



Street House, Street House Farm, North Yorkshire

Optically Stimulated Luminescence Dating of Briquetage from an Early Neolithic Salt Making Site

Jean-Luc Schwenninger, Frances Healy and Stephen Sherlock



Street House, Street House Farm, North Yorkshire

Optically Stimulated Luminescence Dating of Briquetage from an Early Neolithic Salt Making Site

Jean-Luc Schwenninger, Frances Healy and Stephen Sherlock

National Grid Reference: NZ 7390 1930

Print: ISSN 2398-3841

Online: ISSN 2059-4453

The Research Report Series incorporates reports by Historic England's expert teams, their predecessors and other researchers. Many Research Reports are interim, to make available the results of specialist investigations in advance of full publication. Although subject to internal quality assurance, they are not usually refereed externally and their conclusions may sometimes have to be modified in the light of information not available at the time of the investigation. Where no final project report is available, readers should consult the author before citing these reports.

For more information email Res.reports@HistoricEngland.org.uk or write to:

Historic England, Fort Cumberland, Fort Cumberland Road, Eastney,
Portsmouth PO4 9LD

Opinions expressed in Research Reports are those of the author(s) and are not necessarily those of Historic England.

Summary

Three samples of briquetage were collected from a salt production site at Street House near Loftus for dating by optically stimulated luminescence (OSL). A series of radiocarbon dates hinted at the existence of a rare Early Neolithic saltern dating to the early fourth millennium cal BC. There was a distinct possibility, however, that these dates may relate to an earlier phase of occupation with subsequent Bronze Age, Iron Age or even Roman activities potentially having disturbed much older deposits. The aim of the luminescence dating was to provide independent age estimates for the salt-making activity, allowing the taphonomy of the samples submitted for radiocarbon dating to be assessed.

Contributors

Jean-Luc Schwenninger, Frances Healy and Stephen Sherlock

Acknowledgements

Specific thanks are extended to Historic England, who helped secure funding for the dating and their role in co-ordinating and managing the project as well as the reporting. Emma Brownlee kindly collated the maps reproduced here as Figure 1 and Alex Bayliss made useful comments on a draft of this report.

Front cover image

Structure [1707] set into the boulder clay. [photograph by Stephen Sherlock]

Archive location

Land of Iron Museum, Deepdale, Mill Lane, Skinningrove, North Yorkshire, TS13 4AP

Historic Environment Record

Redcar and Cleveland Historic Environment Record, Strategic Planning Team, Redcar and Cleveland Borough Council, Seafield House, Kirkleatham Street, Redcar, TS10 1SP

Date of research

2021–25

Contact details

Historic England, Cannon Bridge House, 25 Dowgate Hill, London, EC4R 2YA. ☎ 020 7973 3700. ✉ customers@HistoricEngland.org.uk

Jean-Luc Schwenninger, Research Laboratory for Archaeology and the History of Art, School of Archaeology, University of Oxford, 1 South Parks Road, Oxford OX1 3TG. ✉ jean-luc.schwenninger@rlaha.ox.ac.uk

Frances Healy, Independent Researcher. ✉ fmahealy@gmail.com

Stephen Sherlock, Independent Researcher. ✉ stevesherlock86@gmail.com

Contents

Introduction	1
Background	1
Sample Collection	4
Methods	7
The physical basis of luminescence dating.....	7
Sample preparation	9
Measurement procedures and conditions.....	10
Dose rate determination	11
Results and Discussion	16
Discussion and Conclusions.....	18
References	19
Appendix 1: Elemental analysis of samples by fusion ICP-MS.....	22
Appendix 2: Typical OSL shine down curves and dose	23
Appendix 3: Palaeodose distribution plots	24

Illustrations

Figure 1: Maps to show the location of Street House, in red. Scale: top right 1:250,000; bottom 1:10,000. [© Crown Copyright and database right 2025. All rights reserved. Ordnance Survey Licence number 100024900].....	3
Figure 2: Fired clay also known as 'briquetage' submitted for luminescence dating. Sample X7743 (left) and X7745 (right).	5
Figure 3: Excavation of three vessels at the base of furnace [1707], which had been lifted in a block. [photograph by Stephen Sherlock].....	6
Figure 4: Determination of the external gamma dose rate contribution for sample X7743 using a scaled gamma dose rate (1.037 ± 0.213 Gy/ka).	13
Figure 5: Determination of the external gamma dose rate contribution for sample X7744 using a scaled gamma dose rate (1.037 ± 0.207).	14
Figure 6: Determination of the external gamma dose rate contribution for sample X7745 using a scaled gamma dose rate (1.073 ± 0.226).	15

Tables

Table 1: Details of samples for Optically Stimulated Luminescence.....	4
Table 2: Summary of luminescence characteristics including tests for recycling, recuperation, feldspar contamination, dose recovery as well as a qualitative assessment of sensitivity and signal saturation based on 12 separate measurements per sample. The results suggest that according to these criteria, the samples and the adopted measurement procedures are well suited for OSL dating.....	17
Table 3: Summary of the optically stimulated luminescence (OSL) dating results. The results are based on luminescence measurements of sand-sized quartz (180–255µm) mounted as small (2–3mm) multigrain aliquots. Dose rate calculations are based on concentrations of radioactive elements (potassium, thorium, uranium and rubidium) obtained on dried, homogenised and powdered material by elemental analysis using ICP-MS/AES combined with a fusion sample preparation technique. The final OSL age estimates include an additional 4% systematic error to account for uncertainties in source calibration. Dose rate calculations were made using the online calculator DRAC (v1.02) developed by Durcan et al. (2015). These incorporated beta attenuation factors (Mejdahl 1979), dose rate conversion factors (Guerin et al. 2011) and an absorption coefficient for the water content (Zimmerman 1971). The contribution of cosmic radiation to the total dose rate was calculated as a function of latitude, altitude, burial depth and average overburden density based on data by Prescott and Hutton (1994). Further details pertaining to individual samples are presented in Appendix 1.....	17

Introduction

Background

Evidence for prehistoric salt production in Britain

The evidence for Iron Age salt manufacture is well known in the British Iron Age, with 'Red Hills' recognised as salt mounds in Burnham, Essex as early as AD 1888 (Fawn et al. 1990, 2). Iron Age salterns are now recognised along the coast from Cornwall in the south-west through to Yorkshire in the east. Furthermore, there are hundreds of salt-making sites dating from the Iron Age and Roman periods have been recognised across eastern England (Lane and Morris 2001). There is evidence for Bronze Age salt working in Britain at c. 30 sites, with notable examples from Billingborough, Lincolnshire, Mucking, Essex, and Brean Down, Somerset (all referenced by Kinory, 2012, 14). The site at Street House, north-east Yorkshire, has been published in an initial paper citing evidence for early Neolithic salt manufacture (Sherlock 2021).

