Quartz Optical Dating Report
Bocholt Oudebaan, Belguim

Abstract: Optically stimulated luminescence (OSL) dating was applied to coarse quartz grains extracted from two samples taken from the Bocholt Oudebaan site, Belgium. Samples responded well to OSL measurement. Both samples measured showed reasonable reproducibility and are considered to have been reset prior to burial (although sample Shfd19022 more so). Ages range from 0.92 ± 0.07 to 1.82 ± 0.12 ka and appear to be in stratigraphically consistent.

1. Introduction

A total of two samples from the Bocholt Oudebaan site, Belgium were submitted for luminescence dating by Mr Wouter Yperman (Studie Bureau Archeologie). The samples were assumed not to have been exposed to sunlight during sampling or transportation. All luminescence work was carried out at the Sheffield Luminescence Laboratory (SLL). Upon arrival at SLL, the samples were allocated Sheffield lab numbers (Table 1), which are used throughout this report. This report provides a brief summary of the procedures employed and results obtained for the samples.

Table 1. *Sample descriptive data.*

Lab No.	**Field Reference**	**Latitude** (° N)	**Longitude** (° E)	**Altitude** (m)	**Sampling Depth** (m below present-day surface)
Shfd19022	2018CV125-PR1L4	51°18'	5°06'	42.6	0.90
Shfd19023	2018CV125-PR1L5	51°18'	5°06'	42.6	0.95

In order to derive an optically stimulated luminescence (OSL) age both the palaeodose (De - the amount of absorbed dose since the sample was buried) and the dose rate (the estimated radiation flux for the sedimentary bodies) have to be determined. Bateman (2019) gives a detailed explanation of both these parameters. To calculate an age, the palaeodose (expressed in Grays) is divided by the annual dose rate (Grays/yr). An inherent assumption in these age calculations is that the sediment was fully reset or 'bleached' by exposure to sunlight during the last transport event or whilst *in situ* prior to burial and that no post-depositional sediment disturbance has occurred.

As part of this investigation, efforts have been taken to establish if these sediments have been bleached prior to burial or disturbed by, for example, bioturbation be measuring up to 24 replicates of each sample. As the OSL signal measured at the small single aliquot level is an average of ~900 grains, the true distribution of De values may be masked. Further measurements at the single grain level would have to be made to check if this is an issue or not.

2. Dose Rate Analysis

Naturally occurring potassium (K), thorium (Th), uranium (U) are the main contributors of dose to sedimentary quartz.. The concentrations of these elements were determined by inductively coupled plasma mass spectrometry (ICP) at SGS laboratories Ontario Canada (Table 2). Elemental concentrations were converted to annual dose rates using data from Adamiec and Aitken (1998), Marsh *et al.* (2002), and Aitken (1998). Calculations took into account attenuation factors relating to sediment grain sizes used, density and palaeomoisture (Table 2). Attenuation of dose by moisture used present-day values with a ± 3 % error to incorporate fluctuations through time (Table 2).

Table 2. *Summary of dosimetry related data.*

Lab Code	U (PPM)	Th (PPM)	K (%)	D_{cosmic}+ ($\mu Gy/a^{-1}$)	Moisture (%)	Dose rate† ($\mu Gy/a^{-1}$)
Shfd19022	0.82	2.0	0.6	187 ± 9	5.7	1087 ± 45
Shfd19023	0.91	2.4	0.7	186 ± 9	7.6	1204 ± 51

+ Cosmic dose is calculated as a linear decay curve at depths below 50 cm. Above this depth, errors in calculation may lead to an underestimation of the cosmic dose contribution.

† Total dose is attenuated for grain size, density and moisture.

The contribution to dose rates from cosmic sources was calculated using the expression published in Prescott and Hutton (1994; Table 2). The Prescott and Hutton (1994) algorithm was used to calculated the cosmogenic derived dose rate. The dose rates calculated are based on analyses of the sediment sampled at the present day. This assumption is only valid if no movement and/or re-precipitation of the four key elements has taken place since sediment burial and the adjacent sediments to those sampled had similar dose rates. Further analysis would have to be undertaken to establish whether radioactive disequilibrium is present in the dose rate and whether this has cause errors in age determination.

