



PERFORMANCE OF DIFFERENT LUMINESCENCE APPROACHES FOR THE DATING OF KNOWN-AGE GLACIOFLUVIAL DEPOSITS FROM NORTHERN SWITZERLAND

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Abstract: Luminescence properties of two samples taken from sand lenses in proglacial outwash deposits of a piedmont glacier that reached the Swiss midlands during the Last Glacial Maximum are investigated in detail. Deconvolution of CW-OSL decay curves shows that the fast component dominates the OSL signal of quartz. The chemistry of single feldspar grains, in particular the K content in different grains, is determined using wavelength dispersive spectrometry (electron microprobe), revealing an average 12.9 wt.% K of the grains contributing to the IRSL signal. D_e distributions are investigated in order to gain insights into partial bleaching, and agreement is found for quartz OSL and feldspar IR₅₀ and pIRIR₂₂₅ ages for small aliquots and single grains when applying the Minimum Age Model. These ages are also consistent with independent age control. For one sample, ages determined using the Central Age Model result in highly overestimated ages for both feldspar and quartz.

Keywords: partial bleaching, single grains, dose distributions, quartz, K-feldspar, potassium content.

1. INTRODUCTION

Natural outcrops and numerous gravel pits composed of till and glaciofluvial deposits in the northern Swiss lowlands represent one of the most complex archives of the Quaternary glaciation history. Graf (2009) defined corresponding sedimentary units by means of lithostratigraphy and identified five Middle to Late Pleistocene glaciations named Möhlin, Habsburg, Hagenholz, Beringen, and Birrfeld (cf. Preusser *et al.*, 2011). The latter glaciation is correlated to the Late Pleistocene and most likely comprises at least three independent glacial advances followed by deglaciation of the foreland (e.g. Ivy-Ochs *et al.*, 2008; Preusser *et al.*, 2011). However, while the relative chro-

nology has been established, there is an urgent need for a reliable numerical chronology for the region to allow correlations with global climatic developments.

A method for dating Pleistocene deposits is radiocarbon which provides precise ages for organic matter found within sediments. Nevertheless, there is usually a lack of organic matter within glaciofluvial sediments and the upper age limit of around 50 ka further limits the application of radiocarbon dating (Hajdas, 2009). Luminescence dating has proven to be a robust method for dating a variety of sediments from different environmental settings (Preusser *et al.*, 2008). Generally two minerals, quartz and potassium-rich feldspar, are used as dosimeters for luminescence dating. When dating quartz the term optically stimulated luminescence (OSL) is used, as stimulation is made by visible blue light. Commonly for feld-

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spar dating, infrared stimulated luminescence (IRSL) is used. Two methods are applied in this article for feldspar dating: IRSL at 50°C (IR₅₀) and IRSL at 225°C following IR₅₀ (pIRIR₂₂₅). A basic pre-requisite of luminescence dating is the resetting of the signal by sunlight prior to deposition. However, the dating of glacier related deposits still bears some methodological difficulties as this requirement is not always fulfilled. Due to short distance transport and the usually high sediment load, the probability of resetting the latent signal in sediment grains transported by glacial melt-water streams is much lower compared to, for example, aeolian transport (e.g. Fuchs and Owen, 2008; Thrasher *et al.*, 2009). This can lead to only partial bleaching of the luminescence signal in the sediment grains and result in overestimation of the apparent luminescence ages. Duller (1994) pointed out that in natural sedimentary systems the luminescence signal will be removed to different residual levels in each individual grain, including grains where the signal is completely removed.

To overcome the problem of partial bleaching various approaches have been developed. One approach is to identify sediments in a section which are likely to be well bleached based on their transport and sedimentation history, and hence glacioaeolian sediments would be preferred over till (e.g. Fuchs and Owen, 2008). However, not all outcrop situations offer a variety of sediment types. Hence, other approaches are required to identify the presence of partial bleaching in samples and determine the burial age. One way of doing so is to measure a large number of aliquots each containing only a few grains. This lowers the averaging effects observed in large aliquots with hundreds to thousands of grains, and increases the chance to isolate completely reset grains (Wallinga, 2002a). Ideally, one would measure individual single grains, but this may be hampered by low numbers of grains exhibiting suitable luminescence signals, often seen in the case of quartz OSL, and hence may be far more time consuming than measuring single aliquots with few grains (Duller, 2008). From the distribution of equivalent doses (D_e) measured, the completely reset population of grains/aliquots at the lower edge of the distribution is extracted using statistical models, such as the 'Minimum Age Model' (MAM; Galbraith *et al.*, 1999); presently the most commonly used approach. All statistical models depend on certain input parameters, for example the expected overdispersion in the case of the MAM, and the proper selection of such parameters can be problematic (Galbraith and Roberts, 2012). In addition, one has to firstly identify whether partial bleaching is actually present in the sample, in order to choose the appropriate statistical model. To do so, Murray *et al.* (2012) have suggested using the comparison of quartz and feldspar palaeodoses to identify well bleached samples, expecting the OSL signal in quartz to bleach more rapidly than the IRSL in feldspar. This test needs to be applied to large

aliquots, and thus on average signals, as other sources of scatter (e.g. dose rate heterogeneity) must be excluded.

As indicated above the two minerals used for luminescence dating have differing characteristics. Quartz has been proven to be a reliable dosimeter in many studies (cf. Preusser *et al.*, 2009), but commonly saturates at relatively low doses and therefore has a limited dating range. Furthermore, the sensitivity (signal intensity) of quartz OSL can be too low to allow for proper detection, as observed, for example, for bedrock samples and glaciofluvial sediments from the South Island of New Zealand (Preusser *et al.*, 2006; Rowan *et al.*, 2012). However, while feldspar can accumulate much larger doses compared to quartz, it is thought to have a lower bleaching rate according to experiments carried out by Godfrey-Smith *et al.* (1988) using a green laser. Fuller *et al.* (1994) therefore proposed that dating of glaciofluvial sediments should be restricted to quartz. A more rapid resetting of quartz OSL compared to feldspar IR₅₀ has also been observed by Thomsen *et al.* (2008) when using a solar simulator in the experiments. In contrast, Preusser (1999a) and Klasen *et al.* (2006) found that the signal resetting behaviour of coarse grain quartz and feldspar were alike when tested under light conditions similar to natural fluvial environments. Another problem related to feldspar is the loss of signal over time, referred to as anomalous fading (Wintle, 1973; Spooner, 1994). When fading is present in feldspar, the age of a sample will be underestimated. While approaches to determine fading rates and correct feldspar ages have been suggested (Huntley and Lamothe, 2001; Auclair *et al.*, 2003), these have sometimes been shown to be problematic, also for samples from the Swiss Alpine foreland (Gaar and Preusser, 2012; Lowick *et al.*, 2012). Alternatively it has been proposed to reduce the problem of fading by using a more stable signal such as the post-infrared-infrared stimulated luminescence (pIRIR; Thomsen *et al.*, 2008; Buylaert *et al.*, 2009). This signal however can have significantly larger residuals and requires a better understanding of their origin (Stevens *et al.*, 2011; Lowick *et al.*, 2012); some work on the better understanding of residuals has been published lately (e.g. Qin and Zhou, 2012; Sohbati *et al.*, 2012). A first study testing the potential of pIRIR for proglacial sediments by Blomdin *et al.* (2012) showed clear evidence for severe incomplete bleaching for samples from Patagonia.

In the above context, there is considerable debate with regard to the levels of partial bleaching present in proglacial sediments. For example, Alexanderson and Murray (2012) concluded that the problem of age overestimation due to partial bleaching was restricted to very proximal (less than 1 km transported from ice front) modern sediments from Svalbard. For Pleistocene samples from Arctic Russia, Thomas *et al.* (2006) argue that partial bleaching is not expected to be a problem and this has led others to generally adopt this assumption and to not investigate proglacial samples for partial bleaching.

In contrast, Klasen *et al.* (2007) and Preusser *et al.* (2007) report clear indications for partial bleaching and age overestimation for sites in Austria and in the Swiss Alpine foreland, respectively, determined from both dose distributions, as well as comparison with independent age control. This controversy has been highlighted by two recent studies along the Hochrhein, *i.e.* the Rhine Valley between Lake Constance and Basel. Kock *et al.* (2009) determined quartz OSL ages for glaciofluvial sediments between 15 ka and 30 ka, hence ages corresponding with the time of the last presence of glaciers in the Alpine foreland. On the other hand, Frechen *et al.* (2010) yielded significantly higher quartz OSL ages, in part from the same outcrops, and concluded a deposition prior to the last glaciation. The difference in the ages has been explained by partial bleaching (cf. Frechen *et al.*, 2012; Preusser *et al.*, 2012), which was deduced by Kock *et al.* (2009) from the D_e distributions, but was assumed not to be ‘dramatic’ by Frechen *et al.* (2010). The major difference between the two studies is that Kock *et al.* (2009) used small (2 mm) aliquots whereas Frechen *et al.* (2010) utilised large aliquots (8 mm), where averaging effects do not allow the detection of partial bleaching.

