 0.1 mGy a^{-1} , and total dose rates of $0.4\text{-}0.5 \text{ mGy a}^{-1}$, combined with expected ages of less than 1 ka, resulted in a requirement for quantitative determination of equivalent doses of $0.2\text{-}0.4 \text{ Gy}$. The bedrock geology of the area are metagabbros, which explain both the exceptionally low dose rates and the lack of autochthonous quartz. Luminescence profiling during fieldwork revealed stratigraphically progressive OSL and IRSL signals, indicating phases in the sediments with dating potential. While laboratory characterization recovered some quartz, its low yields and luminescence sensitivity precluded application of quartz methods. Electron microscopy confirmed the presence of feldspars, which were separated and used for IRSL Single Aliquot Regeneration Additive (SARA) analysis. Counting times for both high resolution gamma spectrometry (HRGS) and thick source beta counting (TSBC) measurements were extended by 1 order of magnitude, resulting in overall uncertainties of $<6\%$ for these low dose rates. Dose estimates were obtained using an adapted SARA protocol, incorporating long overnight preheats before first measurement, with the aim of mitigating short-term fading effects. Using these procedures, archaeologically meaningful dates were obtained from this difficult material, which are internally consistent, coherent with stratigraphy, and concordant with the radiocarbon constraints of the associated archaeological settlement. The dates demonstrate sand accumulation in the early to mid-13th century AD and also in the 18th century AD, which are contemporary with disruptive sand movements registered in other coastal regions of the Northeast Atlantic and North Sea regions. The approaches adopted here have provided solutions to the challenging conditions of this young, insensitive material and can therefore be considered to extend the range and applicability of luminescence dating methods.

1. Introduction

Luminescence dating is now an established technique in dating Quaternary sediments in a range of diverse environments, yet several challenges, largely associated with variability of samples in key association with

critical units and questions, continue to limit applications and outcomes. These include, amongst others, the presence of the targeted dosimeter in a given sediment, its ability to register measureable signals from the age range under study, its sensitivity, and the extent to which its luminescence properties permits recovery of physically meaningful dose estimates. Associated radioactivity and microdosimetry must be both measureable and amenable to modeling over the time period of interest. When the sediment is young, these issues can be further compounded by limited signal levels. Not one, but all of these challenges, were encountered when investigating sediments from the most northerly part of Britain, on the island of Unst (Shetland Isles) at the Norse settlement site of Sandwick South (Fig. 1). These included: (i) exceptionally low field gamma dose rates (below 0.1 mGy a^{-1}) and total dose rates ($0.4\text{-}0.5 \text{ mGy a}^{-1}$); (ii) young expected ages of less than 1 ka, requiring quantitative determination of equivalent doses of $0.2\text{-}0.4 \text{ Gy}$; (iii) sands derived from metagabbros of the Shetland ophiolite complex, and locally, from pelitic country rocks, which are deficient in autochthonous quartz, and from which luminescence sensitivities are low. In this paper we outline the problems encountered and the solutions developed to date these difficult sediments.

The aims of the work described here were threefold: (i) to develop and validate procedures for recovering accurate dose rate measurements from extremely low radioactivity materials; (ii) to develop a luminescence protocol for quantitative determination of equivalent doses less than $<0.4 \text{ Gy}$; and (iii) apply these protocols to date sediments derived from ultra-basic/basic rocks, deficient in quartz, and requiring careful extraction and characterisation of different feldspar fractions. A further objective of the work was to validate a Single Aliquot Regeneration Additive (SARA) dose method against independent age controls and archaeological evidence, so that the protocols can be used elsewhere in other environments in which low dose, low sensitivity and young sands are encountered.

1.1 Case study, archaeological investigations at Sandwick South

The Norse settlement site of Sandwick South, excavated in 1978-80 (Bigelow, 1985), is situated on a remote east-facing bay on the island of Unst, the most northerly inhabited island in Britain (Fig. 1). Archaeological