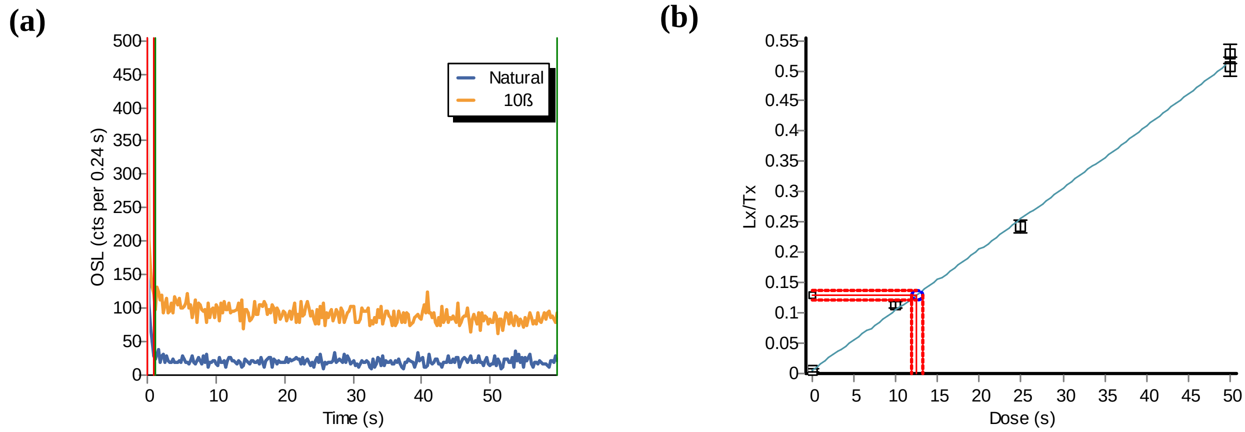

Figure 1 Examples of small aliquot OSL data for sample Shfd19022: (a) OSL decay of naturally acquired signal; (b) SAR growth curve. The red lines in (a) indicate the integration limits for signal measurement, and the green lines background measurement once the signal has been zeroed. In (b) the luminescence response (Lx) to a series of known doses is normalised by test dose response (Tx) and plotted against dose. The red line represents interpolation of the natural dose (De).

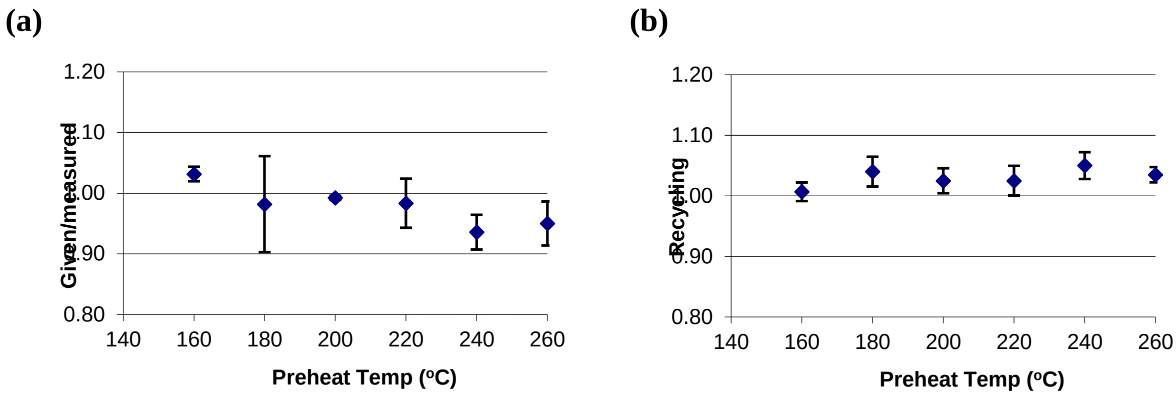

Figure 2. Results of different preheat temperatures in recovering a ~20 Gy beta radiation dose from sample Shfd19022 (a) Given to recovered dose ratio at different preheat temperatures. (b) recycling ratio (ratio between the first and last dose point) at the different preheat temperatures. Data points in both plots are the averages of three measurements performed for each preheat temperature.