In the present study, different approaches of palaeodose estimation are investigated on two samples from one outcrop of glaciofluvial sediments of the Swiss Alpine foreland, for which independent age constraints are available. The aim is to compare the suitability of the different approaches and use this information in forthcoming studies in the same region for the dating of similar sediments of unknown age. As both quartz and feldspar offer advantages and disadvantages, we investigate the luminescence properties of coarse grains (200–250 μm) of both minerals (OSL signal component analyses, fading tests). The internal potassium content of the feldspar grains is also determined. A special focus was given to analysis of dose distributions with regard to partial bleaching, and the comparison of quartz OSL with feldspar IR₅₀ and PIRIR₂₂₅ ages.

2. SITE AND SAMPLE DESCRIPTION

The test site is located in central northern Switzerland near the villages of Gebenstorf and Turgi (Fig. 1), between the rivers Reuss and Limmat, next to the confluence of the Rivers Reuss and Aare. The two samples investigated (GE 2 and GE 3), originate from the gravel pit ‘Geelig’ located within the low terrace which is attributed to the Last Glacial Maximum (LGM), the main advance of the Birrfeld glaciation (Graf, 2009). The glacier reached a position *ca.* 5 km upstream of the sampling location during the LGM (Fig. 1b), which is indicated by a terminal moraine.

From other sites, it is known that the foreland of the Swiss Alps was ice free until *ca.* 30 ka ago and that glaciers had disappeared from the area prior to 17.5 ka (cf. Preusser *et al.*, 2011). Peat and overbank deposits in the

former gravel pit of Gossau (which is 45 km southeast of the sampling site towards the Alps) have been dated to 30 ka using radiocarbon, Uranium series and OSL/IRSL. They are overlain by outwash sediments attributed to the LGM and provide a first age constraint with an upper age limit for our samples (Schl chter *et al.*, 1987; Preusser, 1999b; Preusser *et al.*, 2003).

Independent age control for the Gebenstorf site is additionally provided by a radiocarbon age of a mammoth molar which was stored in the palaeontological museum of the University of Z rich. As its label, named the village Turgi as the locality, and the gravel pit ‘Geelig’ is the only pit in the area during historic times, it is attributed to the same gravel pit our samples originate from (Graf, 2009). AMS radiocarbon dating (ETH-17256) yielded $18,150 \pm 140$ ¹⁴C BP, which corresponds to a calibrated age range of 22.09 to 21.45 ka b2k using OxCal 4.1 (Bronk Ramsey, 2009) applying the IntCal09 calibration curve of Reimer *et al.* (2009). However, it has been shown that the dating of bone material can be problematic with regard to later contamination with young humic acids (Hajdas *et al.*, 2009) and we therefore consider that the age may represent a minimum estimate.

Fig. 2 shows the outcrop face of approximately 12 m height where samples were taken. The succession consists mainly of glaciofluvial gravel intercalated with sand lenses of which some have an erosive base. Their thickness varies and can reach several decimetres. The samples were taken from two sand lenses of around 40 cm thickness, vertically centred, so the risk of inhomogeneous radiation fields is considered to be small. Both sand lenses have a similar appearance and are interpreted to originate from the same sedimentary depositional environment, *i.e.* sandy fills of shallow channels of a former braided river system that was fed by melt-water originating from a glacier.

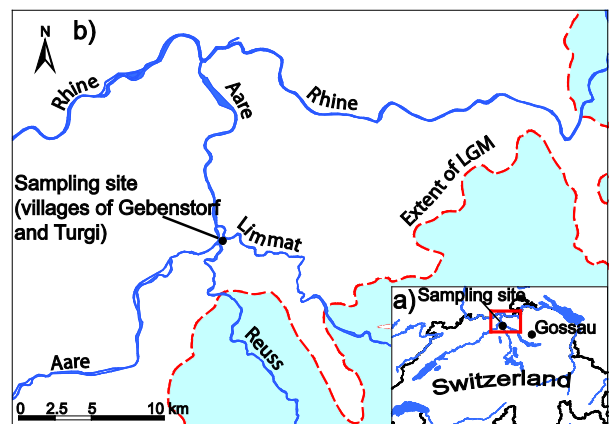


Fig. 1. a) Overview of Switzerland with the sites mentioned in the text. The rectangle indicates extent of map b) which shows the location of the sampling site at Gebenstorf and Turgi between the rivers Reuss and Limmat, next to the confluence of the Rivers Reuss and Aare. Shaded area with the dashed line marks the extent of the ice during the Last Glacial Maximum (adopted from Bini *et al.* (2009)).



Fig. 2. View of the sampling face. Total height of the outcrop is ca. 12 m.

3. METHODOLOGY

Sampling and sample preparation

For sampling, the surface of the outcrop was cleaned and steel tubes were forced into the sediment instantaneously. The samples were emptied into opaque plastic bags avoiding any sunlight exposure. Sample preparation was carried out under subdued red-light conditions in the laboratory. The 200-250 μm fraction was separated by dry sieving. Chemical cleaning consisted of treatments with HCl (32%) and H_2O_2 (30%) to remove carbonates and organics, respectively. This was followed by density separation (2.70 and 2.58 g cm^{-3}) to obtain a quartz-rich and K-feldspar-rich fraction. The quartz fraction was then etched for 60 min in HF (40%) to remove any remaining feldspar and the outer rim of the quartz grains, followed

by another HCl treatment to dissolve fluoride precipitates formed during the HF treatment.

For the luminescence measurements the samples were mounted on stainless steel discs using silicone spray with the aliquot size determined by masks. Aliquot sizes of 6 mm and 2 mm were used for the quartz, 6 mm, 2 mm (in one case for pIRIR) and 1 mm for feldspar, respectively. The different sizes were chosen in order to compare the influence of aliquot size on palaeodose estimation. The average number of grains were counted to be 300 (6 mm), 50 (2 mm), 30 (1 mm) for the different aliquot sizes.

Single grains were measured on aluminium discs with 10×10 holes of 300 μm both in diameter and depth. Single grains were mounted by brushing an excess number of grains over the disc. The chosen grain size (200-250 μm) ensures only one grain per hole. We performed the mounting under a binocular and scrutinised proper placement of the grains in the holes; on average 90% of the holes were filled.

Equipment and equivalent dose determination

Luminescence measurements were made on automated Risø TL/OSL DA-20 readers equipped with 9235QA photomultiplier tubes. For single grain measurements a Risø single grain laser attachment with dual lasers was used. The β -source in the single grain reader has been checked for inhomogeneity of its irradiation field, as this can be of importance for the single grain measurements. Applying the method proposed by Lapp *et al.* (2012) using a β -radiation sensitive self-developing film it was concluded that non-uniformity of the β -source is minor and hence no correction on the single grains measurements was applied.

Palaeodoses were determined using a modified single-aliquot regenerative-dose (SAR) protocol after Murray and Wintle (2000) (Table 1). Several equivalent dose

Table 1. Protocols used in this study. OSL was applied to quartz, IR_{50} and pIRIR₂₂₅ were applied to feldspar. OSL: stimulation with blue LEDs, SG OSL with a green laser, IRSL: stimulation with IR LEDs, SG IRSL with an IR laser. ¹Omitted in 1st cycle to measure L_n , ²only applied in last cycle.