The site at Street House

Street House is situated on the Yorkshire Coast, on cliffs overlooking the North Sea, between Saltburn and Whitby (Fig.1). Excavations between AD 1918 and AD 1930 by William Hornsby recorded eight Bronze Age burial mounds at Boulby, whilst later excavations by Blaise Vyner were focused upon two sites Neolithic sites: a Cairn (Vyner 1984) and a palisaded site (Vyner 1988). More recent excavations, between AD 2004 and AD 2024, have recorded Bronze Age, Iron Age, Roman and Saxon sites (Sherlock 2019). The evidence for Neolithic activity at Street House includes a broad range of datable artefacts and monuments that encompass settlement, burial, ceremonial, boundaries and industrial activity. In AD 2010–11 a small circular structure of Early Neolithic date was excavated and later excavations between AD 2016 and AD 2022 exposed an ovoid structure. The continuation of the excavations since AD 2018 has recorded a furnace and kiln structure for firing vessels and evidence for salt manufacture (Sherlock 2021).

The evidence for salt manufacture at Street House is unequivocal, with structural, artefactual and scientific dating arguing for this site being the earliest saltern in the UK. The structural features incorporate a furnace as the principal source of heat and a flue to conduct the hot air along a channel with an open hearth upon which evaporation occurred. Artefacts include kiln material including trivets to support the in situ tray and as well as ceramic props and spacers, many of which have portable X-ray fluorescence indicators for chloride on the inner surfaces. Thin section analysis of some of the sherds suggests the

briquettage vessels were made using local clay, arguably fired on site. The ^{14}C measurements that are currently available can be modelled to estimate a start date in the fortieth century cal BC for the site. Further ^{14}C dating is ongoing and will yield a more precise and robust chronology.



Figure 1: Maps to show the location of Street House, in red. Scale: top right 1:250,000; bottom 1:10,000. [© Crown Copyright and database right 2025. All rights reserved. Ordnance Survey Licence number 100024900]

Sample Collection

A total of three samples of fired clay associated with the production of salt and sometimes also referred to as 'briquetage' was submitted to the laboratory for luminescence dating in February 2024. These artefacts were collected during two seasons of excavations in 2021 and 2022. Two came from deposits within furnace [1707] and one from a deposit within hearth [1690]. They had been placed in sealed plastic sample bags and then transferred into PVC tubs to protect them from breaking apart. Two additional samples, one of a surrounding deposit and one of an adjacent deposit, were also supplied in order to quantify the external gamma dose rate contribution from the surrounding soil matrix and to help determine a more realistic moisture content. Further details regarding individual samples are presented in Table 1 and examples of two of the briquetage samples (X7743 & X7745) are illustrated in Figure 2. The locations of the samples can be seen in Figure 3.

Table 1: Details of samples for Optically Stimulated Luminescence.

Field code	Laboratory code	Sample weight	Burial depth (cm)
4456 [context 1796]	X7743	351g	246
4062 [context 1753]	X7744	169g	232
4212 [context 1738]	X7745	72g	196
Context 1796	Dosimetry sample for X7743 & X7745	n/a	n/a
4062 [Context 1682]	Dosimetry sample for X7744	n/a	n/a



Figure 2: Fired clay also known as 'briquetage' submitted for luminescence dating. Sample X7743 (left) and X7745 (right).



Figure 3: Excavation of three vessels at the base of furnace [1707], which had been lifted in a block. [photograph by Stephen Sherlock]

Methods

The physical basis of luminescence dating

When ionising radiation (predominantly alpha, beta and gamma radiation) interacts with an insulating crystal lattice (such as quartz or feldspar), a net redistribution of electronic charge takes place. Electrons are stripped from the outer shells of atoms and though most return immediately, a proportion will escape and become trapped at meta-stable sites within the lattice. This charge redistribution continues for the duration of the radiation exposure and the amount of trapped charge is therefore related to both the duration as well as the intensity of radiation exposure. Even though trapped at meta-stable sites, electrons may become 'free' again if the crystal is then subjected to heat or exposed to light. Once liberated, a free electron may become trapped again or may return to a vacant position caused by the absence of a previously displaced electron (a 'hole'). This latter occurrence is termed 'recombination' and the location of the hole is described as the 'recombination centre'. As recombination occurs, a proportion of the energy of the electron is dissipated. Depending upon the nature of the centre where recombination occurs, this energy is expelled as heat and/or light. Therefore, when the crystal (mineral grain) is either heated or illuminated following natural or artificial laboratory irradiation (the 'dose') the total amount of light emitted (luminescence) is directly related to the number of liberated electrons and available recombination sites. This is the fundamental principle upon which luminescence dating is based and a more detailed account of the method (TL and OSL) may be found in Aitken (1985) and Aitken (1998).

In cases where the duration of dosing is not known (as is the case for dating), estimates can be made from laboratory measurements. The response (sensitivity) of the sample to radiation dose (i.e. the amount of light observed for a given amount of laboratory radiation, usually beta-radiation) must be established. From this relationship the equivalent radiation exposure required to produce the same amount of light as that observed following the natural environmental dose can be determined, and this is often referred to as the palaeodose or 'equivalent dose' (D_e). The palaeodose is obtained by measuring the luminescence emitted by crystals extracted from the archaeological sample and comparing it with the luminescence signal measured following the administration of a known absorbed dose provided by a calibrated radiation source. The palaeodose (measured in Gy) is therefore an estimate of the total dose absorbed during the irradiation period (i.e. burial period). When the dose rate (the amount of radiation per unit time, measured in Gy/ka) is determined in situ (or calculated from measured concentrations of radionuclides within the

sample), then the duration of the dosing period (i.e. luminescence age) can be calculated using the following equation:

Luminescence age = Palaeodose ÷ Dose Rate.

