OSL dating report – Easington

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Methods

OSL samples were collected from gravelly sand beds by hammering opaque plastic tubes into the face of the cleaned section. A carbonate cemented sand bed was also sampled as an intact block with the light-exposed edges removed by dissolution using 10% HCl. All samples were processed under subdued orange light in the luminescence laboratories at the Department of Geography, RHUL. Quartz was extracted in the 150-250 or 180-250µm grain size fractions following HCl and H_2O_2 treatment. Heavy minerals were removed by density separation and the remaining grains were etched in 40% HF solution for 50 minutes. All samples were subsequently placed in flurosilicic acid for 5 days to dissolve any remaining feldspar grains, followed by an HCl wash for 1 hour and re-sieving.

External dose rates were calculated from the concentration of radioactive isotopes (U, Th, K) determined by ICP-MS and AES (Department of Geology at RHUL) and/or in situ dose rate measurements using an Ortec MicroNomad γ-spectrometer. The dose rate conversion factors of Adamiec and Aitken (1998) were used throughout and the internal quartz dose rate of 0.06 Gy/ka was assumed based on previous measurements in eastern England and using an alpha efficiency factor of 0.10 ± 0.02 in the dose rate conversion (Mauz et al., 2006). The beta dose attenuation/absorption was accounted for using the factors of Mejdahl (1979). Cosmic ray contributions were calculated from the altitude, latitude and longitude of the section as well as the thickness and density of the overburden (Prescott and Hutton, 1994). In estimating the burial history, it was assumed that site has been buried 13m of Late Devensian glacigenic sediments for the past ca. 23 ka, and prior to this a burial depth of 2±1 m was assumed based on the thickness of raised beach sediments that overlie the sampling locations. If evidence becomes available that the sediments were buried to a significantly greater depth then the age of the site will have to be revised downwards. If the beach had been constantly buried by 10m of sediment prior to the Late Devensian, this will cause a 9% increase in the estimated age. *In situ* water contents were measured after drying the samples at 110°C for 24hrs and saturated water contents were assessed from the volume/density of material within the OSL sampling tubes or undisturbed blocks. Water contents were placed at 0.5 ± 0.3 of the saturated value and dose rates were corrected for water attenuation as described by Aitken (1985).

Luminescence measurements were performed on a Risø OSL/TL-DA-15 system using blue light LED stimulation (470 nm, ~40 mW/cm^3) and a U-340 detection filter. Laboratory irradiation used a ^{90}Sr/^{90}Y beta source which was calibrated against quartz which had been γ-irradiated quartz at the National Physical Laboratory (Teddington, UK). Prepared quartz grains were mounted onto 10 mm sized steel discs with the inner 5 mm part of the disc covered with a monolayer of grains using viscous silicone oil.

The single aliquot regeneration (SAR) protocol was used to estimate sample D_e values and all luminescence measurements were performed for 50s whilst the sample was held at 130°C to prevent re-trapping in 110°C TL trap (Murray and Roberts, 1998; Murray and Wintle, 2000, 2003). Five regenerative doses up to 400 Gy were used to bracket the D_e of each sample and a 15 Gy test dose was used. The luminescence signal was integrated from the initial 0.8s of the decay curve and a background was subtracted from the last 5 s of the stimulation. The test dose background was taken from the previous natural or regenerative measurement (Murray and Wintle, 2000). A residual IR signal presumed to originate from feldspar formed <2% of the blue light (BL) stimulated signal and any small feldspar contribution was effectively eliminated using a post-IR blue SAR procedure with a 50s room temperature IR-shine used prior to each blue OSL measurement.

Equivalent doses were calculated using a saturating exponential plus linear function in custom written software in C++ employing the Levenberg-Marquardt algorithm from the Bevington and Robinson (1992) implementation. Errors on the equivalent dose were calculated using the Monte Carlo method with 1000 iterations. In order to ensure sufficient quality and precision during each measurement, aliquots were rejected from the analysis if their recycling ratios (difference between a repeated data point and the first regenerative measurement) exceeded 10% from unity, and IR/BL ratio on the natural test dose OSL measurement was >10%. Individual D_e estimates were combined using the central age model of Galbraith et al. (1999) and errors in the final age calculations include systematic errors from beta source calibration (3%, Armitage and Bailey, 2005), dose rate conversion (3%, Murray and Olley, 2002) and gamma-ray spectrometry calibration (3%) (Murray and Funder, 2003), and cosmic ray contribution (calculated from Prescott and Hutton, 1994). Random uncertainties include ICP-MS dosimetry measurement (estimated at 5%), moisture content, and the standard error of the D_e estimates.