Observed	OSL – multiple grain aliquots	OSL – single grains	IR ₅₀ – multiple grain aliquots	IR ₅₀ – single grains	postIRIR ₂₂₅ – multiple grain aliquots	postIRIR ₂₂₅ – single grains
	¹ Dose	¹ Dose	¹ Dose	¹ Dose	¹ Dose	¹ Dose
	Preheat at 230°C for 10 s	Preheat at 230°C for 10 s	Preheat at 230°C for 60 s	Preheat at 230°C for 60 s	Preheat at 250°C for 60 s	Preheat at 230°C for 60 s
	² IRSL at 50°C for 60 s	² SG IRSL at 50°C for 5 s			IRSL at 50°C for 100 s	IRSL at 50°C for 100 s
L_n/L_x	OSL at 125°C for 60 s	SG OSL at 125°C for 5 s	IRSL at 50°C for 300 s	SG IRSL at 50°C for 5 s	IRSL at 225°C for 100 s	SG IRSL at 225°C for 5 s
	Test dose	Test dose	Test dose	Test dose	Test dose	Test dose
	Preheat at 230°C for 10 s	Preheat at 230°C for 10 s	Preheat at 230°C for 60 s	Preheat at 230°C for 60 s	Preheat at 250°C for 60 s	Preheat at 230°C for 60 s
					IRSL at 50°C for 100 s	IRSL at 50°C for 100 s
T_n/T_x	OSL at 125°C for 100 s	SG OSL at 125°C for 5 s	IRSL at 50°C for 300 s	SG IRSL at 50°C for 5 s	IRSL at 225°C for 100 s	SG IRSL at 225°C for 5 s

(D_e) values from individual aliquots were used to calculate the palaeodose and uncertainty. Preheat temperature was estimated on the basis of performance tests according to Wintle and Murray (2006). Preheat tests are commonly used to assess appropriate preheat temperatures based on a plateau. However, on samples which are potentially partially bleached due to their sedimentary history, there may be no preheat plateau observable due to strongly varying D_e values. Therefore dose recovery tests at different preheat temperatures with given doses of *ca.* 100 Gy were performed in order to obtain a suitable preheat temperature. Bleaching of the samples for these tests was done in the readers using a normal luminescence readout (*i.e.* 60 s blue shine for quartz at 125°C and 300 s IR shine at 50°C for feldspar). The appropriate preheat temperature was chosen where dose recovery was within 10% of unity and sensitivity change during the SAR protocol was low. Preheat temperatures of 230°C were chosen both for quartz and feldspar. Equivalent doses were calculated using Luminescence Analyst 4.11 (Duller, 2013).

Dose response curves were fitted using a single saturating exponential function for both quartz and feldspar. In Fig. 3 we present two dose response curves for a high dose (*ca.* 100 Gy) of a 2 mm quartz aliquot and a 1 mm feldspar aliquot showing a dose similar to the resulting palaeodoses. A measurement error of 1.5% was included in the D_e determination for single aliquot measurements and 2.4% for single grains according to Trauerstein *et al.* (2012); the error on curve fitting is included.

Measurements on quartz

Stimulation was done using blue LEDs ($\lambda = 470$ nm) delivering *ca.* 35 mW cm⁻² at sample position. The signal was detected through a 7.5 mm Hoya U-340 transmission filter which blocks wavelengths above 390 nm with a peak transmission at 340 nm. Single grain quartz measurements were performed using a Nd:YVO₄ solid state diode-pumped laser emitting at 532 nm with signal detection through a 2.5 mm Hoya U-340 filter. Decay curves for quartz OSL are shown in Figs. 4a and 4d.

Continuous wave (CW) decay curves of quartz can be fitted to several components (Smith and Rhodes, 1994). Each of the components has the form

$$L(t) = n_0 b \exp(-bt) \quad (3.1)$$

(McKeever and Chen, 1997, p. 653) where, L is the luminescence intensity at time t , n_0 is the initial concentration of trapped electrons and b is the detrapping probability. The components are commonly referred to as fast, medium and slow components. For OSL dating the fast component is usually used, although the medium component cannot be completely isolated from this. Problems arise when the medium component is unstable and its influence on the fast component varies strongly as observed by Steffen *et al.* (2009). A short integral is therefore preferred for quartz, in order to minimise the contribution of

a possibly unstable medium component on the fast component. Deconvolution was carried out on several decay curves from aliquots of both samples in order to confirm that the fast component dominates the signal. An example of sample GE 2 is presented in Fig. 5, showing a dominance of the fast component during the integration interval. The relative contribution of the medium component to the total signal is almost identical for both the natural as well as the regenerated dose signal. Therefore we consider the medium component to be stable (*cf.* Steffen *et al.*, 2009). This is supported by the fact that palaeodoses do not differ significantly from each other when using either 0.4 s or 1 s signal integration intervals and applying a late background subtraction. Applying approaches to reduce the contribution of the medium component, such as an early background subtraction (Cunningham and Wallinga, 2010), are not necessary for our samples and only create poorer counting statistics for the

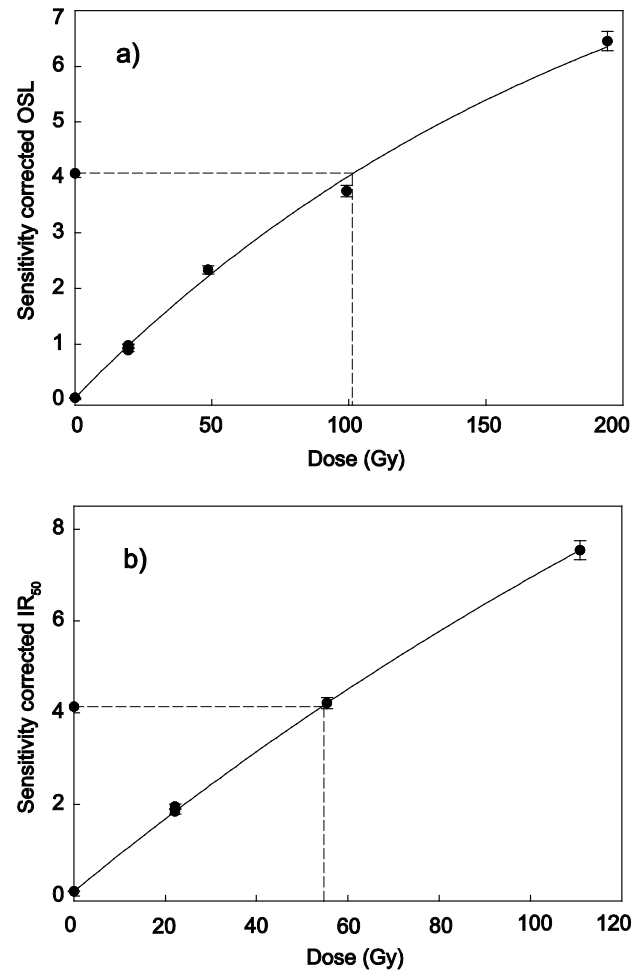


Fig. 3. Dose response curves for a) a relatively high dose (*ca.* 100 Gy) of quartz OSL (Sample GE 3) and b) a dose which represents the expected age of feldspar IR₅₀ (sample GE 2). Note near-linear relationship between sensitivity corrected IR₅₀ and doses up to 110 Gy. Two higher dose points are not shown to improve readability of the area of interest.