The technique of optical dating was first applied to quartz by Huntley et al. (1985), and methodological details were further refined by Smith et al. (1986, 1990) and Rhodes (1988). The method is effectively a development of thermoluminescence (TL) dating (Aitken 1985) and has since been successfully applied to many different types of sedimentary environments and numerous archaeological sites. A major advance has been the introduction of the 'single aliquot regenerative-dose' (SAR) measurement protocol (Wintle 1997, Murray and Wintle 2000) as it generally provides improved accuracy (e.g. through correction of sensitivity change, interpolation rather than extrapolation of D_e values) as well as increased precision. In some instances, this protocol may also be used to provide an indication of incomplete zeroing of the luminescence signal (i.e. insufficient exposure to daylight) at the time of deposition as detected by the relative scatter in individual palaeodose estimates or the asymmetry of dose distributions. Although originally developed for dating the exposure of unheated sediments to light, the OSL dating method can also be successively applied to date a range of heated archaeological artefacts including ceramics and burnt stone. As in thermoluminescence dating (TL), the exposure of materials to heat empties all the traps and the accumulation of charge resumes immediately upon cooling and will then continue until the next heating event (or exposure to light). In the case of stones or ceramics, the luminescent mineral grains are encased by the rock or clay fabric and hence, will be shielded from light or accidental resetting. The grains are exposed to a constant field of radiation comprising alpha and beta radiation from within the crystal itself, beta and gamma radiation from the archaeological sample and the surrounding medium (soil/sediment) as well as cosmic radiation originating from the sun and exploding stars elsewhere in the universe.

The amount of radiation received by individual mineral grains is dependent upon the concentrations of naturally occurring radioisotopes of uranium, thorium, potassium and rubidium which are present in low concentrations in all geological materials. Due to the extremely long half-life (billions of years) of these radionuclides, the radiation field within the burial medium and the sample itself may effectively be seen as being constant over archaeological timescales. The intensity of the flux of radiation is generally referred to as the dose rate which can be quantified by a variety of methods including direct on-site determinations using passive dosimeters, or measurements made with a portable gamma-ray spectrometer. Alternatively, it may also be derived from laboratory-based methods such as thick source alpha counting, high resolution gamma-ray spectrometry or various

types of elemental analyses including neutron activation analysis (NAA) or inductively coupled plasma mass spectrometry (ICP-MS). In order to correctly assess the contribution of cosmic radiation, the burial depth of the samples as well as the altitude and geomagnetic location of the site have to be taken into account. The moisture content of the sample and the surrounding sediment also have an effect on the amount of radiation absorbed by the mineral grains. Whereas in the case of fired clay (i.e. bricks or tiles) removed from historic buildings this value is usually well below 5%, this may not be true for pottery or burnt clay collected from buried archaeological contexts and where the water content within the sample and the surrounding sediment may be much higher or even saturated.

Sample preparation

All laboratory treatments were performed at the luminescence dating laboratory of the Research Laboratory for Archaeology and the History of Art, University of Oxford under low intensity laboratory safe-lighting from purpose-built amber LED's emitting at ~593 nm. The sample preparation procedures adopted in this study were designed to yield pure coarse-sized quartz within a selected size range of 180–255 μ . The grains were extracted from the samples of briquetage using standard procedures in use at the laboratory and in this instance, based on the quartz inclusion technique first proposed by Aitken (1985) for dating pottery. To ensure enough quartz could be extracted for dating the samples were taken through a standard preparation procedure as outlined below.

All three samples presented an overall thickness of circa 40–50mm. After air drying at room temperature, cleaning and removal of the exposed outer 5mm of the sample, the remaining lumps of clay were then cut open in order to remove material from the inner core of the sample. This was achieved by gouging out the clay fabric and releasing grains with the help of a hooked dentist tool and then gently crushing the extracted material or, if required, using a drill fitted with a water-cooled diamond burr revolving at slow speed. The obtained mineral debris was then passed through sieves with water to isolate the 180–255 μ m grain size fraction for dating. The separated fraction was treated with diluted (10%) hydrochloric acid (HCl) to remove carbonates and then etched in concentrated HF (40%) for 60 minutes. This treatment serves two purposes: (i) to dissolve feldspar grains, and (ii) to remove (etch) the outer surface rind (10 μ) of quartz grains which is affected by alpha irradiation during burial. The remaining coarse grains were then treated with HCl for a second time in order to remove fluorides that could have precipitated during the HF etching process. Any heavy minerals were subsequently removed by gravity separation using a sodium polytungstate solution at a density of 2.72g cm⁻³. Finally, each sample was re-sieved to remove smaller, heavily etched grains including any feldspar which may not

have completely dissolved during the etching process. The dried prepared quartz samples were then mounted as multigrain aliquots on 10mm circular aluminium discs for luminescence measurement using a viscous silicone oil as a mounting medium. The aliquot size was limited to 2 to 3mm in order to improve the detection of poorly bleached grains (from the spread and symmetry of individual palaeodose estimates) and to help reduce any potential effects on age overestimation. In the case of sample X7744, measurements were performed on single grains extracted from the fired clay.

Measurement procedures and conditions

Luminescence measurements were performed using three automated luminescence measurement systems housed within the laboratory. This included two instruments (a *TL/OSL-DA-12 reader* -upgraded to a *TL/OSL-DA 20-* and a *TL/OSL-DA-15* single grain reader) manufactured by the Risø National Laboratory in Denmark (Bøtter-Jensen 1988, 1997; Bøtter-Jensen et al. 2000) as well as a *LexsygSmart* device made by Freiberg Instruments in Germany (Richter et al. 2015). The instruments were fitted with inbuilt $^{90}\text{Sr}/^{90}\text{Y}$ ceramic β -sources calibrated against the most recent standard available from the Risø National Laboratory in Denmark (Hansen et al. 2015; Autzen et al. 2022). Both Risø readers allowed laboratory irradiations of the quartz grains using low but very uniform dose rates of ca. 0.011Gy s^{-1} and ca. 0.034Gy s^{-1} whereas a stronger source inside the Freiberg device delivered ca. 0.12Gy s^{-1} .

In the case of both instruments made by Risø, optical excitation was provided by filtered blue diodes (emitting at $\sim 410\text{nm}$) whereas for the *LexsygSmart*, stimulation originated from ten filtered green LED's (emitting at $\sim 525\text{nm}$). For the latter device, the UV quartz OSL emission at $\sim 370\text{ nm}$ was detected through a 2.5mm Hoya U-340 filter and a 2.5mm Delta BP 365/50EX interference filter placed in front of a photomultiplier tube (Hamamatsu H7360-02) fitted with a blue-green sensitive bi-alkali photocathode offering enhanced sensitivity from the visible to near infrared (UV-VIS). For the two Risø readers, the emitted quartz OSL signal was detected in the UV region using bi-alkali photomultiplier tubes made by EMI and filtered with Hoya U-340 glass filters.

