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OPTICALLY STIMULATED LUMINESCENCE (OSL) ANALYSES

SCIENTIFIC DATING REPORT

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SUMMARY

Optically stimulated luminescence (OSL) dating of fluvial deposits was attempted at eight sites in the lower Ribble Valley, Lancashire, as part of a study of the aggregate resources and archaeological potential of this area. Considerable difficulties were encountered during the OSL dating programme, which have so far proven insurmountable. The full experimental procedures and testing that were undertaken are detailed in this report. Recommendations for the advisability of, and a methodological protocol for, OSL dating of sediments of similarly provenance and depositional environments are also made.

CONTRIBUTORS

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The Aggregate Extraction and the Geoarchaeological Heritage of the Ribble Valley project was a joint project between the University of Liverpool Geography Department and Oxford Archaeology North, and was funded by the Aggregates Levy Sustainability Fund (ALSF) administered by English Heritage (project 3928). At the University of Liverpool, Dr Richard Chiverrell was the Principal Investigator, examining the long-term (Quaternary) evolution and geoarchaeology of the Ribble Valley. Radiocarbon dating was coordinated by Derek Hamilton of the English Heritage Scientific Dating Service, in collaboration with Dr Peter Marshall.

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INTRODUCTION

In 2005–7, a programme of investigation into the potential impact of aggregate extraction on the archaeological resources of the Ribble Valley, Lancashire, was undertaken jointly by Oxford Archaeology North and the Department of Geography, University of Liverpool. The investigation focussed on two areas within the Ribble basin, an area of the headwaters of the Ribble, to the south of Settle, and the main reach of the Ribble between Clitheroe and Preston, a potential area of aggregate reserves. As part of this programme, optically stimulated luminescence (OSL) dating was attempted at eight sites in the lower part of the catchment (Fig 1). The OSL dating was undertaken at the OSL facility in the Department of Geography, University of Liverpool.

The objective of the luminescence dating programme was to secure the chronological framework for the glacial, fluvial, and hillslope geomorphology, and to provide preliminary chronological control for potential palaeoecological sites within the Ribble catchment. The most significant advance arising from this programme would be a comprehensive understanding of the late Devensian and Holocene evolution of the Ribble.

Luminescence dating was undertaken in concert with a programme of radiocarbon dating funded by the English Heritage Scientific Dating Service, in collaboration with Derek Hamilton and Peter Marshall. The assemblage of samples selected for radiocarbon dating comprised an integrated geochronological package with ten OSL samples, and addressed the core aims of the project.

In the case of the OSL dating, considerable difficulties were encountered during the dating programme. These have proven insurmountable, so the full experimental and testing procedures undertaken are detailed here to advance this approach. Recommendations for the advisability of, and a methodological protocol for, OSL dating of sediments of similarly provenance and depositional environments are also made.

SAMPLING LOCATIONS

Samples were collected by hammering opaque plastic tubes into the sediments on freshly cleaned outcrop faces (Fig 2) or by drilling with a percussion corer using opaque steel tubes. The tubes were recovered and immediately sealed in opaque plastic bags. Care was taken to sample only sediment layers of sufficient thickness for a homogeneous γ-dose rate, and showing no signs of post-depositional changes (bioturbation, soil formation, reduction, or oxidation processes). For each luminescence sample, an additional sample was taken to determine water content and radionuclide concentrations. All samples and sampling locations are listed in Table 1.