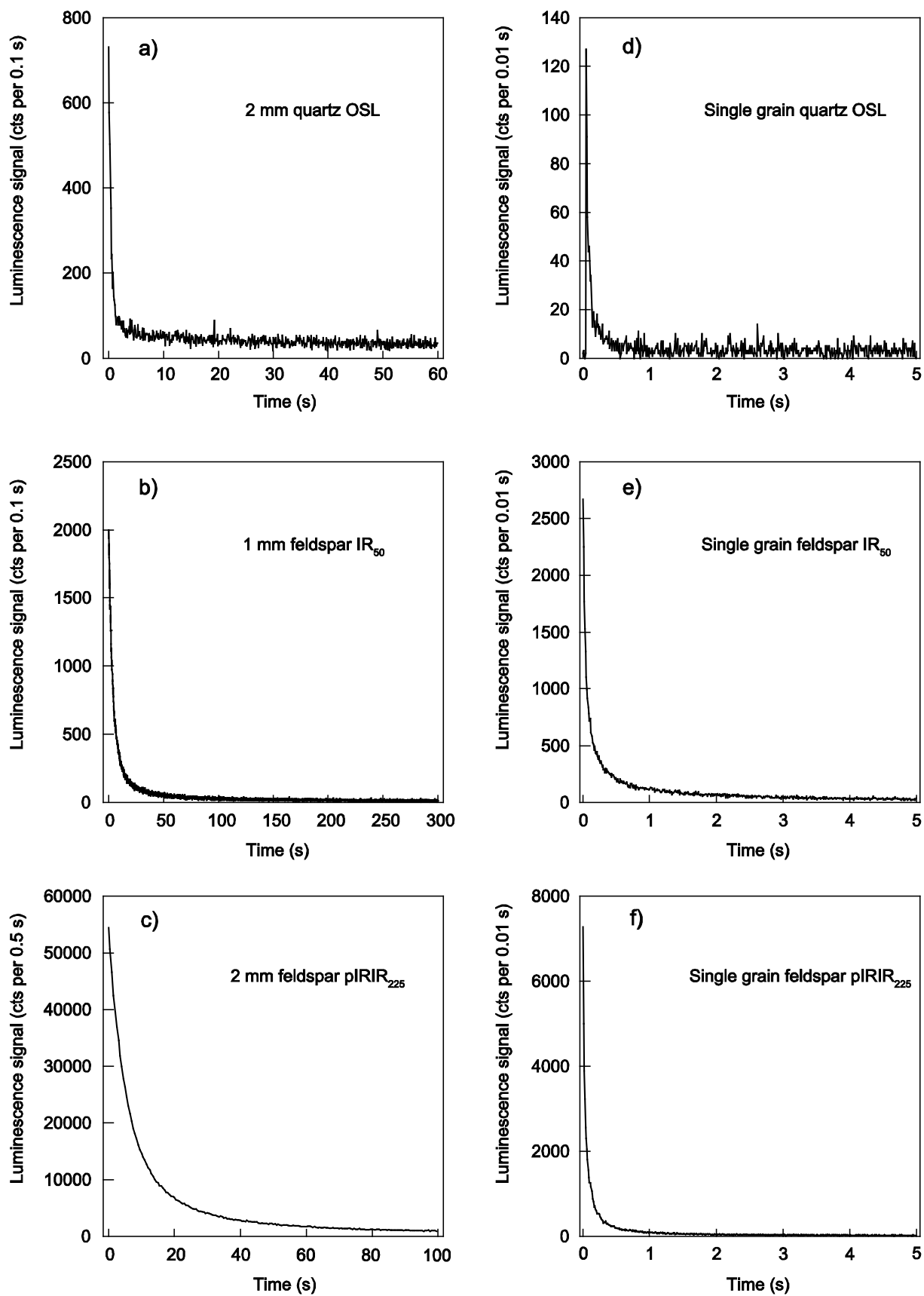


Fig. 4. Natural decay curves of selected aliquots from sample GE 3 (plots a to e) and GE 2 (plot f).

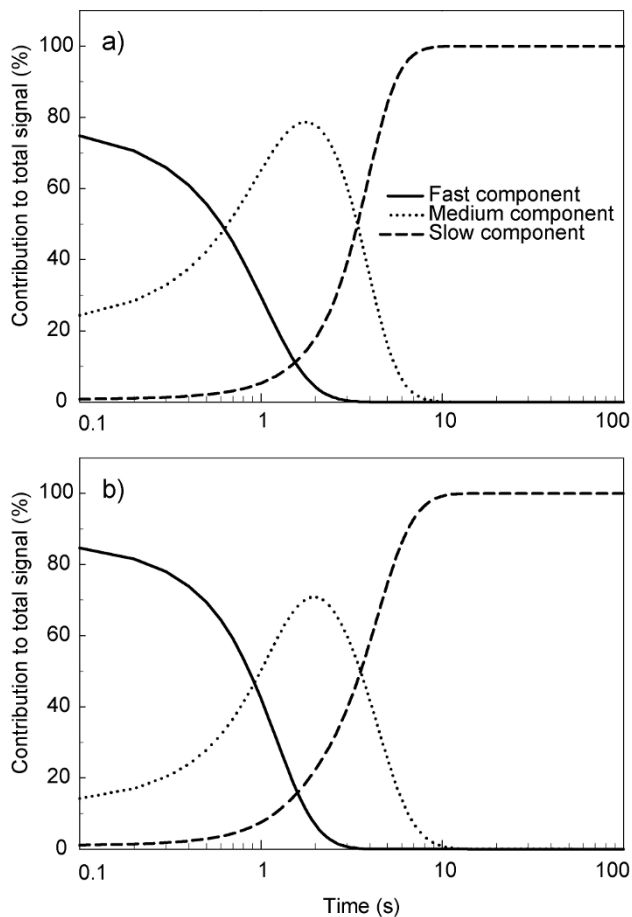


Fig. 5. Deconvolution of decay curves from sample GE 2 quartz (sample GE 3 displayed similar behaviour). a) natural decay curve, b) laboratory regenerated decay curve. A dominance of the fast component during the integration interval (0.4 s) and a stable medium component (only minor change between natural and laboratory regenerated dose) are shown.

relatively dim samples under consideration here. Hence, a conventional late background subtraction was used. For multi grain aliquots the first 0.4 s, minus the signal between 40 s and 60 s for background estimation was used to calculate D_e . The signal integral of the first 0.04 s minus the background estimate between 2 s and 5 s was used for single grains.

Acceptance criteria for quartz

Aliquots (multi grain and single grain) were rejected when the signal was less than three times the background, where test dose uncertainty was larger than 20%, or when uncertainty of D_e was larger than 30%. Murray and Wintle (2000) propose a recycling ratio rejection criterion within 10% from unity. However, for quartz with low sensitivity counting statistics may be poor, due to the low signal intensity. This is especially valid for low doses from small aliquots or single grains; hence the recycling ratio might not meet this criterion all the time. Rejecting

these aliquots would bias the D_e distribution towards higher doses and decrease the size of the statistical basis (number of D_e). Both effects are problematic when statistical models need to be applied. Applying the strict rejection criterion of 10% variability reduces our dataset for the 2 mm quartz aliquots to 23 out of 72 aliquots (GE 2), and 41 out of 67 aliquots (GE 3). The single grains are reduced to 52 out of *ca.* 4140 (GE 2) and 43 out of *ca.* 1890 (GE 3). Allowing a recycling ratio within 20% of unity includes 34 out of 72 2 mm aliquots (GE 2) and 58 out of 67 (GE 3). The single grain distribution contains 111 out of 4140 for sample GE 2, whereas 76 grains of the *ca.* 1890 measured grains of sample GE 3 pass. The mean dose does not change significantly using one or the other recycling ratio criterion: using the 10% criterion on 2 mm quartz aliquots of sample GE 2 results in mean dose of 69.3 ± 6.5 (CAM: 63.3 ± 5.5) whereas the 20% criterion yields a mean of 69.0 ± 5.0 (CAM: 63.7 ± 4.3). We therefore use a recycling ratio rejection criterion within 20% of unity for the quartz samples.

To minimise the contribution of a possible feldspar contamination to the quartz measurements we apply the OSL IR depletion ratio as proposed by Duller (2003) for both multi grain aliquots and single grains. For a comparison of quartz and feldspar, Duller (2003) reports a bimodal distribution of OSL IR depletion ratios where the feldspars are clearly separated (ratios lower than 0.4) from the quartz (ratios greater than 0.8). Based on this study we consider aliquots and single grains as being quartz-dominated when the OSL IR depletion ratio is above 0.8.

Recuperation, expressed as the response to a zero dose compared to the natural dose, was generally low in our samples; on average, for quartz OSL recuperation was less than 5%. D_e values that show more than 10% recuperation were rejected following arguments outlined in the section on feldspar measurements.

Saturation levels were monitored for all measurements and D_e values larger than $2D_0$ were rejected as suggested by Wintle and Murray (2006). For our samples, only 20 D_e values for single grains of sample GE 2 were rejected applying this criterion. We therefore consider the problem of biasing our distribution towards lower doses as minor. In addition we use age models which are not strongly dependent on values at the upper end of a distribution.

Altogether, 47-86% of the OSL measurements on multi grain aliquots pass the above discussed criteria, and only 3% of the quartz single grains.

Measurements on feldspar

For multiple grain feldspar aliquots stimulation was done using IR-LEDs ($\lambda = 870$ nm) delivering *ca.* 115 mW cm^{-2} at the sample position. Single grain feldspars were stimulated using an 830 nm IR-laser in the Risø single grain laser attachment. The signal was detected through a L.O.T.-Oriel D410/30 nm interference filter

and one Schott BG-39 (effective detection wavelength of 410 ± 30 nm). Decay curves for IRSL are given in **Figs. 4b** and **4e** (IR_{50}) and **Figs. 4c** and **4f** ($PIRIR_{225}$)

There is controversy whether the IR_{50} signal consists of multiple components or not. While it was originally concluded that the IRSL signal comprises only one component (Hütt *et al.*, 1988; Trautmann *et al.*, 2000), Tsukamoto *et al.* (2006) identified different components with different stabilities using time resolved luminescence. To investigate if the IRSL signal in our samples consists of different components, we calculated D_e with different integration intervals (**Fig. 6**) and found no dependency between D_e and the integration interval within the first 10 s. Calculation of D_e for multi grain aliquots using IR_{50} , utilises the first 10 s, minus background estimation using the final 50 s. For IR_{50} single grain measurements we use the first 1 s, and use the final 2 s for background estimation.