3. Palaeodose Determination

Samples were prepared under subdued red lighting following the procedure to extract and clean quartz outlined in Bateman and Catt (1996). Material for dating was taken from prepared quartz isolated to a size range of 125-180 µm. The samples underwent measurement using a Risø DA-20 luminescence reader with radiation doses administered using a calibrated ^{90}strontium beta source. Grains were mounted as a 5 mm diameter monolayer on 9.6 mm diameter stainless still disks using silkospray. Stimulation was with blue/green LEDs and luminescence detection was through a Hoya U-340 filter. Samples were analysed using the single aliquot regenerative (SAR) approach (Murray & Wintle, 2000; Murray & Wintle, 2003), in which an interpolative growth curve is constructed using data derived from repeated measurements of a single aliquot which has been given various laboratory irradiations (Figure 1a and 1b). Five regeneration points were used to characterise growth curves, with the first regeneration point being identical to the last in order to check if sensitivity changes caused by repeated measurement of the same grains are correctly monitored and corrected for by the SAR protocol (known as the “recycling ratio”). The most appropriate preheat temperature for the samples was selected using a dose recovery preheat plateau test (Figure 2). This resulted in selection of preheat temperatures of 200 °C for 10 seconds which was applied to prior to each OSL measurement to remove unstable signal generated by laboratory irradiation.

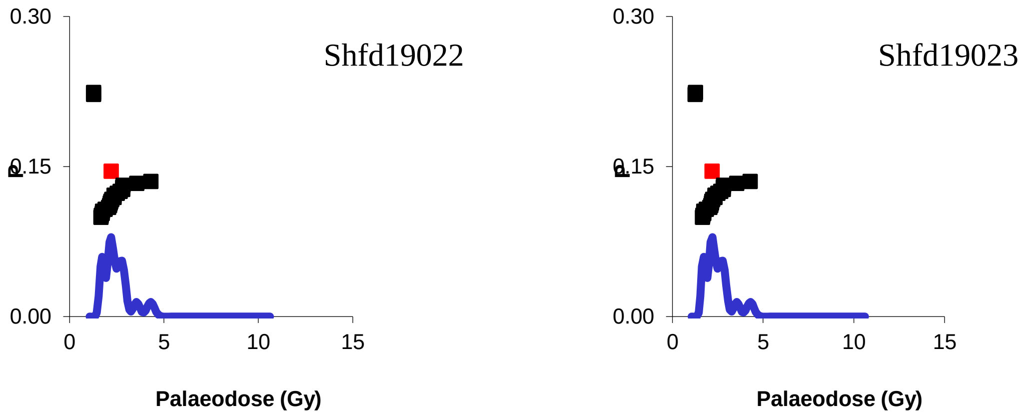

Figure 3 De distribution plots for samples. Blue line is combined probability density for all grains. Black points are results from individual grains. Note dose is scaled as appropriate for data.

De values from individual aliquots were only accepted if they exhibited an OSL signal measurable above background, good growth with dose, recycling values within ± 10 % of unity, and the error on the test dose used within the SAR protocol was less than 20 %. The samples possessed good luminescence characteristics with a rapid decay of OSL with stimulation and OSL signals dominated by a fast component (e.g. Fig. 1a). Within the SAR protocol results which grew well with laboratory dose (e.g. Fig. 1b).