OSL measurements were made at a raised temperature of 125°C (to ensure no re-trapping of charge to the 110°C TL trap during measurement) and using a modified single aliquot regenerative (SAR) measurement protocol (Wintle 1997; Murray and Wintle 2000; Wintle and Murray 2006). In order to minimize the contribution of potential feldspathic components to the quartz signal, each OSL measurement was also preceded by an infrared bleach (Banerjee et al. 2001; Wallinga et al. 2002). The presence of infrared-sensitive minerals (e.g. feldspars) from the prepared quartz was checked using an array of

IR LED's (850Δ20nm; 300mW/cm²) at 50°C for 50s before blue/green light stimulation. The natural and regenerative doses were preheated to 240°C for 10s, and the fixed test doses used to correct for sensitivity changes, were preheated at a reduced temperature of 220°C, before optical stimulation (100s). The decision to opt for this preheat combination was based on a series of initial tests to check the overall suitability and signal characteristics of each sample. This also included dose recovery tests which resulted in near perfect recovery for a known administered laboratory dose.

Data generated by the luminescence measurements were analyzed using the *Analyst* software (v.4.57) developed by Duller (2015). The software package *Luminescence* (v. 09.21) developed by Kreutzer et al. (2012) for the statistical programming language 'R' was used to calculate mean palaeodose estimates (see Table 3) and for plotting individual palaeodose distributions (see Appendix 3).

Dose rate determination

Dose rate calculations are based on Aitken (1985) and, in the absence of on-site radioactivity measurements using either a portable gamma-ray spectrometer or passive dosimeters, these had to be derived entirely from the concentrations of radioisotopes (potassium, thorium, uranium and rubidium) as determined from representative subsamples of the briquetage including two samples of sediment from archaeological contexts 1682 and 1796 and elemental analysis by ICP-MS/AES. These analyses were outsourced to a certified specialist laboratory (*Actlabs*) in Canada who, after crushing the sample to a fine powder, applied a fusion sample preparation technique to ensure complete release of all radionuclides within the sample. Dose rate calculations were obtained using the dose rate conversion factors of Guerin et al. (2011) and calculated using the '*Dose Rate and Age Calculator*' software (DRAC v1.02) developed by Durcan et al. (2015). The contribution of cosmic radiation to the total dose rate was calculated as a function of latitude, altitude, burial depth and an average overburden density of 1.9gcm⁻³ based on data by Prescott and Hutton (1994).

The final OSL age estimates (see Table 3) include an additional systematic error (4%) to account for uncertainties in source calibration and measurement reproducibility.

In the case of routine pottery dating the environmental dose rate contribution can be reasonably well assessed by considering the gamma dose rate as originating primarily from the surrounding sediment and the beta dose rate as originating from radionuclides present within the clay fabric of the sample. Due to the considerable thickness of the fired clay objects submitted for dating in this study, however, a non-negligible internal gamma rate contribution from the sample itself also has to be factored in. Furthermore, the

samples of briquetage were not present as isolated individual pieces but rather encountered in stacks or groupings of amalgamated pieces (Fig. 2). It would therefore be wrong to assume a simple scenario for the calculation of the environmental dose rate, and a scaled gamma dose rate according to Appendix H in Aitken (1985) was considered to be more appropriate for this set of samples. In essence, this simple model assumes an average thickness of 5cm for each sample with the immediate surroundings (radius of 20cm) also being comprised for the most part of similar burnt clay material and the related deposit only contributing towards the external gamma dose rate beyond a radius of 20cm.

This situation is further complicated by the differential water content observed between the harder and therefore less permeable pieces of burnt clay and the loose texture of the related organic sediment which has a much higher capacity to retain moisture. For reference, the modern recorded water content as determined in the laboratory by weighing the sample before and after oven drying at 50°C within the samples of briquetage was 1.5% for sample X7743, 2.3% for X7744 and 27.4% for X7745. For the additional dosimetry soil sample X7743DosA, the recorded moisture content was 26.7% and for X7744DosA it was 17.02%.

In order to take account of some of these factors and to achieve a more realistic approximation of the applicable environmental dose rate, the function '*Scale_gammadose*' developed by Riedesel et al. 2023 for the R '*Luminescence*' package (Kreutzer et al. 2012) was used to determine the external dose rate (see Figs 4, 5 and 6 below). This was based on an assumed mean water content of $7\pm5\%$ for the briquetage samples (referred to as target layer A in Figs 4, 5 and 6), $10\pm5\%$ for the surrounding burnt clay matrix (referred to as layer B in Figs 4, 5 and 6) and $20\pm5\%$ for the related sediment (referred to as layer C in Figs 4, 5 and 6). A relatively large uncertainty was attached to these values to account for variability throughout the burial period including seasonal effects and difficulties in correctly assessing the moisture content within each of the samples and their surroundings.

Due to the considerable uncertainty in accurately determining the environmental dose a rather large uncertainty of 20% was also applied to the scaled external gamma dose rate. On a more positive note, it is worth noting that the concentrations of radioisotopes within the samples of briquetage as well as the additional dosimetry samples obtained from the related sediment are similar (see Table 3). This is rather reassuring and gives extra confidence in the correct assessment of the total environmental dose rate.