Acceptance criteria for feldspar

Acceptance criteria for IRSL are the same as for OSL with the exception of the recycling ratio where feldspar samples are rejected when the recycling ratio is outside 10% of unity following Murray and Wintle (2000). Recuperation with the IR_{50} protocol is low (below 2% on average) for multiple grain aliquots, but higher for single grains (7.3% on average for sample GE 3 and 5.0% for sample GE 2). Setting the threshold to 5% reduces the dataset of IR_{50} single grains for sample GE 3 to 24 D_e values instead of 125 values when a threshold of 10% is applied. This supports the observation of Li *et al.* (2011), who found that a threshold of 5% would not change the resulting dose of the distribution compared to a 10% threshold, but strongly decreases the number of values in the distribution. We therefore only reject aliquots that show recuperation larger than 10%. Some D_e values of

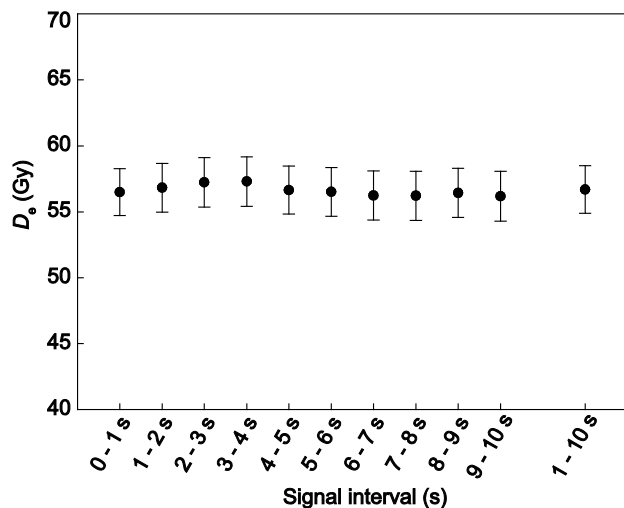


Fig. 6. Plateau plots on feldspar IR_{50} showing independence of integration interval and resulting D_e (sample GE 3, 6 mm aliquots).

sample GE 2 had to be rejected as they were above the saturation level (8% of the small aliquots, 3% of the single grains). In total, more than 70% of the multi grain aliquots measured with IR_{50} pass these criteria; for the IR_{50} single grains it is about 17%.

Fading of IR_{50} signal

Fading tests on multi grain aliquots were carried out on the feldspar fraction of both samples in order to detect anomalous fading. The fading tests were based on delayed L_x/T_x measurements, with the preheat directly following irradiation and different storage times before L_x measurements (Auclair *et al.*, 2003). Delay of the measurements was up to 10 hours after irradiation. The L_x/T_x ratios measured after different delays are plotted against the time delay between irradiation and IR_{50} measurement on a log scale, the calculated percentage of signal loss per decade is referred to as the g -value (Aitken, 1985).

The fading test on three multiple grain aliquots of sample GE 3 results in a mean g -value of $2.5\pm 0.1\%$ per decade (**Fig. 7**). The same test on Sample GE 2 yields a mean of $2.6\pm 0.3\%$ per decade. These g -values are used for fading correction using the R-package 'Luminescence' (Kreutzer *et al.*, 2012) which corrects according to Huntley and Lamothé (2001). This correction is restricted to D_e values fitting to the linear part of the dose response curve. Our samples have palaeodoses that lie in the (quasi) linear part of the dose response curve (cf. **Fig. 3b**). The fading rates assessed are moderate and in the same range as samples from other sites in the region presented by Lowick *et al.* (2012). The uncorrected and corrected ages are presented in **Table 2**.

$PIRIR_{225}$

The $PIRIR$ measurements on multi grain aliquots were done using the same $PIRIR_{225}$ protocol used by Lowick *et al.*

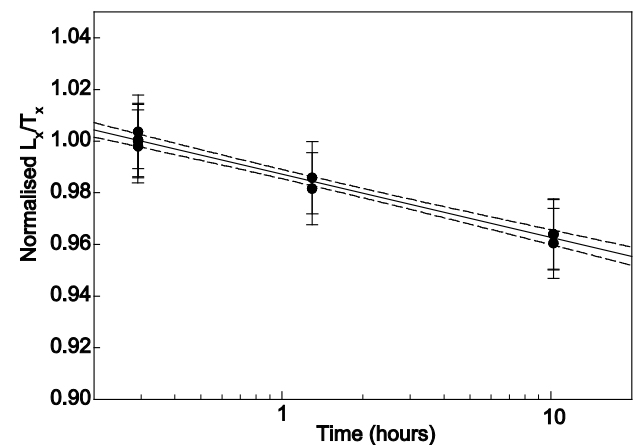


Fig. 7. Example of a fading test on feldspar from sample GE 3, for which a mean g -value of $2.5\pm 0.1\%$ was assessed (based on 3 aliquots). Solid line: regression through data, dashed lines: 95% confidence intervals.

Table 2. Palaeodoses and ages of different aliquot sizes and different age models. SG: single grains. n_{tot} : total number of aliquots or grains (SG) analysed (grain number assumed on 90% filling of SG discs). n : number of D_e used for palaeodose calculation. OD: overdispersion of D_e distribution. σ_b : value used in the MAM. CAM: central age model. MAM: minimum age model. n/a.: not applicable to 6 mm aliquots due to averaging effect on large aliquots (see discussion in text). *pIRIR₂₂₅: a mean residual has been subtracted from each D_e .

Sample	Mineral	Protocol	Aliquot size	n_{tot}	n	OD	σ_b	Palaeodose (Gy)		Age (ka)		Age (ka) fading corrected	
								CAM	MAM	CAM	MAM	CAM	MAM
GE 2	F	IR50	6 mm	12	12	0.22	n/a	228.0±14.7	n/a	107.8±9.7	n/a	141.8±13.3	n/a
GE 2	F	IR50	1 mm	61	36	0.71	0.18	119.0±14.0	49.4±7.2	56.2±7.5	23.4±3.7	73.2±9.8	30.0±4.6
GE 2	F	pIRIR225	1 mm	48	26	0.58	0.18	204.2±23.6	85.7±13.9	96.5±12.7	40.5±7.1	-	-
GE 2	F	*pIRIR225	1 mm	48	26	0.63	0.18	186.4±25.2	69.1±12.0	88.1±12.5	34.6±6.0	-	-
GE 2	F	IR50	SG	720	139	0.77	0.28	129.1±8.57	45.1±5.3	61.0±5.61	21.3±2.9	79.2±8.8	27.3±5.2
GE 2	F	pIRIR225	SG	810	136	0.68	0.28	108.6±6.4	50.0±6.4	51.3±4.5	23.6±3.4	-	-
GE 2	F	*pIRIR225	SG	810	136	0.81	0.28	90.1±6.3	31.5±3.9	42.6±4.0	14.9±2.1	-	-
GE 2	Q	OSL	6 mm	7	6	0.23	n/a	57.3±5.4	n/a	44.4±4.8	n/a	-	-
GE 2	Q	OSL	2 mm	72	34	0.39	0.19	63.7±4.3	42.5±5.5	49.4±4.3	32.9±4.6	-	-
GE 2	Q	OSL	SG	4140	111	0.63	0.32	66.8±4.1	35.2±4.9	51.8±4.3	27.3±4.1	-	-
GE 3	F	IR50	6 mm	12	12	0.12	n/a	54.6±1.9	n/a	25.0±1.9	n/a	31.7±2.4	n/a
GE 3	F	pIRIR225	2 mm	12	12	0.13	0.18	61.4±2.3	60.0±5.1	28.3±2.1	27.5±3.0	-	-
GE 3	F	*pIRIR225	2 mm	12	12	0.15	0.18	53.2±2.3	51.5±4.8	24.4±1.9	23.6±2.7	-	-
GE 3	F	IR50	1 mm	52	48	0.25	0.18	50.9±1.8	44.9±6.1	23.4±1.7	20.6±3.1	29.6±2.3	26.1±3.9
GE 3	F	IR50	SG	720	125	0.44	0.28	59.4±2.4	47.7±10.7	27.2±2.1	21.9±5.1	34.5±2.8	27.7±6.8
GE 3	Q	OSL	6 mm	12	9	0.17	n/a	41.7±2.4	n/a	30.6±2.5	n/a	-	-
GE 3	Q	OSL	2 mm	67	58	0.23	0.19	39.6±1.2	35.5±4.2	29.1±1.9	26.1±3.4	-	-
GE 3	Q	OSL	SG	1890	76	0.35	0.32	44.1±2.0	39.3±5.9	32.4±2.4	28.9±4.7	-	-

al. (2012). Single grain pIRIR₂₂₅ was performed using a modified version of the protocol of Reimann *et al.* (2012). Details for both protocols are shown in **Table 1**. D_e calculation is based on the first 2 s of the decay curve, and using the last 20 s for background estimation for the multiple grain aliquots, and on the first second minus the last 2 s for the single grains. Acceptance criteria are the same as for the IR₅₀ measurements mentioned above. Recuperation with the pIRIR₂₂₅ protocol is on average below 4% for the selected aliquots. 3% of the single grains and one multigrain aliquot measured with the pIRIR₂₂₅ protocol were rejected for being in saturation. About 17% of the measured single grains pass the rejection criteria. For multi grain aliquots the acceptance ratio varies between 54% (sample GE 2), and 100% (sample GE 3).