Table 3. *Summary of single grain palaeodose data and ages for Bocholt Oudebaan site.*

Lab Code	Field Ref.	Depth (m)	De (Gy)	Overdispersion (%)	Dose rate (µGy/a^{-1})	Age (ka)
Shfd19022	2018CV125-PR1L4	0.90	1.00 ± 0.06	11	1087 ± 45	**0.92 ± 0.07**
Shfd19023	2018CV125-PR1L5	0.95	2.19 ± 0.11	25	1204 ± 51	**1.82 ± 0.12**

* De extracted using Minimum age model due to high overdispersion.

4. Sedimentary bleaching behaviour and sample saturation

The effects of incomplete bleaching of the sediment during the last period of transport or exposure *in situ* can be profound. Typically, poorly bleached sediments retain a significant level of residual signal from previous phases of sedimentary cycling, leading to inherent inaccuracies in the calculation of a palaeodose value. By plotting the replicate De data for the sample as a probability density function (Figure 3) some assessment of whether older or younger material has been included in the sample measurements can be made. In principle a well-bleached sample that has not been subjected to post-depositional disturbance should have replicate De data which is normally distributed and highly reproducible (see Bateman *et al.*, 2003, Figure 3; Bateman *et al.*, 2007a). Where post-depositional disturbance or incomplete bleaching prior to sample burial has occurred skewing of this distribution may occur and/or replicate reproducibility may be lower (Bateman *et al.*, 2007a; Bateman *et al.*, 2007b). In the case of poorly bleached material skewing should be evident with a high De tail (e.g. Olley *et al.*, 2004). High De tails may also be indicative of saturated samples and interpolation of the De values from the upper, low gradient part of the growth curve (Murray & Funder, 2003).

As Figure 3 demonstrates (see also Appendix 1), the De replicate distributions for sample Shfd19022 is normally distributed (especially after outliers are removed) with a low level of De replicate scatter (OD= 11%). This data shows no indication of either partial bleaching or post-depositional disturbance of this sample. Whilst replicate scatter is higher for sample Shfd19023 (OD=25%), when outliers are removed OS drops to acceptable levels so this sample is also considered to have been well bleached prior to burial. De values for age calculation purposes have therefore been extracted using the Central Age Models (CAM) of Galbraith and Green (1990).

5. Age Calculation and Conclusions

Ages are quoted in years from the present day (2019) and are presented with one sigma confidence intervals which incorporate systematic uncertainties with the dosimetry data, uncertainties with the palaeomoisture content and errors associated with the De determination. Table 3 shows the final OSL age estimates. Aliquot-specific data for both samples is included in Appendix 1. Both samples are considered to have been reset prior to burial (Although samples Shfd19022 more so). Ages range from 0.92 ± 0.07 to 1.82 ± 0.12 ka and appear to be in stratigraphically consistent.

Prof Mark D. Bateman

6. References

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Appendix 1

OSL data and plots for the Bocholt Oudebaan site

Sample specific data including:-

- list of De values derived from individual aliquots
- calculated means based on a range of statistical models
- histogram plot of distribution of De within a sample
- probability density plot (curve) with ranked De data (black points) and probability mean (uppermost red point).

Field Code:	2018C125-pr1l4		**Bocholt Oudebaan**
Lab Code:	**Shfd19022**		**Belguim**
Aliquot Size:	**5 mm**		

Aliquot	Palaeodose (Gy)	error
1	0.991	0.059
2	0.952	0.068
3	0.856	0.062
4	1.111	0.062
5	1.266	0.064
6	1.034	0.084
7	0.972	0.043
8	1.051	0.061
9	1.010	0.046
10	0.863	0.063
11	0.783	0.058
12	1.044	0.069
13	1.006	0.069
14	1.134	0.053
15	0.881	0.057
16	1.260	0.057
17	1.222	0.074
18	0.936	0.049
19	0.928	0.047
20	1.084	0.055
21	1.010	0.049