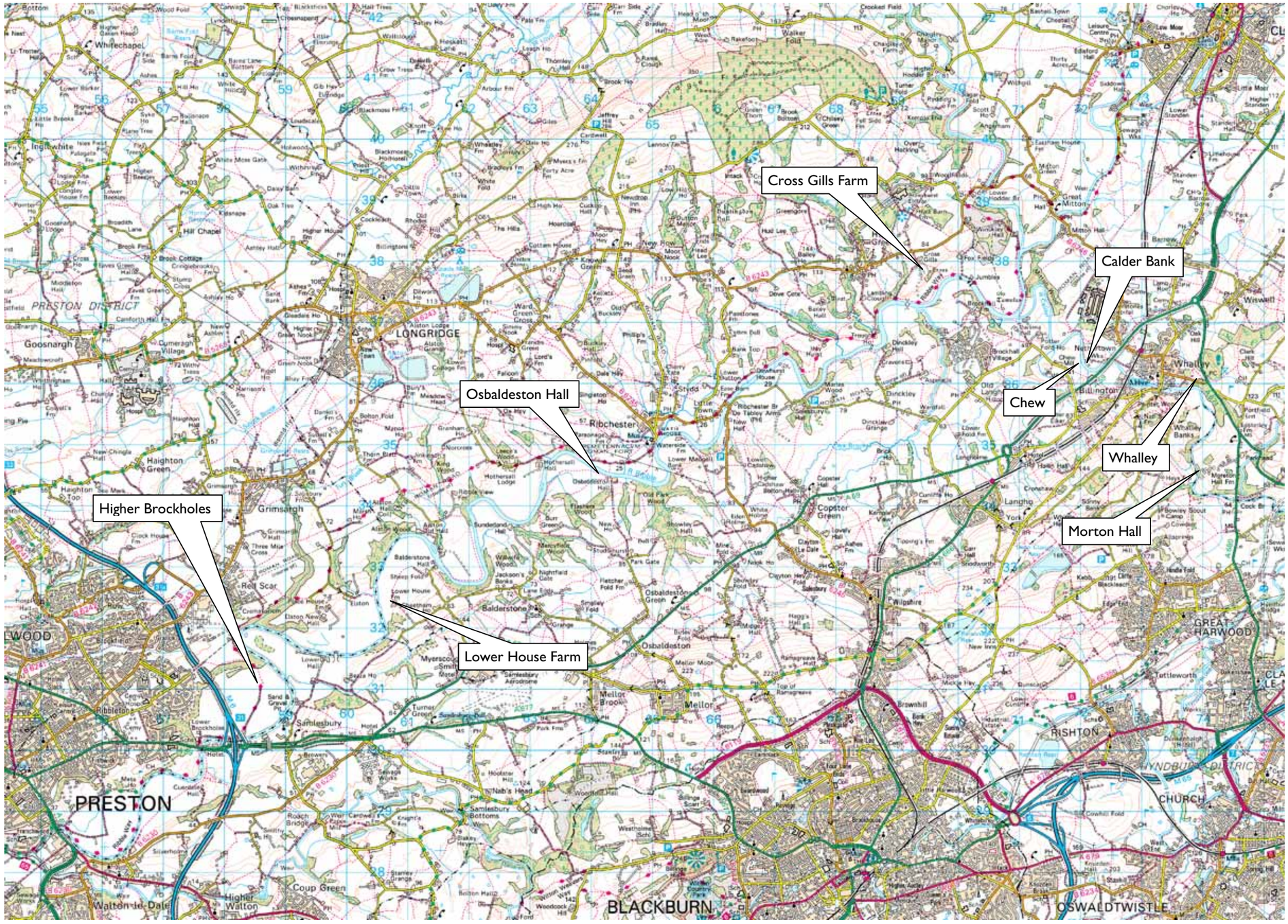

Figure 1: locations of sampling sites, lower Ribble Valley, Lancashire. © Crown Copyright. All rights reserved. English Heritage 100019088. 2007

Table 1: Samples collected for optical dating

Field code	Laboratory code	Grain size for OSL (μm)	NGR	Details
CHEW	LV213	250–300	SD 720 362	Chew Mill (Calder): dug-out section within well-sorted flood sands, 1.5–2.0m below terrace surface
C Bank	LV214	250–300	SD 721 362	Calder Bank: section; flood sand layer within upper channel fill sequence
Morton	LV215	100–300	SD 739 344	Morton Hall: section; well-sorted sand. Back bar within very coarse fluvial gravel terrace
Whalley	LV216	210–300	SD 739 360	Whalley: highest terrace; well-sorted sands overlying probable laminated lacustrine muds and diamict. Sample 3–4m below surface in degraded road-cut section. Coversands?
Brock 1	LV217	250–300	SD 585 311	Higher Brockholes: quarry section flood-laminated sands *c* 2.5m below surface (sampled in large metal tube)
Brock 2	LV218	250–300	SD 584 310	Higher Brockholes: quarry section flood-laminated sands *c* 3m below surface and 0.5m above gravel (sampled in plastic tube)
Cross 1	LV219	250–300	SD 695 378	Cross Gill Farm: Delta section, lower sample coarse sands
Cross 2	LV220	250–300	SD 695 378	Cross Gill Farm: Delta section, upper sample fine sand and silts
OSB T1	LV221	250–300	SD 641 345	Osbaldeston Hall, Terrace, bank section, *c* 1.5m beneath terrace surface
OSB T2 U	LV222	250–300	SD 638 347	Osbaldeston Hall, Terrace 2, bank section, upper sample *c* 1.15m beneath terrace surface
OSB T2 M	LV223	250–300	SD 638 347	Osbaldeston Hall: Terrace 2, bank section, upper sample *c* 1.8m beneath terrace surface
OSB T2 B	LV224	250–300	SD 638 347	Osbaldeston Hall: Terrace 2, bank section, upper sample *c* 2.3m beneath terrace surface
Lower House	LV225	250–300	SD 608 326	Lower House Farm: Terrace 1, sampled with Stitz corer, sample depth 3–4m beneath terrace surface