evidence from the Norse period is substantial on Unst, beginning with the first arrivals of Norse immigrant populations as early as 1200 yrs BP (Bigelow et al., 2005). The available radiocarbon, artefact typology, and tephra evidence that exists for the site suggest occupation from ca. 1100 into the mid-14th century (i.e. the later Norse period). Shetland's location and small island topography make it a region particularly vulnerable to North Atlantic climatic regimes typified by frequent and intensive storms. The location of Unst as the most northerly and remote island in the Shetland archipelago (and therefore potentially the first Viking footfall in Britain), suggests that the Norse immigrant populations would have had to remain resilient to extreme climatic and environmental changes in the lead up to the Little Ice Age. A high-resolution chronology is needed of past, abrupt, short-term and extreme environmental changes, such that one can discern past environmental catastrophes, and determine the roles played by humans in their development, and their impacts on human populations. This study aims to constrain temporally the final phase(s) of occupation in the Norse settlement of Sandwick South, leading to abandonment, by examining the windblown sand deposits in the lee of the Norse settlement. Luminescence investigations of wind-blown sands from similar geomorphological contexts in northern Scotland have identified period(s) of increased sand movements immediately preceding the start of the Little Ice Age (our data; Gilbertson et al., 1999; Kinnaird et al., 2011; Rhodes et al., 2003; Sommerville, 2003; Sommerville et al., 2007; Wilson et al., 2002).

Figure 1 [single column]: Location map, showing the position of the Shetland Isles in relation to Britain; and Unst as the most northerly of the Shetland Isles. The black circle denotes the site of Sandwich South.

The Norse longhouse of Sandwich South is situated in the south of the bay, on the stony crest of the most seaward of the Sandwich dunes, occupying an advantageous position overlooking the bay (Bigelow, 1985). The complex of buildings measure approximately 15 - 18 m long by 3.5 m wide, delineated by drystone walls 0.6m high, and c.1.6m wide. The sediment stratigraphy examined here is located SE of the main structure, in the lee of the prevalent wind direction (Midden Unit 3; Fig. 2), and consists of a sequence of intercalated sands and midden deposits: including, an early phase midden (c. 25 cm thick; SU6), overlain by sterile sands (15-20 cm; SU5), a later phase midden (25-30 cm; SU4), then humic sands (that become less organic-rich with height in the succession, 25-30 cm), passing into clean, sterile sands (c. 10 cm; SU3).

The bedrock geology to the bay consists of metagabbros of the Shetland ophiolite complex, and locally, outcrops of pelitic country rock (Spray and Dunning, 1991). The local geology compounds the known challenges in attempting to date the windblown sands in vicinity to the Sandwich complex: indicating that (if locally derived) the sands will be comprised of extremely low radioactivity materials, contain little quartz and alkali feldspar, with only subordinate plagioclases and Na-rich feldspars.

2. Methodology

2.1 Sampling/field-based luminescence screening measurements. Sampling was conducted on 2nd December 2013, coinciding with a short visit to Shetland by GB and DS. An area was re-opened to the west of the outer exposure of Midden Unit 3 from the 1978-80 excavations, facilitating access to the lower of the midden phases and their interdigitating sands (Fig. 2). Given the constraints of winter fieldwork in a remote location in NW Shetland, all stratigraphic units were profiled, sampled and backfilled during daylight hours, and luminescence measurements were delayed until the evening. Small quantities of sediment (1-5g each) were collected under dark cover from 10 positions within the sequence, for screening measurements and further laboratory characterisation with the aim of generalising the luminescence stratigraphy throughout the sequence. Samples for potential luminescence dating were collected from all four stratigraphic units exposed using steel tubes, 3.5 cm in diameter, which were inserted into the cleaned face of the section, extracted, and sealed. In-situ field gamma spectrometry (FGS) measurements (see below) were taken from these positions using a Rainbow Multichannel Analyser coupled with a 2"× 2" NaI probe. In addition, 'bulk' samples of sediment were recovered for laboratory water content and dosimetry measurements.

Initial screening measurements using the SUERC portable OSL reader (Sanderson and Murphy, 2010) provided near real-time information on the luminescence signal progression through the stratigraphy, thus facilitating dialogue on dating priorities relative to archaeological objectives. Natural luminescence signals were measured following an interleaved sequence of system dark count (background), infra-red stimulated luminescence (IRSL) and OSL, similar to that described by Sanderson & Murphy (2010). The results are shown in Figure 2 (Table S1). The basal samples (P1/10 - P1/8; SU7/8), which may represent the primary sand blow during the archaeological period of interest (in the position of the test pit) show very measureable IRSL (c 2000 counts) and OSL (c 10-15 kcounts) intensities. Further up the profile, the samples enclosing intercalated midden/sand sequence show similar signal intensities (SU5/6), suggesting rapid accumulation, or re-deposition of materials of similar age. At the top of the profile, the clean sands above the dark unit SU4

show IRSL and OSL signal intensities which are one order of magnitude lower (c. 300 and 2500 counts, respectively), and lower depletion indices. The variation in signal intensities across this boundary suggests that the boundary represents a significant age break (Fig. 2). The field profiling data confirmed that measureable luminescence could be observed in all units, with the lower units showing similar signal levels to each other, which were significantly greater than those of the upper unit. Dating samples were taken from the lower sands (SU2603), the interdigitated midden/sand sequence (SUTL2602/2601), and at the base of the clean sands (SUTL2600).