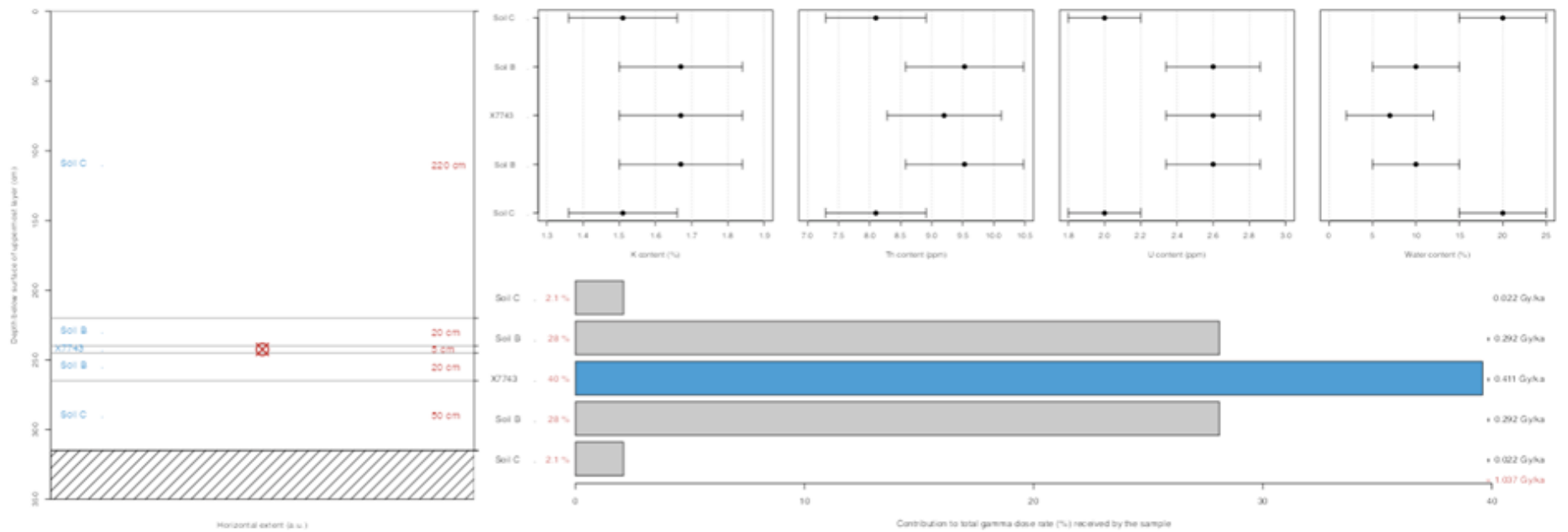


Figure 4: Determination of the external gamma dose rate contribution for sample X7743 using a scaled gamma dose rate (1.037 ± 0.213 Gy/ka).

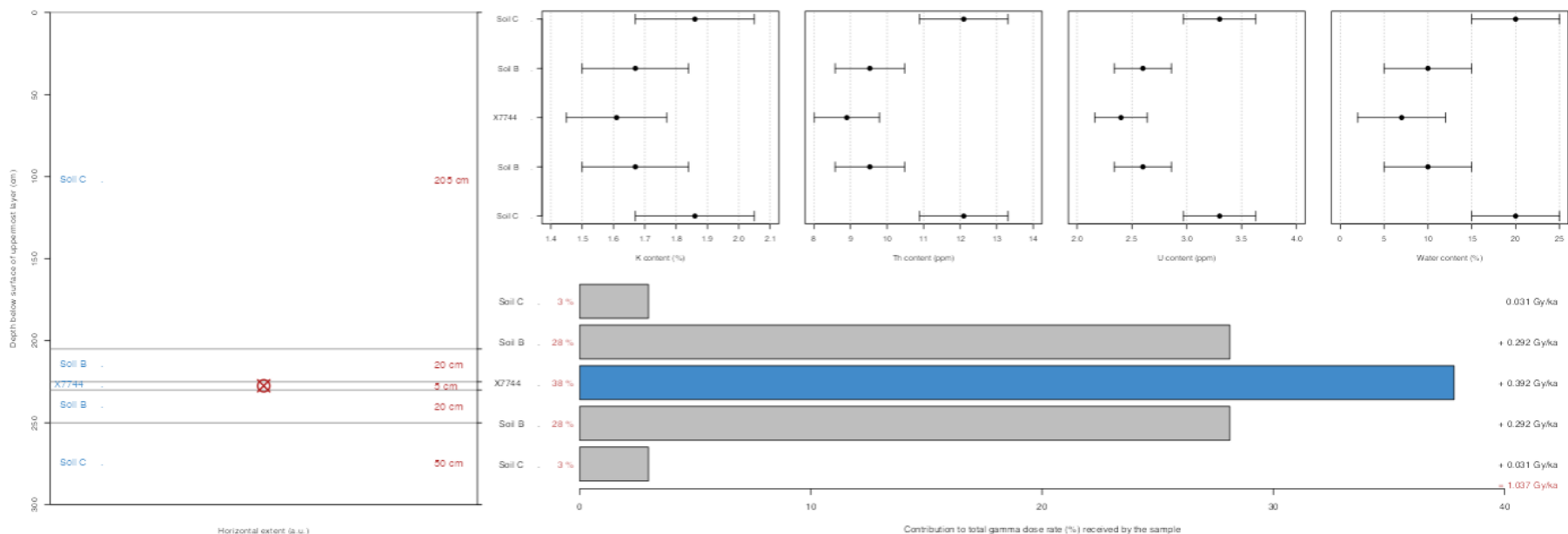


Figure 5: Determination of the external gamma dose rate contribution for sample X7744 using a scaled gamma dose rate (1.037 ± 0.207).