Results

Prior to each OSL measurement, pre-heating of each aliquot is needed to ensure that charge from light-sensitive, thermally-unstable traps is emptied. A pre-heat plateau test was performed on sample EAS05 to assess whether the measured doses are dependent on the preheat conditions. Groups of 6 aliquots were measured using the SAR protocol but the pre-heat temperature (PH1) was increased in 20°C increments from 160 to 300°C, and PH2 kept at 160 or 220°C. No dependence of D_e, recycling ratio, or signal recuperation relative to pre-heat temperature was observed (results not shown) although the residual IR signal was reduced to <2% when a 280°C PH1 was used. Dose recovery tests of 200 Gy were performed on groups of 4 aliquots in each sample with the average measured/given dose ratio being 1.06 ± 0.04, very close to unity and confirming the ability of these samples to measure a known laboratory dose.

The results of the quartz OSL dating are shown in Table 1 alongside the dose rate data and D_e estimates from between 12 and 22 aliquots per sample. When the OSL ages are compared to the marine isotope curve (Fig. 1) derived from the ODP677 site (Shackleton et al., 1990), four out of five dates are within errors of MIS 7 and range from 153 ± 17 to 250 ± 30 ka BP. The age of sample EAS05 taken from a carbonate-cemented bed also falls within the age range of the other samples implying that the cementation occurred early on and has not caused a significant dose rate change over time. Because the samples were taken from time synchronous beds and represent replicate measurements, the weighted mean and standard error of 201 ± 17 ka. It should also be noted that this error only includes the random uncertainties between samples. If the systematic uncertainties scaled on each date are combined in quadature, a mean and error of 201 ± 28 ka is obtained which will cover systematic uncertainties in beta source calibration, gamma-ray spectrometry calibration, dose rate conversion factors, beta dose attenuation factors, water content, cosmic dose/burial depth, and internal quartz dose.

Sample	*n*	Burial depth (m)	K (%)	U (ppm)	Th (ppm)	Water (%)*	Dose rate (Gy/ka)	De (Gy)	Age (ka)
EAS01	16	2.0 ± 1.0	0.43	2.01	2.05	5	1.20 ± 0.11	184 ± 9	153 ± 17
EAS02	13	2.0 ± 1.0	0.45	2.31	1.66	5	1.14 ± 0.11	223 ± 17	196 ± 26
EAS03	12	2.0 ± 1.0	0.37	1.61	1.98	5	1.10 ± 0.11	260 ± 27	237 ± 35
EAS04	22	2.0 ± 1.0	0.46	1.84	2.02	5	1.20 ± 0.11	300 ± 21	250 ± 30
EAS05	14	2.0 ± 1.0	0.30	0.90	0.70	7	0.70 ± 0.10	130 ± 14	188 ± 33

Table 1. Summary of sample details, number of aliquots measured (*n*), radioisotope concentrations, equivalent doses (D_e), and sample ages. Radioisotope concentrations are given from ICP-MS/AES measurements for samples EAS04 and EAS05, whilst samples EAS01-03 are based on γ-spectrometry because the pebbly texture made it impossible to obtain a representative sub-sample. Gamma dose rates included within the total are based upon γ-spectrometry apart from in EAS05 where only ICP-MS/AES measurements were available due to the cementation. An internal quartz dose rate of 0.06 ± 0.02 Gy/ka is also included.

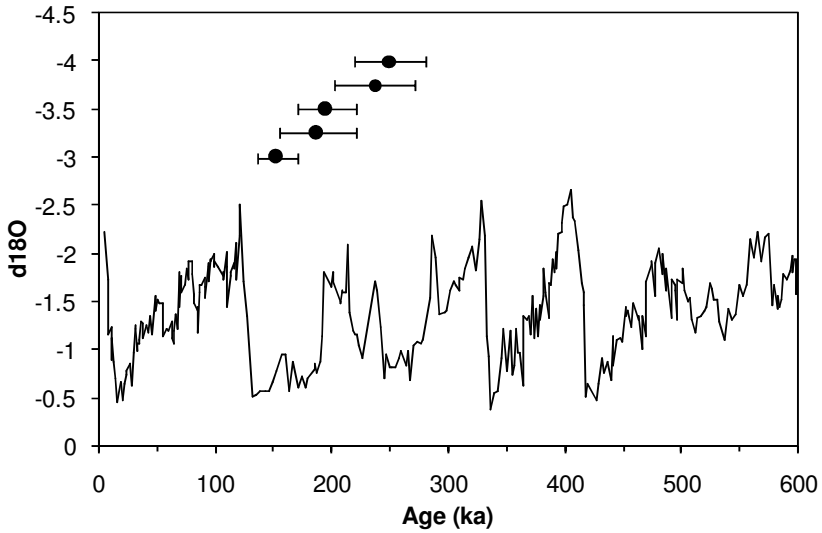

Figure 1. Comparison of OSL ages with the marine isotope record derived from the ODP677 site with MI Stages numbered (Shackleton et al., 1990).