Figure 2: Net signal intensities vs depth, stored dose-depth and sensitivity-depth plots for the sediment stratigraphy sampled in Midden Unit 3. A. from bulk sediment; B. from 90-250 μ m, 15%HF-etched polymineral separates. Key as follows: Blue circles, OSL signal intensity or depletion ratio; Red circles, IRSL signal intensity or depletion ratio; Open and closed grey circles, aliquots 1 and 2; Closed black circles, mean values [full page width]

2.2 Laboratory-based luminescence characterisations. Sample preparation protocols as previously utilised in the SUERC luminescence laboratories (cf. Sanderson et al., 2001; 2007; Burbidge et al., 2007) were used

to obtain 15%HF-etched polymineral and 40%HF-etched quartz concentrates. Simple luminescence screening measurements (cf. Sanderson et al., 2001, 2003; Burbidge et al., 2007), were undertaken on the polymineral and quartz fractions to provide the first, preliminary assessment of sensitivities and stored dose estimates throughout the sampled stratigraphy. In this technique, paired aliquots of each fraction were assessed using a simplified two-step single-aliquot regenerative dose procedure. On the basis of this data, luminescence sensitivity vs. depth and apparent dose vs. depth profiles were generated (IRSL sensitivities and stored dose estimates are presented in Fig. 2; Table S2). Notably, quartz OSL sensitivities were low through the section. None of the natural curves had visible OSL decays, and percentage errors on net signals after background subtraction ranged from -162% to 179%, with a median value of 25%. The response to test dose showed only four aliquots out of 20 with visible OSL decays and sensitivities above 1000 counts per Gy. For these the stored dose estimates carried 15-20% uncertainties and showed a heterogeneous dispersion. Coupled with low quartz yields it was concluded that other approaches would be needed.

Polymineral IRSL signal levels were larger and registered variations in stored dose in the sub-Gy region. Basal samples (P1/10-P1/9) returned stored dose values in excess of 2 Gy, suggesting that the bottom sands may contain mixed age materials, some with large residuals (Fig. 2). Further up the section, profile samples P1/8 to P1/5, all yielded similar IRSL stored dose values, consistent with the hypotheses first raised by the field profiling data, that these sediments accumulated rapidly or contain re-deposited materials of similar age. In contrast, the profiling samples P1/4-P1/1 enclosing the upper sands, yielded lower stored dose values, again supportive of the postulated age discontinuity across the stratigraphic boundary. These results therefore provided links to the signals seen in field profiling. While the sensitivity of these fractions was only a factor higher than the low sensitivity quartzes these results pointed to the possibility of using purified feldspar extracts as potential dating targets.

2.3 Dosimetry measurements. Dose rate estimates from these sands were assessed using a combination of field gamma spectrometry, high resolution gamma spectrometry (HRGS) and thick source beta counting

(TSBC; Sanderson, 1988b), reconciled with each other and with the water contents and micro-dosimetry of the model. Further details are given in supplement data section S3. However, the very low dose rates of this material, which are amongst the lowest activity samples measured in the SUERC laboratory since 1986, required significant extensions to normal counting times for HRGS and TSBC to improve the precision of net count rate and dose rates. For HRGS the sample counts were recorded for 400ks (compared with 25-50ks measurements for routine samples), and interleaved with prolonged sequences of background and reference material measurements. The activity concentrations of potassium, uranium and thorium (Table S3.1) were converted into infinite-matrix dose rates (Table S3.2). TSBC measurements were conducted for extended periods (42 replicated 600s counts, compared with 6 cycles for routine measurements), coupled with background counts over the same durations. They have been combined using weighted statistics with dry beta dose rate estimates from HRGS. Field (ranging from 11-16% of dry weight) and saturated (25-27% of dry weight) water contents were determined from all samples in the laboratory (Table S3.3) and a working value of $19 \pm 7 \%$ adopted for effective dose rate evaluation. The internal alpha effective dose rate was estimated on the basis of an alpha efficiency of 6.7 % (based on Burbidge et al., in prep), and mean U and Th internal contents of 0.18 ppm and 0.55 ppm, respectively (Anthony et al., 2001). The internal alpha effective dose rate as calculated for these samples is $0.06 \pm 0.03 \text{ mGy a}^{-1}$.