Residuals and dose recovery tests (given dose of *ca.* 100 Gy) were measured after bleaching aliquots for up to 30 h under incandescent light with a daylight spectrum (Sunlux Ambiance bulb); additional light sources, which may have contributed to bleaching were conventional indoor fluorescent illumination and window-filtered sunlight.

External dose rate determination

The concentration of dose-rate relevant elements (U, K, Th) was determined using high-resolution γ -spectrometry on bulk sediment samples of *ca.* 450 g (cf. Preusser and Kasper, 2001); results and resulting dose rates are shown in **Table 3**. No indication for radioactive disequilibria in the Uranium decay chain is observed, when comparing the activity of ²³⁸U and ²²⁶Ra (cf. Preusser and Degering, 2007). The dose-rate is calculated using ADELE software (Kulig, 2005), which follows Prescott and Hutton (1994) for the cosmic dose rate and uses the conversion factors of Adamiec and Aitken (1998). A sediment overburden of 6 m (sample GE 2) and 9 m (sample GE 3) with a density of 2 g cm⁻³ is assumed during burial, representing the present day depth situation. The calculated contribution from cosmic radiation is 83 mGy ka⁻¹ (GE 2) and 104 mGy ka⁻¹ (GE 3), respectively. The values for cosmic contribution are taken into account with a relative uncertainty of 10%.

The efficiency of alpha particles in causing radiation damage (alpha efficiency, a -value) is assumed based on

Table 3. Results of high-resolution γ -spectrometry and resulting dose rates during burial. (Q: quartz, F: feldspar).

Sample	K (%)	Th (ppm)	U (ppm)	Cosmic dose rate (mGy ka ⁻¹)	Total dose rate to Q (Gy ka ⁻¹)	Total dose rate to F (Gy ka ⁻¹)
GE2	0.84±0.02	3.42±0.09	1.53±0.03	83	1.29±0.07	2.12±0.13
GE3	0.88±0.02	3.52±0.16	1.51±0.04	104	1.36±0.08	2.18±0.14

literature values of the geographically closest assessments. For the feldspar an a -value of 0.05 ± 0.01 was applied (Preusser, 1999b; Preusser *et al.*, 2001). Since the affected rim is removed by HF-etching of coarse grain quartz, the a -value is not relevant for palaeodose estimates on this mineral.

Field water content is assessed by drying the sample at 50°C . A water content of 12% (of dry mass) for both samples was measured. To account for possible change in sediment moisture in the past, a value of $12 \pm 4\%$ is used to correct for the attenuation of radiation by water.

Internal dose rate of feldspar

To assess the internal dose rate for feldspars an accurate estimate of the potassium content is crucial since the unstable ^{40}K isotope (β^+ and β^- decay) is the main source of internal radiation within feldspar grains. In general, a content of 12.5 ± 0.5 wt.% K following the recommendation of Huntley and Baril (1997) is often assumed. Lamothe *et al.* (1994) measured potassium contents in single feldspar grains using a microprobe, and obtained a mean of 11.66 wt.% K. Measurements on a coastal dune sample from the North Island of New Zealand using laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) by Smedley *et al.* (2012) shows lower values (assumed to originate from perthitic exsolution structures) and these authors suggest a potassium content of 10 ± 2 wt.% as an appropriate value for dating.

We used electron microprobe analysis (EMPA) to assess K-contents on single grains of sample GE 3 that had previously been measured for their luminescence to enable a comparison of IR_{50} signal and K-content. After the luminescence measurements were performed, the grains were fixed within the single grain discs by dropping liquid, quick hardening cyanoacrylate onto the discs. Later the discs were embedded in acryl, ground down in order to access the interior of the grains, and the surface was polished. To avoid charging during measuring the polished surface of the discs was carbon coated.

The EMPA were carried out on a JEOL 8200 SUPERPROBE. Reproducibility was tested by repeated measurements within homogeneous phases on different grains. The average on the standard deviations of those measurements yielded a reproducibility of 0.13% for K. Calibration was tested on a K-rich feldspar ($[\text{K}_{0.65}\text{Al}_{0.32}\text{Si}_3\text{O}_8]$) standard with 9.272 wt.% K. Repeated measurements on the standard yielded 9.44 wt.% K and we therefore deduce a relative uncertainty of 2% on these measurements.

4. RESULTS

Internal dose rate of feldspar

Scanning electron microscopy is able to display different chemical phases based on backscattered electrons (BSE). Visual inspection of the grains using the BSE

pictures before the EMPA measurements revealed no evidence for perthitic exsolution lamellae. Multiple measurements on grains showing different phases did not reveal different potassium contents of the phases. Hence, our measurements, despite being measured on small portions of the grains, reflect the chemical composition of the entire grain. For a total of 52 different grains, both luminescence and chemical composition were measured. Of these 52, only three grains were quartz and one grain was Na-feldspar (albite). Four further feldspar grains had an intermediate composition between the Na- and K-end members with less than 10 wt.% K. The remaining 44 grains were of a composition higher than 10 wt.% K. This confirms that the density separation process is effective in isolating K-rich feldspar. For luminescence dating it is important which feldspars give a signal that passes the rejection criteria. For 27 of the original 52 grains, a D_e could be calculated in dose recovery tests. The albite grain gave a very weak signal in the given detection window and did not pass rejection criteria; two grains with intermediate composition passed, and these feldspars would therefore be included in the calculation of palaeodose. The average composition of all K-feldspars yielding a D_e is 12.9 ± 0.4 wt.% K. As our analysis covers a rather small number of grains we do not use the measured value, but rely on the data of Huntley and Baril (1997), which are confirmed by our measurements but are statistically more robust. However, we use a value of 12.5 ± 1.0 wt.% K to account for the 95% confidence interval as discussed by Huntley and Baril (1997).

D_e distributions

The two samples GE 2 and GE 3 are expected to be of very similar depositional age. Their total dose rates differ only by about 5% for quartz and less than 3% for feldspar (Table 3). Therefore the D_e values (and the resulting palaeodoses) can be directly compared. The two samples have distinctly different distributions as shown in Fig. 8. Only distributions of small multigrain aliquots and single grains measured with the OSL and IR_{50} protocol are compared. For the 6 mm aliquots and the pIRIR₂₂₅ protocol a smaller number of aliquots were measured and so the statistical requirements for a representative distribution are not fulfilled.

Sample GE 3 has relatively narrow, near Gaussian distributions for the approaches shown in Fig. 8. Together with the low overdispersion values (Table 2), this is interpreted to indicate complete bleaching of the luminescence signal prior to deposition. It can, therefore, be assumed that applying the CAM to these distributions will yield accurate burial doses. However, Fig. 8 reveals for each of the distributions of sample GE 3 (open symbols) a few D_e values with significantly higher doses, which could reflect incomplete resetting prior to deposition for these few aliquots.