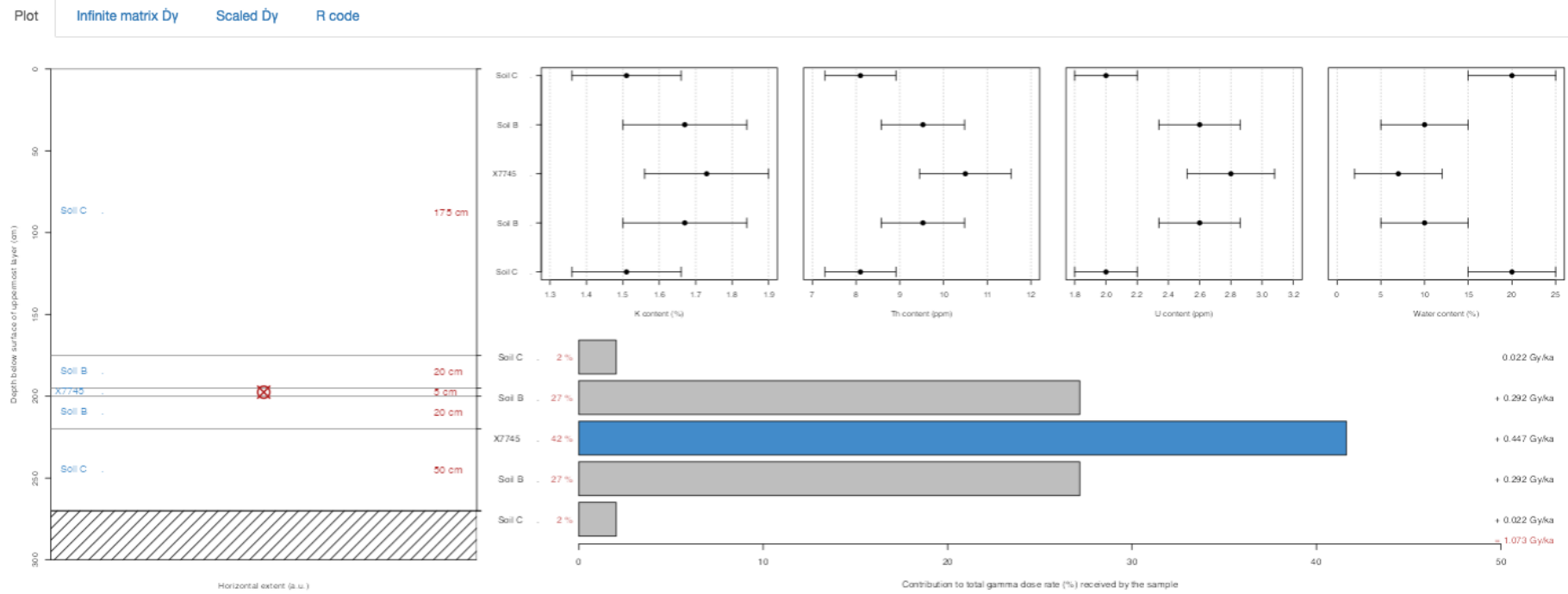


Figure 6: Determination of the external gamma dose rate contribution for sample X7745 using a scaled gamma dose rate (1.073 ± 0.226).

Results and Discussion

A summary of the luminescence characteristics is presented in Table 2 and the results of the OSL measurements, radioactivity data and age estimates are presented in Table 3. More detailed information pertaining to the age calculations and geochemical composition of individual samples are provided in Appendices 1 and 2. Typical OSL signal plots and dose response curves are presented in Appendix 3 and palaeodose distribution plots are provided in Appendix 4.

The yield of quartz mineral grains derived from these samples was plentiful and aliquots generally showed excellent response to laboratory irradiation. Visual checks of the initial signal intensity and the form of the decay curve show a fast decrease in OSL intensity which is characteristic of quartz. This is further evidenced by a well defined 110°C TL peak and stimulation using infrared (IR) laser diodes also confirmed the purity of each aliquot with negligible contributions from potential feldspathic contaminants (<3%; see IRSL/OSL ratio in Table 2). In the SAR measurements a low irradiation dose was repeated (recycling point) at the end of the measurement cycle to test how well the SAR sensitivity correction procedure was working. If the sensitivity correction is adequate then the ratio of the signal from the repeated dose to that of the initial regeneration dose should fall within the range of 0.9–1.10. Good recycling ratios close to unity were recorded for all three samples (see Table 2). A further test on the thermal transfer also showed that no significant recuperation of the OSL signal was detected in the majority of aliquots in response to a zero dose. Dose response curves generally pass through the origin when a zero Gy beta dose is included thus indicating that thermal transfer of charge from optically insensitive traps into OSL traps is not an issue. Similarly, the results from the dose recovery tests (ratio of the given dose to the recovered dose) performed on multiple aliquots from each sample showed excellent agreement (see Table 2) and give confidence in the adopted measurement procedures and instrument settings.

Table 2: Summary of luminescence characteristics including tests for recycling, recuperation, feldspar contamination, dose recovery as well as a qualitative assessment of sensitivity and signal saturation based on 12 separate measurements per sample. The results suggest that according to these criteria, the samples and the adopted measurement procedures are well suited for OSL dating.

Sample code	Mean Recycling ratio	Mean IRSL/OSL ratio	Mean Dose recovery ratio	Thermal transfer	Sensitivity	Signal saturation
X7743	tbc	tbc	0.982	Negligible	Excellent	No
X7744	tbc	tbc	0.996	Negligible	Excellent	No
X7745	tbc	tbc	1.006	Negligible	Excellent	No

Table 3: Summary of the optically stimulated luminescence (OSL) dating results. The results are based on luminescence measurements of sand-sized quartz (180–255µm) mounted as small (2–3mm) multigrain aliquots. Dose rate calculations are based on concentrations of radioactive elements (potassium, thorium, uranium and rubidium) obtained on dried, homogenised and powdered material by elemental analysis using ICP-MS/AES combined with a fusion sample preparation technique. The final OSL age estimates include an additional 4% systematic error to account for uncertainties in source calibration. Dose rate calculations were made using the online calculator DRAC (v1.02) developed by Durcan et al. (2015). These incorporated beta attenuation factors (Mejdahl 1979), dose rate conversion factors (Guerin et al. 2011) and an absorption coefficient for the water content (Zimmerman 1971). The contribution of cosmic radiation to the total dose rate was calculated as a function of latitude, altitude, burial depth and average over-burden density based on data by Prescott and Hutton (1994). Further details pertaining to individual samples are presented in Appendix 1.

Field Code	Lab Code	Radioisotopes				Scaled External γ -dose rate (Gy/ka)	Cosmic dose rate (Gy/ka)	Total dose rate (Gy/ka)	Palaeodose (Gy)	OSL date
		K %	Th ppm	U ppm	Rb ppm					
4456	X7743	1.67	9.2	2.6	90	1.037 ± 0.213	0.156 ± 0.016	2.70 ± 0.23	15.47 ± 0.67	5720 ± 550
4062	X7744	1.61	8.9	2.4	94	1.037 ± 0.207	0.158 ± 0.016	2.64 ± 0.23	15.57 ± 1.43	5895 ± 740
4212	X7745	1.73	10.5	2.8	101	1.037 ± 0.226	0.166 ± 0.017	2.83 ± 0.25	16.74 ± 0.63	5900 ± 560

Discussion and Conclusions

It was important to obtain direct dates for the fired clay unequivocally associated with salt-making at Street House Farm because these samples were part of the pyrotechnology undertaken at the site rather than possibly accidental inclusions in the sediments. All three luminescence measurements are compatible with the early fourth millennium BC age indicated by the available radiocarbon dates and the associated artefacts. This is highly significant because salt making at such an early date is otherwise unknown in Britain, where previously salt making is evident only from the late second millennium BC onwards.