2.4 Mineral preparation. Having shown that there are measureable IRSL signals from the polymineral extracts, it was then necessary to obtain and characterize the pure feldspar fractions. Standard mineral preparation procedures were followed to obtain concentrates of alkali feldspar ($2.51 - 2.58 \text{ gcm}^{-3}$) and plagioclase ($2.58 - 2.62 \text{ gcm}^{-3}$) from the 15% HF-etched polymineral extracts. Optical microscopy and SEM analysis was performed to check mineral type, the purity of the prepared fractions, and obtain approximate elemental compositions. SEM analysis confirmed the presence of a pure Na-rich fraction (albite) within all samples, and a subordinate K-rich potassium feldspar (sanidine) within a single sample (SUTL2603; Fig. S1).

2.5 Equivalent dose determinations

2.5.1 IRSL SAR luminescence measurements. Equivalent dose determinations were made, initially, on 4 aliquots per sample using an IRSL SAR procedure (cf. Wallinga et al. 2000), with the objective of establishing whether reproducible data could be obtained, and to develop a dose response curve for the regenerative cycles. Subsequently, the investigation was extended to a further set of 32 aliquots for SUTL2603 (for which more material was available), to build statistical power to appraise selected preheat groups, and with a view to potentially cross checking the SARA data described below. IRSL measurements were conducted using a Risø DA-15 automatic reader equipped with a $^{90}\text{Sr}/^{90}\text{Y}$ β -source for irradiation, infrared (laser) diodes emitting around 830 nm for optical stimulation, and a U340 detection filter pack to detect in the region 270-380 nm. Here, regenerative dose response curves were constructed using doses of 0, 1, 2, 3, 0 and 2 Gy, with a test dose of 1 Gy. Within true SAR procedures it is only possible to run dose recovery tests on the basis of regenerative cycles, where a dose determined from a normalised ratio measured after removal of the natural signal and preheating is quantified and compared with the given dose. This does have limitations, not least of which is the use of a single laboratory source, whose heterogeneities or calibration errors are hidden in this process, however it provides a check that the data handling and dose response fitting are operating correctly, at least in the laboratory cycles. In order to overcome the potential effects of sensitivity changes associated with the readout or removal of the natural signals, these exploratory SAR measurements were followed by thermally stabilised SARA analyses.

2.5.2 IRSL SARA luminescence measurements. Equivalent dose determinations were made using a single aliquot regenerative additive dose protocol combining the SARA approach (Mejdahl and Bøtter-Jensen, 1994, 1997) with longer overnight preheating as discussed by Alexander (2007) to mitigate short term fading. In the original SARA method groups of aliquots were formed with added doses, and a linear dose estimate made for each aliquot by scaling luminescence signals between the first (natural plus added dose) and a regenerated signal (here, defined as the ‘pseudo- dose estimate’). Equivalent dose estimates are formed by regression of the pseudo-dose curves against the known added dose for each aliquot and extending this

fitted relationship to zero pseudo-dose. SARA estimates are thus independent of sensitivity changes taking place after the natural readout, since they are referred directly to the natural cycle. In the modification suggested by Alexander (2007), long overnight preheating was introduced before the first readout, so that both natural and added doses are stabilised in the manner suggested by Sanderson (1988a). Automated readout with regenerative dose pre-heated in the instrument completes the sequence. This should combine corrections of SAR data for first cycle sensitisation effects, with compensation for differential short term fading between the long slow preheat preceding the first readout, and the short high temperature preheat within the Risoe system, providing the proportion of stabilised and unstabilised signals is the same across the aliquots. This approach was implemented here with sets of 32 aliquots (16 in the case of SUTL2603), subdivided into 4 groups of 8 aliquots (natural, natural + 0.5 Gy, natural + 1 Gy, natural + 1.5 Gy; 4 groups of 4 aliquots for SUTL2603). Irradiations were performed using an automated Elsec irradiator equipped with a 1.85GBq ^{90}Sr source (dose rate 1.76 Gy/min at time of the experiment). The experimental conditions were as follows (Table S4): the IRSL from the natural and natural + β dose aliquots was recorded after a 16 hr preheat at 120°C; a 0.9 Gy test dose was given using the Risø TL-DA-15 ^{90}Sr 1.48GBq source; then the test dose response was recorded after a 30 s preheat at 200°C. IRSL measurements were made at 50°C and conducted over 60 s, with backgrounds recorded before and after stimulation.