Sample GE 2 (Fig. 8, closed symbols), in contrast, shows wide and positively skewed distributions in all

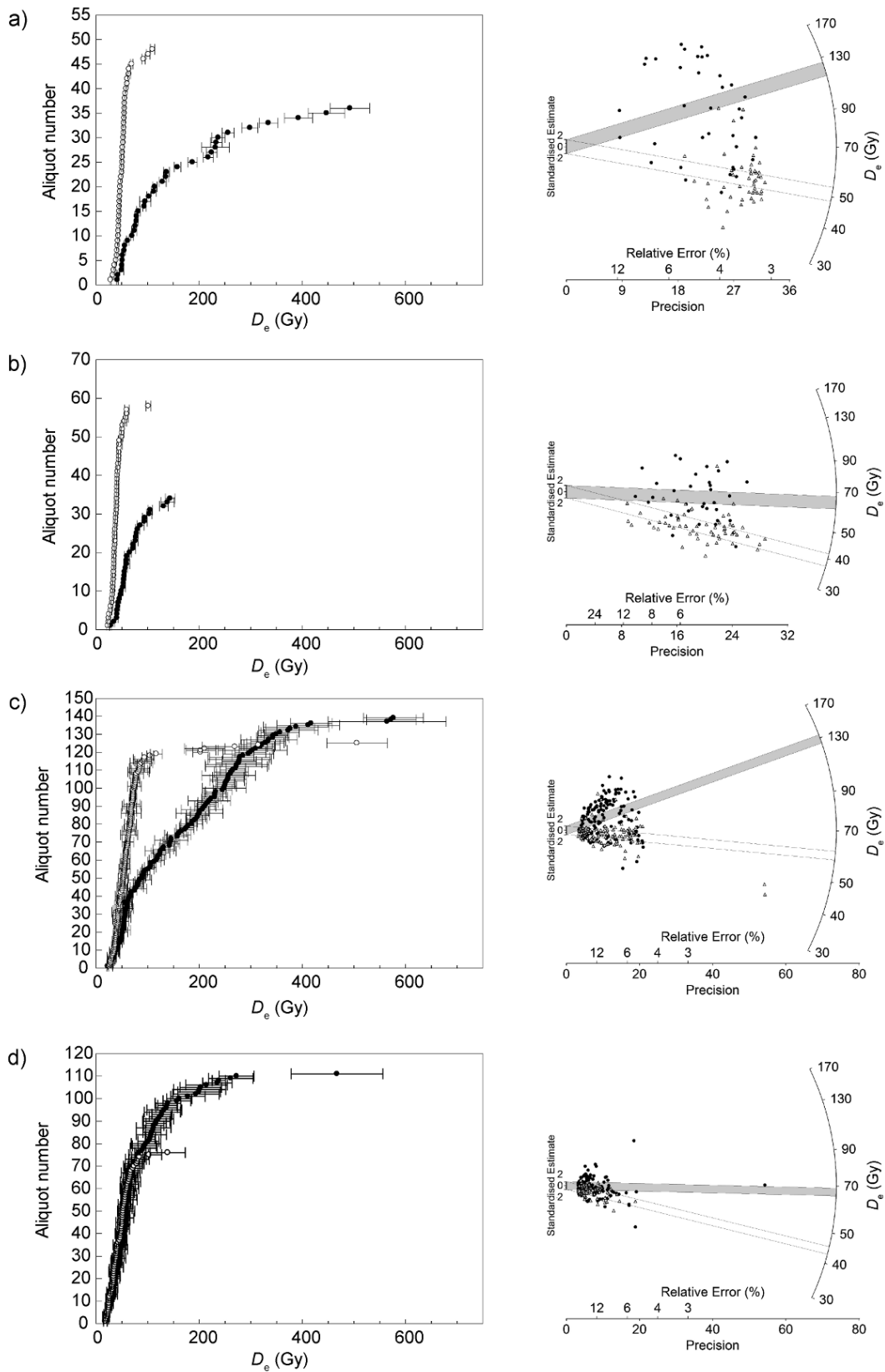


Fig. 8. D_e distributions of selected measurement approaches. Left: cumulative distribution, right: corresponding radial plots; shaded area: CAM of sample GE 2, open area: CAM of sample GE 3. Closed symbols: sample GE 2, open symbols: sample GE 3. a) 1 mm feldspar IR₅₀, b) 2 mm quartz OSL, c) single grain feldspar IR₅₀, d) single grain quartz OSL.

approaches displayed. Differences in internal dosimetry in the feldspar are excluded as a cause for the spread in D_e , as no correlation between potassium content and D_e was observed. High overdispersion values of 0.39 to 0.81 are calculated; this is interpreted to be due to partial bleaching. Support for this hypothesis comes from the test presented by Murray *et al.* (2012), which we apply here in a simplified way. For quartz of sample GE 3 we calculated a mean (CAM) palaeodose of 41.7 ± 2.4 Gy (6 mm aliquots). Due to the higher dose rate, a palaeodose of 69 Gy is expected for feldspar IR₅₀, but this value has to be multiplied by 0.76 to correct for the observed fading. The fading corrected expected IR₅₀ palaeodose of 52 Gy agrees very well with the measured value of 54.1 ± 2.3 Gy. This consistency indicates a well bleached sample. For sample GE 2 with a quartz palaeodose of 57.3 ± 5.4 Gy, an expected fading corrected IR₅₀ palaeodose is 72 Gy. Here, the measured IR₅₀ dose of 220.8 ± 13.4 Gy far exceeds the expected dose, which strongly implies that partial bleaching is present in this sample. Nevertheless, the D_e distributions of GE 2 show a clustering of D_e values at the lower end of the distribution ('leading edge'; Lepper *et al.*, 2000), in the same dose range as the distributions of sample GE 3. This leads to the conclusion that a substantial number of grains were completely reset prior to deposition, while other grains were bleached to lesser degrees.

Performance of the pIRIR₂₂₅ protocol

Dose recovery tests on multiple grain aliquots of both samples using the pIRIR₂₂₅ approach overestimate the given dose; GE 2 overestimates by 16%, GE 3 by 42%. Failing these tests recommends caution when interpreting the results of these measurements. However, Buylaert *et al.* (2012) observed no correlation between the performance of dose recovery tests and accuracy of the age when compared to independent age control. Interestingly, single grain measurements on sample GE 2 recovered the given dose within 4%. Mean residuals of 8.5 ± 1.2 Gy (GE 3, single aliquots), 15.7 ± 1.6 Gy (GE 2, single aliquots) and 14.8 ± 0.1 Gy (GE 2, single grains) on the burial dose have been measured, and the subtraction of residuals from the palaeodose has been recommended (e.g. Thiel *et al.*, 2011). In contrast, Sohbaty *et al.* (2012) discuss this issue and decide not to subtract residuals for their alluvial samples from SE Spain as these were so low compared to the natural doses. For our samples, subtraction of mean residuals for multi-grain aliquot dose recovery tests still does not prevent them from overestimating, while such a subtraction would have resulted in the single grain dose recovery tests significantly underestimating the given dose.

Minimum Age Model

When applying the MAM of Galbraith *et al.* (1999) to extract the palaeodose of partially bleached sediments,

the expected overdispersion, referred to as σ_b , has to be determined. The best way to obtain an appropriate σ_b is to compare it to the overdispersion of a well bleached sample of a similar setting, ideally of the same source, mineral and age (Galbraith and Roberts, 2012), as it has been applied, for example, by Anderson *et al.* (2006). We are in the favourable situation of having an almost completely reset equivalent (sample GE 3) to the poorly bleached sample GE 2. To assess appropriate σ_b values it has to be ensured that aliquots with partially bleached signals are not included in the overdispersion calculations. Therefore, we exclude some values at the upper end of the D_e distributions of sample GE 3 that are significantly larger than the rest of the distribution, assuming they represent incompletely reset signals. From the 1mm aliquots measured with IR₅₀, three values are excluded, and an overdispersion of 19% is calculated. The 2 mm OSL aliquot distribution has one outlier (Fig. 8b) and the remaining distribution has an overdispersion of 19%. Removing the six highest D_e values from the IR₅₀ single grains (Fig. 8c) results in an overdispersion of 27% and, excluding two values with a typically large uncertainties and large doses from the OSL single grains (Fig. 8d), yields a remaining overdispersion of 32%.

The differences in overdispersion for small aliquots and single grains are in the same order as predicted by Cunningham *et al.* (2011). Applying the MAM to the 6 mm aliquots was not considered advisable as, for aliquots with 300 grains, completely bleached grains are masked by the luminescence from grains carrying a large inherited dose and so, only an average signal is measured (Olley *et al.*, 1999; Wallinga, 2002b; Duller, 2008). To also test the MAM on pIRIR₂₂₅ data, σ_b values assessed for the corresponding IR₅₀ approaches are used. When applying the MAM we use the complete datasets, including all values we have identified as partially bleached.