SAMPLE PREPARATION

The majority of the fluvial sediments have a coarse-grained texture, and sample preparation took into account the dominant grain-size fraction of the sediment and the requirements for luminescence dating. Conventional techniques were applied to extract quartz grains in the size of 200–300μm from the sediment (Mauz *et al* 2002). The quartz sub-sample was subsequently etched in 48% hydrofluoric (HF) acid for 40 minutes to remove the outer α-particle penetrated rim of the grains and to clean the grain surfaces. For all samples the yield of quartz after HF etching was small, indicating highly-fractured quartz grains.

For luminescence measurement, the grains were sprinkled onto stainless steel discs coated with silicon oil. Aliquots of different sizes were produced: 4–5 mm aliquots (~400 grains) and 1–2 mm aliquots (~50-100 grains).

Figure 2: OSL sampling from exposures at Brockholes gravel pit (right); and (left) typical sands targeted for OSL dating that predominantly are reworked Permo-Triassic bedrock

EQUIVALENT DOSE (D_e) DETERMINATION OF QUARTZ SAMPLES

All measurements were conducted with an automated Risø TL/OSL DA-15 reader, equipped with a β-source (~6.8Gy min^{-1}) using blue diodes (470±20nm, delivering ~30mW cm^{-2}) for stimulation, and an ultraviolet-transmitting optical filter for detection. All measurement protocols were based on a single aliquot regenerated dose protocol, using the standard version (Murray and Wintle 2000) or modifications. With a set of reconnaissance samples considerable difficulties were encountered, like feldspar contamination and poor signal/noise ratios. An extended series of test was therefore employed to address these difficulties.

Luminescence components

All samples displayed slowly decaying OSL signals and the OSL signals recorded after extended infra-red stimulation still showed the slow decay This characteristic is commonly observed in feldspars, but is also known from quartz when the fast OSL component is not dominant. The etching procedure employed in sample preparation should be sufficient to remove feldspar grains completely, but may not impact on feldspar inclusions in the quartz grains.

There are two possible explanations for the observation: (i) feldspar inclusions in quartz emit an ultra-violet luminescence under blue-light stimulation, or (ii) the quartz is dominated by medium and slow components. Linearly-modulated OSL measurements (LM OSL; Bulur *et al* 2000) were employed for clarification. Qualitative inspection of the

LM-OSL curves (Fig 3) support the second hypothesis, but as a result of the low signal intensities the first hypothesis cannot be fully ruled out. Using a fitting procedure based on a multiple component function (Choi *et al* 2006) to the LM OSL data, sample LV213 was further analysed. The fitting results (Table 2; Fig 4) indicate that the fast component is almost absent. The OSL recorded in the first seconds of stimulation time seems to derive from an ultra-fast component, which passes rapidly into a medium component. This indicates that the quartz of this particular sample cannot be used for SAR-based optical dating.