2.5.3 Fading measurements. Fading tests were performed on 32 aliquots per sample. For each sample, 16 aliquots were given a 1 Gy dose before storage (set 1); the other 16 aliquots were given a 1 Gy dose immediately after storage (set 2). Irradiations were performed using the automated Elsec irradiator. For an initial readout the samples were stored in the dark at ambient temperatures for periods ranging between $7\text{--}8 \times 10^5$ s. All aliquots were given a 16hr preheat at 120°C (following the suggestions of Sanderson, 1988a), before IRSL readout at 50°C, followed by a 1 Gy test dose, pre-heat at 200°C for 30 s and a further IRSL at 50°C i.e. replicating the experiment conditions used in the SARA protocol. Subsequently, half the aliquots were re-irradiated (set 1), then both sets stored in the dark for 9×10^6 s, before all aliquots were subjected to

the same readout procedure as above. Following the last readout 16 aliquots were again irradiated before being placed in storage for a further fading test to be conducted in the future (set 1).

3. Results

3.1 Dose rate determinations. Infinite matrix alpha, beta and gamma dose rates from HRGS are listed for all samples in tables S3, together with infinite matrix beta dose rates from TSBC, and in situ gamma dose rates from FGS (section 2.1). The Sandwich South samples had extremely low activity, with K, U and Th concentrations ranging between 0.1 and 0.2 %, 0.2 and 0.3 ppm and 0.4 and 0.8 ppm, respectively. The available data from HRGS, corrected for water content, was reconciled with the measured beta (TSBC) and gamma (FGS) dose rates, an estimate of the cosmic dose contribution, and the internal alpha effective dose, in a dosimetric model to obtain total environmental dose rates. It is noted that there was good agreement between HRGS, TSBC and FGS. Through incorporating the three independent dose determinations, and running duplicate measurements over extended periods in consecutive weeks, it was possible to achieve gamma and beta dose rate estimates with < 6% error.

3.2 Equivalent dose determinations

3.2.1 IRSL SAR equivalent dose determinations. IRSL signals were integrated over the initial 2.4 s of stimulation, and a background based on the last 9.6 s of stimulation. Natural and regenerated signals were normalised to the subsequent test-dose response, and equivalent doses evaluated for each disc, and for groups of discs by preheat, and across the sample set, by interpolation of linear fits to the dose response plots. The parameters obtained for the linear fits were noted, and later used, with the IRSL SARA data to generate pseudo-dose recovery ratios and dose distributions. The Sandwich sands exhibited reasonable sensitivity and produced acceptable SAR quality criteria. Recycling ratios were within error of unity - ranging between 1.01 ± 0.03 and 1.07 ± 0.09 across the sample set. Zero dose values were slightly inflated, indicating some recuperation of signal during the SAR sequence, or incomplete bleaching within a single

SAR cycle, with zero dose values <10% of the normalised natural signals. A positive sensitivity change, varying between 6 and 11 % per cycle, was noted for each sample.

To provide an independent control on the IRSL SARA ages reported below, the IRSL SAR protocol was applied to 32 aliquots of Na-rich feldspar from SUTL2603. This material exhibited similar sensitivities and SAR quality criteria to that previously examined: recycling ratios were in error of unity, 0.99 ± 0.02 ; normalised zero dose values (independent of whether 1st or 2nd cycle) ranged between 0.01 and 0.1; and the Na-rich feldspar exhibited a positive sensitivity change, 3-6% per cycle. As there was no evidence of a significant differences in normalised IRSL ratios (both in natural and regenerated dose points) between subsets of discs pre-heated at temperatures between 200°C to 260°C, a composite dose response was generated and used to obtain equivalent dose values for each aliquot. The composite dose response plot for this sample is shown in figure 3a. The corresponding radial plot (Fig. 3b) indicated a significant population of equivalent doses around 0.35Gy, which corresponds with the lowest dose population, and is interpreted here as the best-bleached component. The estimate of stored dose is 0.35 ± 0.05 Gy.