References

- Aitken, M. J. 1985 *Thermoluminescence Dating* (London).
- Aitken, M. J. 1998 *An Introduction to Optical Dating: The dating of Quaternary Sediments by the use of Photon-Stimulated Luminescence* (Oxford).
- Autzen, M., Andersen, C. E., Bailey, M., and Murray, A.S. 2022 'Calibration quartz: An update on dose calculations for luminescence dating' *Radiation Measurements* 157: <https://doi.org/10.1016/j.radmeas.2022.106828>
- Banerjee, D., Murray, A. S., Bøtter-Jensen, L., and Lang, A. 2001 'Equivalent dose estimation using a single aliquot of polymineral fine grains', *Radiation Measurements*, 33, 73–94: [https://doi.org/10.1016/S1350-4487\(00\)00101-3](https://doi.org/10.1016/S1350-4487(00)00101-3)
- Bøtter-Jensen, L. 1988 'The automated Risø TL dating reader system' *Nuclear Tracks and Radiation Measurements*, 14, 177–180: [https://doi.org/10.1016/1359-0189\(88\)90060-X](https://doi.org/10.1016/1359-0189(88)90060-X)
- Bøtter-Jensen, L. 1997 'Luminescence techniques: instrumentation and methods', *Radiation Measurements* 27, 749–768: [https://doi.org/10.1016/S1350-4487\(97\)00206-0](https://doi.org/10.1016/S1350-4487(97)00206-0)
- Bøtter-Jensen, L., Bulur, E., Duller, G. A. T., and Murray, A. S. 2000 'Advances in luminescence instrument systems', *Radiation Measurements* 32, 523–528: [https://doi.org/10.1016/S1350-4487\(00\)00039-1](https://doi.org/10.1016/S1350-4487(00)00039-1)
- Duller, G. A.T. 2015 'The Analyst software package for luminescence data: overview and recent improvements', *Ancient TL* 33, 35–42: <https://doi.org/10.26034/la.atl.2015.489>
- Durcan, J. A., King, G. E., and Duller, G. A. T. 2015 'DRAC: Dose rate and age calculator for trapped charge dating', *Quaternary Geochronology* 28, 54–61: <https://doi.org/10.1016/j.quageo.2015.03.012>
- Fawn, A. J., Evans, K. A., Macmaster, I., and Daview, G. M. R. 1990 *The Red Hills of Essex: Salt Making in Antiquity*, Colchester Archaeological Group Annual Bulletin 30 (Colchester)
- Guerin, G., Mercier, N., and Adamiec, G. 2011 'Dose-rate conversion factors', *Ancient TL* 29(1), 5–8: <https://doi.org/10.26034/la.atl.2011.443>
- Hansen, V., Murray, A. S., Buylaert, J-P., Yeo, E-Y., and Thomsen, K.J. 2015 'A new irradiated quartz for beta source calibration', *Radiation Measurements* 81, 123–127: <https://doi.org/10.1016/j.radmeas.2015.02.017>

- Huntley, D. J., Godfrey-Smith, D. I., and Thewalt, M. L. W. 1985 'Optical dating of sediments', *Nature*, 313, 105–7: <https://doi.org/10.1038/313105a0>
- Kinory, J. 2012 *Salt Production: Distribution and Use in the British Iron Age*, British Archaeological Reports B559 (Oxford)
- Kreutzer, S., Schmidt, C., Fuchs, M. C., Dietze, M., Fischer, M., and Fuchs, M. 2012 'Introducing an R package for luminescence dating analysis', *Ancient TL*, 30(1), 1–8: <https://doi.org/10.26034/la.atl.2012.457>
- Kreutzer, S., Burow, C., Dietze, M., Fuchs, M.C., Schmidt, C., Fischer, M., Friedrich, J., Mercier, N., Philippe, A., Riedesel, S., Autzen, M., Mittelstrass, D., Gray, H.J., and Galharret, J. 2023 'Luminescence: Comprehensive Luminescence Dating Data Analysis. R package version 0.9.21' <https://CRAN.R-project.org/package=Luminescence>
- Lane, T. and Morris, E. L. 2001 *A Millenium of Salting: Prehistoric and Romano-British Salt Production in the Fenland*, Lincolnshire Archaeology and Heritage Report Series 4 (Sleaford)
- Mejdahl, V. 1979 'Thermoluminescence dating: beta-dose attenuation in quartz grains', *Archaeometry* 21, 61–72: <https://doi.org/10.1111/j.1475-4754.1979.tb00241.x>
- Murray, A. S., and Wintle, A. G. 2000 'Luminescence dating of quartz using an improved single-aliquot regenerative-dose protocol', *Radiation Measurements*, 32, 57–73: [https://doi.org/10.1016/S1350-4487\(99\)00253-X](https://doi.org/10.1016/S1350-4487(99)00253-X)
- 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*, 23, 497–500: [https://doi.org/10.1016/1350-4487\(94\)90086-8](https://doi.org/10.1016/1350-4487(94)90086-8)
- Rhodes, E. J. 1988 'Methodological considerations in the optical dating of quartz', *Quaternary Science Reviews*, 7, 395–400: [https://doi.org/10.1016/0277-3791\(88\)90035-2](https://doi.org/10.1016/0277-3791(88)90035-2)
- Richter, D., Richter, A., and Dornich, K. 2015 'Lexsyg Smart – A luminescence detection system for dosimetry, material research and dating application', *Geochronometrika* 42, 202–209: <https://doi.org/10.1515/geochr-2015-0022>
- Riedesel, S., Autzen, M., Burow, C., 2023 'scale_GammaDose(): Calculate the gamma dose deposited within a sample taking layer-to-layer variations in radioactivity into account (according to Aitken, 1985) Function version 0.1.2' in Kreutzer (et al. 2023)

Sherlock, S. J. 2019 *A Neolithic to Late Roman Landscape on the North-East Yorkshire Coas: Excavations at Street House, Loftus, 2004–17*, Tees Archaeology Monograph Series 7 (Hartlepool)

Sherlock, S. J. 2021 'Early Neolithic salt production at Street House, Loftus, north-east England', *Antiquity* 95, 648–69: <http://dx.doi.org/10.15184/aqy.2021.25>

Smith, B. W., Aitken, M. J., Rhodes, E. J., Robinson, P. D., and Geldard, D. M. 1986 'Optical dating: methodological aspects', *Radiation Protection Dosimetry*, 17, 229–33: <https://doi.org/10.1093/oxfordjournals.rpd.a079813>

Smith, B. W., Rhodes, E. J., Stokes, S., Spooner, N. A., and Aitken, M. J., 1990 'Optical dating of sediments: initial results from Oxford', *Archaeometry*, 32, 19–31: <https://doi.org/10.1111/j.1475-4754.1990.tb01078.x>