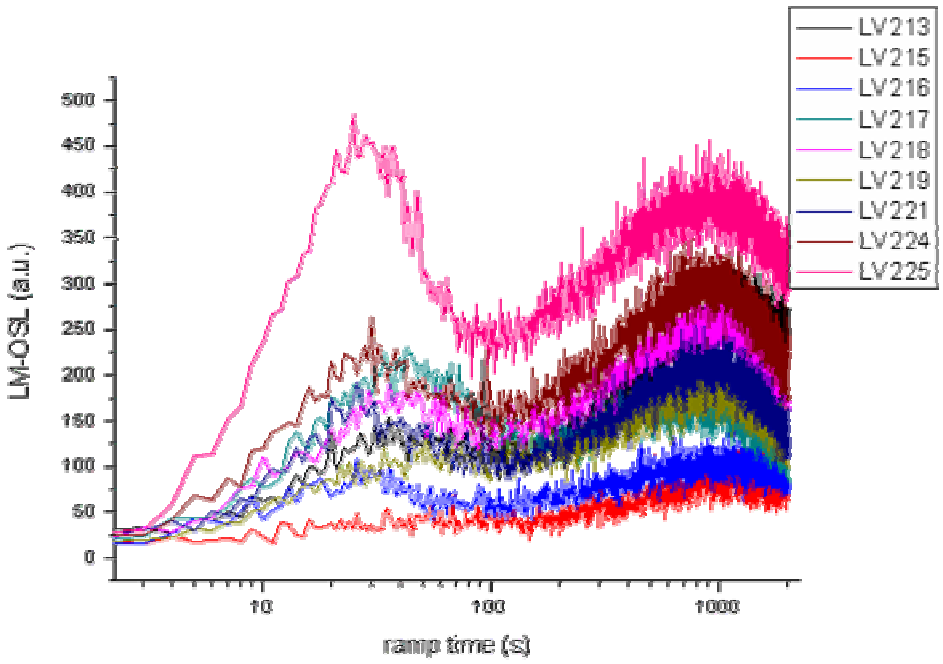

Figure 3: A: Linear-modulated OSL of most of the samples investigated. Ramp time is the time used to ramp the power of the blue light stimulation from 0% to 92%. On all aliquots the natural dose was bleached and a laboratory dose of ~89 Gy given. A preheat of 240°C for 10 s was used and the LM-OSL was recorded at 160°C.

*Table 2: LM OSL components. Results from fitting a five-component function to the LM-OSL curve of LV213 (each component is defined as: I=n*b*t/p*exp(-bt^2/2p, p=2000 s) with n: the initial concentration of trapped electrons; b: the detrapping probability; σ: the photoionisation cross section*

Component	n	b	σ (cm)
1	2719±273	2.97±0.281	4.19^{-17}±4.78^{-18}
2	6676±284	0.500±0.039	7.05^{-18}±7.11^{-19}
3	13170±757	0.043±0.003	6.07^{-19}±5.74^{-20}
4	96904±9030	0.0050±0.0003	7.05^{-20}±6.18^{-21}
5	692569±13966	0.00068±0.00005	9.59^{-21}±9.34^{-22}

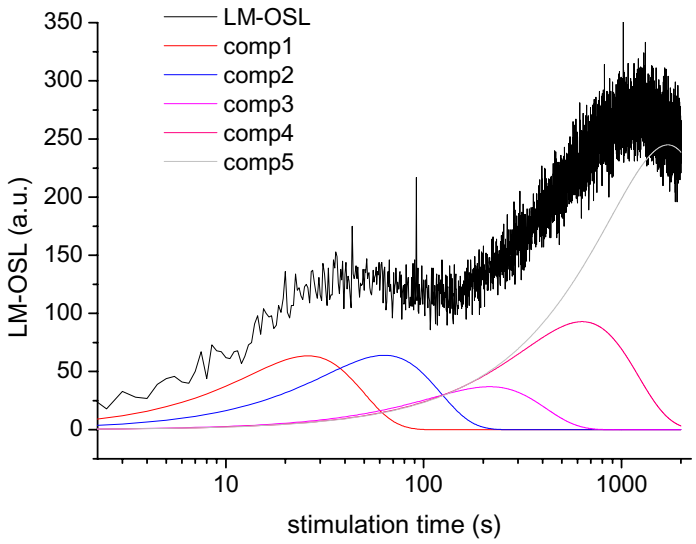

Figure 4: B: LM-OSL analysis of LV213. 5 components were fitted to the measured data (in black)

D_e tests

Four samples were subjected to a standard SAR protocol to assess their D_e. Thermal treatments included a pre-heat of 260°C/10 s and a cut-heat of 220°C. Four major problems were encountered: low luminescence sensitivity; poor response to the SAR procedure (ie poor recycling ratios); possible feldspar contamination; and thermal transfer.

Five samples were then given a pre-heat of 200°C/10 s and a cut-heat of 190°C, in an attempt to avoid thermal transfer and improve recycling ratios. To aid the latter, the size of the test dose was also increased.

All samples, except one (LV217), indicated that a feldspar OSL component still contaminated the quartz luminescence. As it was not feasible to subject the samples to further acid treatment, a SAR method for removing the feldspar component was employed (double SAR protocol involving infra-red stimulation; Banerjee *et al* 2001).