3.2.2 IRSL SARA equivalent dose determinations. Data were reduced by extracting net signals from the first 2.4 s of stimulation after subtraction of integrated signals from the last 9.6 s of measurement. Figure 4a illustrates representative IRSL decay curves for both a natural and test dose response in the (first cycle of the) SARA protocol; and indicates that the Na-rich feldspar fraction carries sufficient sensitivity to register natural and test dose responses in the sub-Gy region. For each sample, robust and weighted mean statistical approaches were used to appraise distributions in normalised values obtained for each 8 aliquot set (natural, natural + β_1 , natural + β_2 etc.). A representative SARA additive dose plot (for SUTL2602), together with the regression line fitted through the weighted mean values, and the intercept with the additive dose axis is shown in figure 4b). Regression analysis was performed using software developed in house for TL dating (Sanderson, 1987; Supplementary Data); which produced stored dose estimates of 0.38 ± 0.05 Gy (2603; Table S5), 0.33 ± 0.02 Gy, 0.37 ± 0.02 Gy and 0.11 ± 0.02 Gy (2600). It is noted that the corrected (for

fading) stored dose estimates from the SARA method are concordant with radiocarbon ages (Table S6), and the preliminary uncorrected stored dose estimate from IRSL SAR (Fig. 3c).

To examine whether the SARA procedure masks a wide distribution in equivalent dose values, pseudo-dose distributions were generated for each sample, through scaling the normalised natural + additive dose values minus the normalised additive dose to the sample-dependent SAR linear dose response to obtain approximate equivalent doses. This method, together with the appraisal of the SARA growth response, was used to appraise any heterogeneity in equivalent dose values, potentially encompassing information on its depositional mechanism/setting. The scatter in pseudo equivalent dose values obtained for individual samples, and across the sample set, reveal some heterogeneity, but importantly, that there are very few aliquots which tail to a lower apparent dose (which could be indicative of post-depositional mixing), and that excess scatter is to higher apparent dose populations (which is presumably do to luminescence residuals). Similarly, pseudo-dose recovery tests were performed for each sample, through subtracting the sample-dependent SARA stored dose value from each aliquot given an additive dose value, and comparing this nominal normalised value to the known given dose. It is recognised that this method will only work for well-bleached sediment. The ratio of the nominal dose to the given dose obtained across the entire sample set is 1.07 ± 0.10 ($n = 80$), with the large standard error reflecting the scatter in the equivalent dose distribution.

3.2.1 Fading results. The mean ratio of sensitisation-independent stored to prompt normalised signals after 10^5 s storage (after Sanderson, 1988a) from all samples is 0.98 ± 0.02 . The results of the fading tests are shown in figure 4c. g-values were calculated for each sample (Table S5), ranging between 6.1 %, 0.7% and 2.7-2.4 % per decade (SUTL2600, 2601, 2602 and 2603, respectively). Age estimates before and after fading correction using sample-specific g-values are equivalent within uncertainties (Table S5). While it may be difficult to ascertain whether the long preheat before first measurement in the SARA procedure has mitigated short-time fading, it is clear that very low fading rates have been observed giving dates which are aconsistent

with the archaeological constraints. This is consistent with previous studies at SUERC using similar preheating conditions (e.g. Spencer and Sanderson, 2012).

(A) SUTL2602, Natural

(B)

(C)

Figure 3: [single column, left] (a) Linear SAR dose response plot for SUTL2603; (b) Abanico Plot (after Dietze et al., 2014) for 32 aliquots of SUTL2603; (c) Kernel density estimate plots for SARA and SAR equivalent dose estimates for

SUTL2603. The 6 SARA IRSL equivalent dose estimates are those obtained from the pseudo-dose distribution (details outlined in text), with the weighted mean estimate derived from this distribution; the equivalent dose estimates obtained by SAR IRSL are limited to the aliquots which cluster around 1σ of the weighted mean (identified in the text, as best representing the well-bleached population of equivalent doses)

Figure 4: [single column, right] (a) Representative IRSL decay curves; (b) SARA dose response plot; and (c) fading test results. Red, SUTL2600; Green, 2601; Yellow, 2602; Blue, 2603. SARA dose response plots for each sample are shown in the Supplementary Data file.