Age comparison

Fig. 9 and Table 2 present the ages obtained by the different approaches. For the well-bleached sample GE3, for small aliquots we obtained a quartz OSL age of 29.1 ± 1.9 ka (2 mm) and a fading corrected IR₅₀ feldspar age (1mm) of 29.6 ± 2.3 ka when using CAM. The MAM ages are 26.1 ± 3.4 ka (OSL) and 26.1 ± 3.9 ka (fading corrected IR₅₀), respectively. The 6 mm aliquot ages are slightly (insignificantly) higher (OSL: 30.6 ± 2.5 ka, fading corrected IR₅₀ 31.7 ± 2.4 ka). Quartz single grains yield ages of 32.4 ± 2.4 ka (CAM) and 28.9 ± 4.7 ka (MAM). Fading corrected ages of feldspar IR₅₀ single grains of 34.5 ± 2.8 ka (CAM) and 27.7 ± 6.8 ka (MAM) are very similar to OSL single grains. pIRIR₂₂₅ ages from small aliquots (CAM: 28.3 ± 2.1 and MAM: 27.5 ± 3.0 ka) are also in agreement with the OSL and IR₅₀ ages and fit the expected age range. However, these ages are uncorrected and it should be remembered that the pIRIR₂₂₅ signal has been observed to fade, although usually less than the IR₅₀ signal (Buylaert *et al.*, 2009).

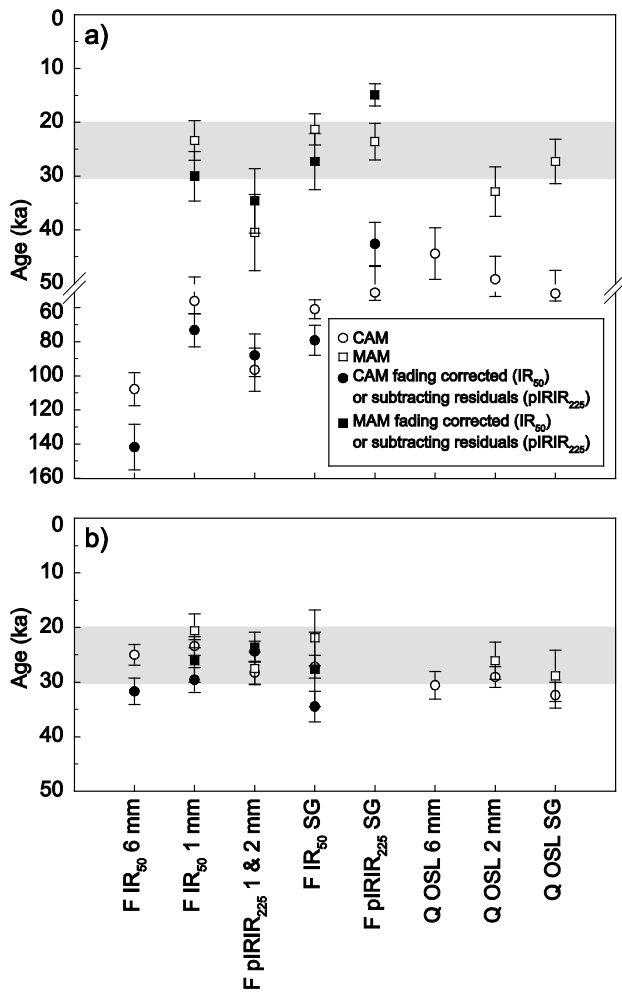


Fig. 9. Comparison of ages assessed by different approaches. a) sample GE 2, b) sample GE 3. Shaded area marks the expected age. Note break and change of scaling in the vertical axis in plot a).

It is noteworthy that MAM and CAM ages (OSL, IR_{50} and $pIRIR_{225}$) for this well bleached sample are consistent within uncertainties, with the central value of MAM ages being only slightly younger than CAM ages. This indicates that applying the MAM to a well bleached sample with an appropriate σ_b value does not necessarily lead to inaccurate ages. This is in contrast to the observations of Murray *et al.* (2012), who found a significant underestimation of MAM ages compared to CAM ages on well bleached samples.

The ages for sample GE 2 differ significantly (Fig. 9a). A severe overestimation occurs with 6 mm aliquots where the fading corrected feldspar IR_{50} (CAM: 141.8 ± 13.3 ka) predates the expected age of the sample (20–30 ka) by more than 100 ka and gives an age beyond the Last Interglacial. It should be noted that the palaeodose corrected here is beyond the range for which the applied fading correction is designed. The uncorrected age, which is usually (in well bleached samples) expected

to underestimate, yields 107.8 ± 9.7 ka. The CAM 6 mm quartz OSL age of 44.4 ± 4.8 ka is also significantly overestimating. With small aliquots the CAM ages (IR_{50} fading corrected: 73.2 ± 9.8 ka, $pIRIR_{225}$: 51.3 ± 4.5 ka, OSL: 49.4 ± 4.3 ka) all significantly overestimate as well. The same observation is made for the CAM on single grains, where IR_{50} (fading corrected) yields 79.2 ± 8.8 ka, $pIRIR_{225}$ 42.6 ± 4.0 ka and quartz OSL 51.8 ± 4.3 ka. The observed age overestimations for sample GE 2 confirm the presence of partial bleaching, as already deduced from the large spread in the D_e distributions and the test for well bleached samples following Murray *et al.* (2012). This is confirmed by the application of the MAM to the small aliquots of quartz (32.9 ± 4.6 ka) and the fading corrected IR_{50} (30.0 ± 4.6 ka), which do fit the expected age. However, the MAM $pIRIR_{225}$ age (40.5 ± 7.1 ka) clearly overestimates the expected age. All age overestimations for GE 2 are interpreted as due to partial bleaching and recommend the measurement of small aliquots as a minimum requirement, but preferably single grain analysis. Applying the MAM on feldspar IR_{50} single grains returns an age of 27.3 ± 5.2 ka (fading corrected), 27.3 ± 4.1 ka for quartz OSL, and 23.6 ± 3.4 ka for $pIRIR_{225}$ single grains. In contrast to Blomdin *et al.* (2012) (who used large aliquots for a test), we observe a bleached population in our $pIRIR$ data when using single grains. However, the data are not fully comparable as Blomdin *et al.* (2012) used the $pIRIR_{290}$ protocol while here the $pIRIR_{225}$ signal was used.

The MAM age obtained for the small aliquots of the well bleached sample GE 3 using the $pIRIR_{225}$ protocol agrees well with the age obtained from quartz OSL on small aliquots, when the residual dose is not subtracted. A subtraction of the residual is not necessarily indicated, as shown by the MAM $pIRIR_{225}$ single grain age of the partially bleached sample GE 2. A clear underestimation (14.9 ± 2.1 ka) of the expected age range occurs when the residual is subtracted, but agreement within uncertainties with the other single grain data is reached when the residual is not subtracted. Only the $pIRIR_{225}$ age from small aliquots of sample GE 2 would be improved with regard to the expected age range when the residual is subtracted, but it appears likely that partial bleaching is the reason for the overestimation. The methodological difficulties call for caution with regard to the reliability of $pIRIR_{225}$, despite the fact that they are consistent with age control.

5. CONCLUSION

We have shown data for two glaciofluvial samples from the same outcrop with only a few meters distance between the sampling spots and from the same sedimentological context. Despite this, the samples show clearly different levels of luminescence signal resetting, which require special attention when dating them. One sample is found to have a nearly complete resetting of the signal. This sample passes the test for well bleached quartz pre-

sented by Murray *et al.* (2012), has low overdispersion values, and a non-skewed distribution. The other sample yields broad and skewed distributions, has much higher overdispersion values, and fails the test by Murray *et al.* (2012). It is therefore interpreted to be partially bleached prior to deposition. However, using small aliquots or single grains together with the MAM resulted in ages within the expected age range. As would be expected, using large aliquots led to inaccurate ages. Our results support the findings of Duller (2008) that single grains are preferred in environments where partial bleaching is likely. We have shown a significant age overestimation using the CAM for the partially bleached sample GE 2, and application of the MAM is a necessity. In both samples the IR₅₀ ages are in better agreement with the quartz OSL ages when corrected for fading. Our findings imply that fading corrections are necessary, and produce accurate results, for the moderate fading rates observed in this study and palaeodose values within the linear part of the dose response curve.

The advantage of feldspar single grain dating, at least for the samples investigated here, is that a large number of D_e values can be produced with relatively little effort, and therefore provides a more solid basis for the statistical analyses. This is due to the fact that a relatively large proportion of feldspar grains (>17%) deliver suitable IR₅₀ signals.

Despite methodological problems which call for caution, the applied pIRIR₂₂₅ protocol yields ages consistent with other approaches.

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