Pre-heat and dose recovery tests

Two samples (LV215 and LV216) were selected for pre-heat tests using the double SAR protocol and including a high-temperature stimulation with blue LEDs at the end of each SAR cycle to prevent thermal transfer. Low sensitivity and poor recycling continued to be major problems.

In a further step, a combined pre-heat/dose recovery test was performed on LV215. The test results indicated that a dose could be recovered but with only a poor precision. Employing a reduced temperature for OSL stimulation (to increase signal-to-noise ratio)

in a similar test on LV216 resulted in a ratio of recovered to given dose of 0.96 with a relative standard deviation of 10% for a pre-heat of 200°C/10s and cut-heat of 190°C. In the next step these parameters, namely double SAR, pre-heat of 200°C/10s and cut-heat of 190°C, were combined with an OSL stimulation at 110°C/20s (to optimise signal extraction) followed by stimulation at room temperature for 40 seconds (to remove all signals before further steps in the procedure). The results of this test are listed in Table 3.

Table 3: Results of dose recovery tests applied to six aliquots of each sample

Sample	Aliquots accepted	Dose ratio	RSD %
LV213	3	0.93	3.5
LV217	3	0.98	5.7
LV218	5	0.97	9.8
LV219	4	1.05	10.3

Estimating the D_e

Encouraged by the results of the dose-recovery tests shown in Table 3, two samples were selected for the application of the modified SAR protocol (24 aliquots of each sample). Medium-sized aliquots (4mm) were used for LV217, and small (2mm) aliquots for LV218. The results were not encouraging, however (Table 4).

Table 4: SAR data for LV217 and LV218 from 24 aliquots of each sample. D_e is given as mean with its standard error.

Sample	Aliquots rejected			Aliquots accepted	D_e (mean ±se Gy)
	Recycling	Thermal transfer	Low signals		
LV217	8	7	6	3	8 ± 2
LV218	1	3	18	1	18 ± 3

Adjustments to SAR

The high-temperature stimulation was re-introduced to the SAR protocol to avoid thermal transfer and applied to three samples (LV221, LV224, and LV225) for a dose-recovery test on six aliquots per sample. Although thermal transfer was reduced to acceptable levels, the high-temperature stimulation affected the pattern of sensitivity changes and was the probable cause of the dose being overestimated for LV221 and LV225 (Table 5). Additionally, an ultrafast component was observed in two aliquots of LV221.

Table 5: Results of dose-recovery tests incorporating a high-temperature stimulation at the end of each SAR cycle. Six aliquots of each sample were used for the test.

Sample	Aliquots accepted	Dose ratio	RSD %
LV221	2	1.12	0.9
LV224	3	0.84	5.7
LV225	4	1.10	3.3

LV225 was the brightest of all the samples, so six aliquots were subjected to a normal

SAR procedure, whilst a further six were given a double SAR protocol in a dose-recovery experiment. All OSL signals were measured at a standard temperature of 125°C. All but one aliquot in the normal SAR group had to be rejected due to feldspar contamination, thermal transfer and poor recycling. The recovered dose was overestimated (ratio 1.15). All aliquots in the double SAR group were rejected due to thermal transfer and poor recycling.

Dose rate

The samples were measured in a high resolution low-level gamma spectrometer and the results for four samples are listed in Table 6. They yielded radionuclide activity data which are expected for a natural environment. The potassium activity is relatively low, indicating that potassium-rich rocks are not abundant in the Ribble catchment.

Table 6: Radionuclide concentrations in samples LV215, LV216, LV218, and LV225

sample code	U (µg g-1)	Th (µg g-1)	K (wt %)
LV215	1.91±0.06	8.04±0.19	0.85±0.02
LV216	2.28±0.07	7.46±0.18	0.84±0.02
LV218	2.03±0.06	8.73±0.21	1.14±0.03
LV225	1.96±0.06	8.00±0.20	1.48±0.03

AGE DETERMINATION

Fluvial sediments generally show skewed equivalent dose distributions as a result of heterogeneous bleaching. Statistical techniques are available to analyse such distributions (e.g.Galbraith *et al* 1999) but can only be successfully applied if single aliquots or small aliquots (containing small numbers of grains) are used (Lang and Mauz 2006). From the OSL test measurements on the Ribble samples it is clear that, given the very low luminescence sensitivity, the application of small aliquots is unfeasible. In addition, poor recycling and thermal transfer are major problems that could not be solved, despite extensive testing and modification of SAR procedures. This renders any OSL ages obtained using the SAR method unreliable.

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