3.3 Age determinations

Luminescence ages were calculated using standard microdosimetric models, with uncertainties that combined measurement and fitting errors from the SARA/SAR analysis, all dose rate evaluation uncertainties, and allowance for the calibration uncertainties of the sources and reference materials (Table S5). Individual fading corrected IRSL SARA ages fall in the range $AD\ 1190 \pm 130$ (SUTL2603), $AD\ 1200 \pm 80$, $AD\ 1200 \pm 80$ and $AD\ 1750 \pm 60$ (SUTL2600; Table S5). The IRSL SARA age estimates were assessed relative to the stratigraphy and existing chronological constraints for the site, and using conventional age assimilation methods and a Bayesian approach. A weighted combination of the lower three SARA ages (indistinguishable within error) suggests a mid-13th century date for the lower sand accumulation (Bayesian assimilations produce a similar result). The IRSL SARA age determinations add further to the growing body of OSL dating of windblown sands on archaeological sites in Shetland; the stratified sequences at Sandwick South, in combination the field- and laboratory-profiling, yield internally coherent sets of dates confirming sand blows in the 12-13th centuries and the 18th century.

4. Conclusions

1. The SARA approach applied to these Unst sands, coupled to careful dose rate measurement, has succeeded in dating this challenging material. The reconciliation of HRGS data, converted to infinite matrix dose rates, with measured beta and gamma dose rates, counted over extended periods and in duplicate, enabled total environmental dose rates to be estimated with less than 6% uncertainties. The modified SARA approach, which incorporates a long overnight preheat before first measurement, yielded archaeologically

meaningful ages, concordant with independent age controls. The SARA approach, combined with long, low temperature preheats, has the potential for dating sediment in other young, low dose-rate systems.

2. Multiple luminescence methods, from initial luminescence screening using portable OSL equipment, through laboratory characterisation measurements, to Na-rich feldspar IRSL SARA dating, have been successfully applied to archaeologically significant windblown sands in Shetland. The initial luminescence screening, using portable OSL equipment, provided the first appraisal of net signal variations within the sampled section at Sandwick South. The field profile obtained from Sandwick South showed an overall increase in luminescence signals with depth, consistent with a normal age-depth progression. Furthermore, the initial luminescence screening showed that each of the stratigraphic breaks identified in the field, corresponded with a step in signal level, suggesting large temporal breaks across sediment boundaries. Laboratory profiling reproduced the apparent trends/maxima in the field profiling dataset. Furthermore, it provided the first indication that the quartz mineral phase at Sandwick South would prove problematic for dating (suggesting that one would have to exploit other minerals, and luminescence signals). At Sandwick South the stratified sequences accompanied by luminescence profiling have produced internally coherent sets of dates which confirm the presence of sand blows in the 12th-13th century AD and the 18th century AD.

3. IRSL SAR, IRSL SARA and radiocarbon chronologies for the site are concordant. Furthermore, the combination of conventional and Bayesian statistical approaches, provides a tighter chronological control on the phasing within the Midden Unit 3 and a robust constraint on when sands inundated the site, which may have led to its eventual demise.

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Figure captions:

Figure 1: Location map, showing the position of the Shetland Isles in relation to Britain; and Unst as the most northerly of the Shetland Isles [*single column*]

Figure 2: Net signal intensities vs depth, stored dose-depth and sensitivity-depth plots for the sediment stratigraphy sampled in Midden Unit 3. Key as follows: Blue circles, OSL signal intensity or depletion ratio; Red circles, IRSL signal intensity or depletion ratio; Open and closed grey circles, aliquots 1 and 2; Closed black circles, average of the two aliquots [*full page width*]

Figure 3: [*single column, left*] (a) Linear SAR dose response plot for SUTL2603; (b) Abanico Plot (after Dietze et al., 2014) for 32 aliquots of SUTL2603; (c) Kernel density estimate plots for SARA and SAR equivalent dose estimates for SUTL2603. The 6 SARA IRSL equivalent dose estimates are those obtained from the pseudo-dose distribution (details outlined in text), with the weighted mean estimate derived from this distribution; the equivalent dose estimates obtained by SAR IRSL are limited to the aliquots which cluster around 1σ of the weighted mean (identified in the text, as best representing the well-bleached population of equivalent doses)

Figure 4: [*single column, right*] (a) Representative IRSL decay curves; (b) SARA dose response plot; and (c) fading test results. Red, SUTL2600; Green, 2601; Yellow, 2602; Blue, 2603. SARA dose response plots for each sample are shown in the Supplementary Data file.