Vyner, B. E. 1984 'The Excavation of a Neolithic cairn at Street House, Loftus, Cleveland', *Proceedings of Prehistoric Society* 50, 151–96: <https://doi.org/10.1017/S0079497X00007519>

Vyner, B. E. 1988 'The Street House Wossit: the excavation of a later Neolithic and early Bronze Age palisaded ritual monument at Street House, Loftus, Cleveland', *Proceedings of Prehistoric Society* 54, 173–202: <https://doi.org/10.1017/S0079497X00005818>

Wallinga et al. 2002

Wintle, A. G. 1997 'Luminescence dating: laboratory procedures and protocols', *Radiation Measurements* 27, 769–817: [https://doi.org/10.1016/S1350-4487\(97\)00220-5](https://doi.org/10.1016/S1350-4487(97)00220-5)

Wintle, A. G. and Murray, A. S. 2006 'A review of quartz optically stimulated luminescence characteristics and their relevance in single-aliquot regeneration dating protocols', *Radiation Measurements* 41, 369–391: <https://doi.org/10.1016/j.radmeas.2005.11.001>

Zimmerman, D. W. 1971 'Thermoluminescent dating using fine grains from pottery', *Archaeometry* 13, 29–52: <https://doi.org/10.1111/j.1475-4754.1971.tb00028.x>

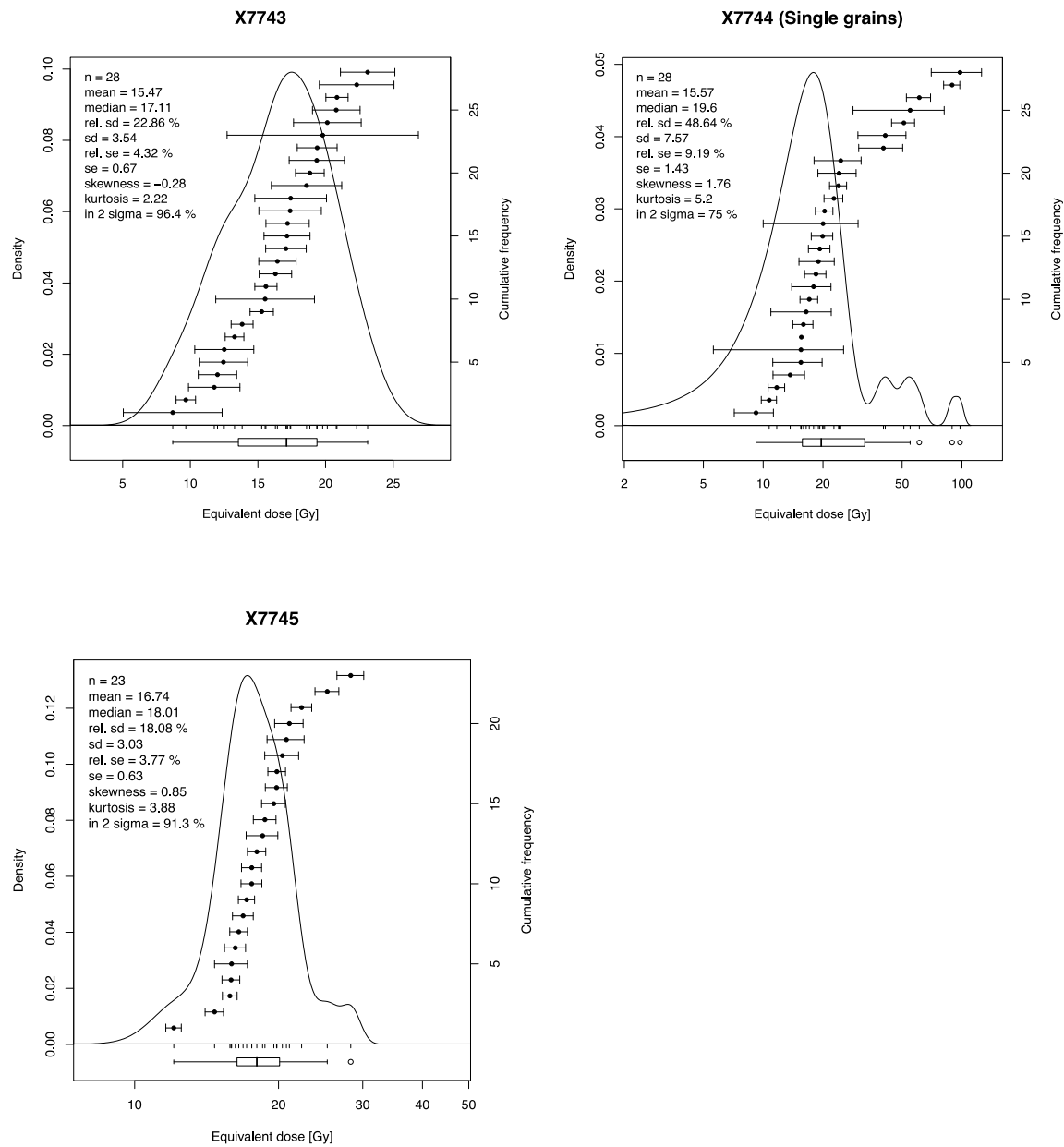
Appendix 1: Elemental analysis of samples by fusion ICP-MS

To be added

Appendix 2: Typical OSL shine down curves and dose

To be added

Appendix 3: Palaeodose distribution plots





Historic England

Historic England's Research Reports

We are the public body that helps people care for, enjoy and celebrate England's historic environment.

We carry out and fund applied research to support the protection and management of the historic environment. Our research programme is wide-ranging and both national and local in scope, with projects that highlight new discoveries and provide greater understanding, appreciation and enjoyment of our historic places.

More information on our research strategy and agenda is available at

HistoricEngland.org.uk/research/agenda.

The Research Report Series replaces the former Centre for Archaeology Reports Series, the Archaeological Investigation Report Series, the Architectural Investigation Report Series, and the Research Department Report Series.

All reports are available at HistoricEngland.org.uk/research/results/reports. There are over 7,000 reports going back over 50 years. You can find out more about the scope of the Series here: HistoricEngland.org.uk/research/results/about-the-research-reports-database.

Keep in touch with our research through our digital magazine *Historic England Research*

HistoricEngland.org.uk/whats-new/research.