	De (Gy)	error
Minimum	0.78	0.06
Maximum	1.27	0.06
N	21	

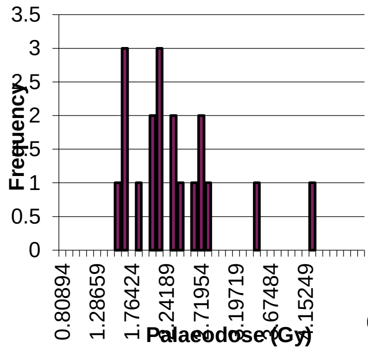
Unweighted	All Data	Minus Outliers
Mean (Gy)	1.02	1.00
SD	0.13	0.10
SE	0.03	0.02
N	21	18

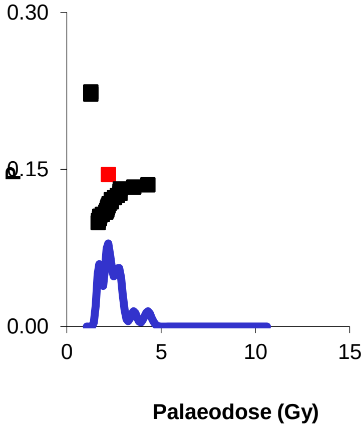
Weighted	All Data	Minus Outliers
Mean (Gy)	1.01	1.00
SD	0.13	0.11
SE	0.03	0.02
N	21	18

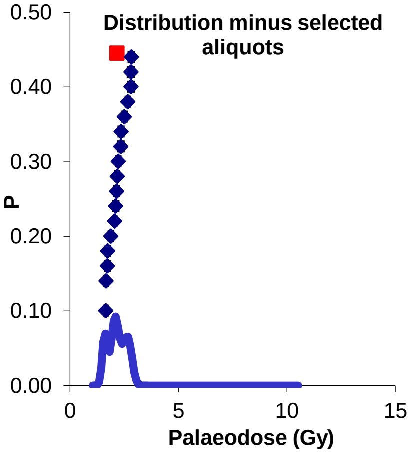
Probability	All Data	Minus Outliers
Mean (Gy)	1.00	1.00
SD	0.12	0.10
SE	0.03	0.02
N	21	18

Central Age Model	All Data	Minus Outliers
Mean (Gy)	1.02	1.00
SD	0.07	0.06
OD (all data)	10.70%	6.70%
N	21	18

De Distribution	**All Data**	**Minus Outliers**
Skewness	0.55	0.01
Kurtosis	-0.17	0.14
Median	1.01	1.01
Sorting	0.12	0.09







Field Code:	2018C125-pr1l5		**Bocholt Oudebaan**
Lab Code:	**Shfd19023**		**Belguim**
Aliquot Size:	**5 mm**		

Aliquot	**Palaeodose (Gy)**	**error**
1	2.513	0.103
2	1.879	0.080
3	2.061	0.069
4	2.833	0.116
5	2.112	0.109
6	2.350	0.112
7	2.811	0.126
8	2.147	0.087
9	1.723	0.096
10	1.738	0.080
11	2.177	0.080
12	3.547	0.120
13	4.294	0.135
14	2.814	0.135
15	2.667	0.085
16	2.355	0.103
17	2.228	0.088
18	1.670	0.067
19	1.653	0.079

	De (Gy)	**error**
Minimum	1.65	0.08
Maximum	4.29	0.14
N	19	

Unweighted	All Data	Minus Outliers
Mean (Gy)	2.40	2.22
SD	0.67	0.41
SE	0.15	0.09
N	19	17

Weighted	All Data	Minus Outliers
Mean (Gy)	2.21	2.11
SD	0.55	0.38
SE	0.13	0.09
N	19	17

Probability	All Data	Minus Outliers
Mean (Gy)	2.19	2.17
SD	0.43	0.37
SE	0.10	0.09
N	19	17

Central Age Model	All Data	Minus Outliers
Mean (Gy)	2.32	2.19
SD	0.15	0.11
OD (all data)	24.58%	17.42%
N	19	17

De Distribution	**All Data**	**Minus Outliers**
Skewness	1.64	-0.21
Kurtosis	2.47	-1.16
Median	2.23	2.18
Sorting	0.24	0.18