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Testing optically stimulated luminescence dating of sand-sized quartz and feldspar from fluvial deposits

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Abstract

We apply single-aliquot optically stimulated luminescence (OSL) dating to quartz- and feldspar-rich extracts from fluvial channel deposits of the Rhine–Meuse system in The Netherlands. The time of deposition of these deposits is tightly constrained by radiocarbon dating or historical sources. This allows us to compare OSL ages obtained on quartz and infrared OSL (IR-OSL) ages obtained on potassium-rich feldspar with independent ages over the range of 0.3–13 ka. We show that the quartz OSL ages are in good agreement with the expected age. Using IR-OSL dating of feldspar, we find a slight age overestimate for the youngest sample, whereas for older samples the age is significantly underestimated. We also apply OSL dating to older fluvial and estuarine channel deposits with limited independent chronological constraints. Comparison of feldspar IR-OSL ages with the quartz OSL ages up to ~200 ka shows a clear trend, where the former severely underestimates the latter. This trend is similar to that found for the samples with independent age control, indicating that the feldspar IR-OSL ages are erroneously young for the entire age range. In the youngest samples, incomplete resetting of the IR-OSL signal prior to deposition probably masks the age underestimation. We show that the IR-OSL age underestimation is partly caused by changes in trapping probability due to preheating. Correction for this phenomenon improves the IR-OSL ages slightly, but does not provide a complete solution to the discrepancy. We suggest that, in the light of the problems encountered in the IR-OSL dating of feldspar, quartz is the mineral of choice for OSL dating of these deposits. However, feldspar dating should continue to be investigated, because it has potential application to longer time scales.

Keywords: optically stimulated luminescence dating; quartz; feldspar group; stream sediments; age

1. Introduction

Optically stimulated luminescence (OSL) dating is a rapidly developing technique that provides absolute chronologies for late Quaternary clastic

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sedimentary records [1]. OSL dating of the sandy fraction can be applied to quartz up to an age of 100–200 ka, or to feldspar, which offers the potential to extend the age range to 1 Ma. Fluvial records now represent one of the most common sedimentary environments dated by OSL methods. However, a potential problem in fluvial systems is that light exposure of sand during transport may not have been sufficient to completely reset the OSL signal of all grains prior to deposition. Incomplete resetting of the OSL signal will lead to an overestimation of the age. Most studies applying OSL dating to fluvial deposits have focused on chronologically unconstrained, or poorly constrained, Pleistocene successions. With the exception of a few studies (notably [2–5]), little effort has been made to provide a sound justification for the use of luminescence dating in fluvial settings.

Rigorous comparison of OSL ages with independent ages is only possible if the latter are highly accurate and if the stratigraphic relationship between OSL samples and independent ages is indisputable. Studies must also provide enough background information to assess the quality of luminescence and independent ages. For coarse-grain quartz OSL dating, several comparative studies that meet these guidelines have been conducted in aeolian environments [6,7]. For coarse-grain feldspar infrared OSL (IR-OSL) dating, on the other hand, validation of the technique is surprisingly limited; few studies fit the criteria for rigorous comparison outlined above. Good agreement with independent age control was found for samples younger than 30 ka for aeolian dune sands from New Zealand [8]. On the other hand, IR-OSL ages on samples from the same type of deposit from Germany showed an underestimation of age [9].

In the present study, we apply OSL dating to both quartz and feldspar extracts from sandy channel deposits from the Rhine–Meuse system in The Netherlands. We sampled four channel belts for which the period of activity is accurately known from historical sources or radiocarbon dating. Comparison of OSL dating results from these samples with unusually tight independent age constraints presents an unparalleled opportunity to test the accuracy of OSL dating results from submodern (0.3 ka) to Late Weichselian ($\sim$13 ka) fluvial sediments. Additionally, OSL dating of samples from chronologically unconstrained fluvial and estuarine channel deposits from the same area allows comparison of quartz and feldspar OSL ages up to an age of $\sim$200 ka.

2. Study area and independent age constraints

The Rhine–Meuse Delta (Figs. 1 and 2) is located in the southeastern part of the North Sea Basin. The Holocene delta is underlain by Weichselian (oxygen-isotope stages (OIS) 2–4), sandy to gravelly fluvial channel deposits. The Holocene Rhine–Meuse Delta was formed in response to relative sea-level rise [10,11] and contains wide-

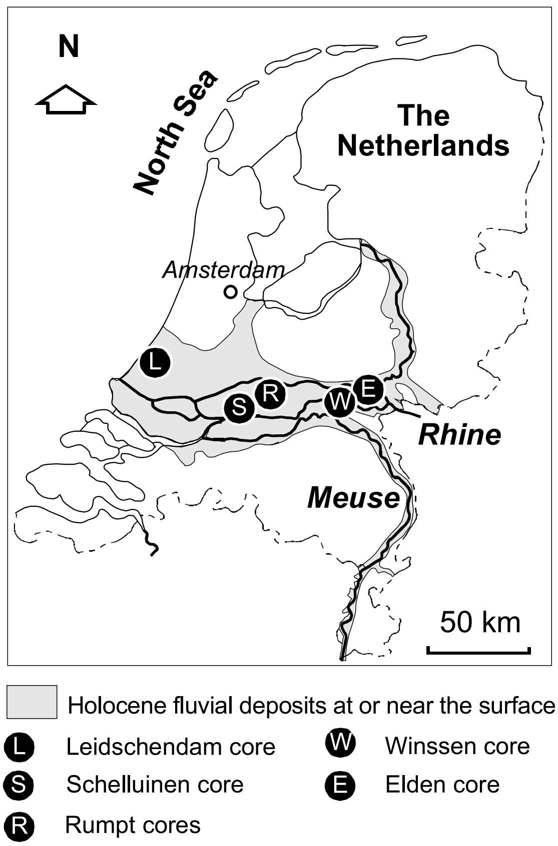

Fig. 1. The Rhine–Meuse Delta in The Netherlands and location of sampling sites.

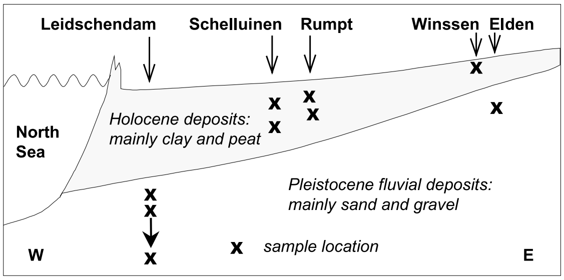

Fig. 2. Schematic east–west cross section showing relative positioning of the samples. The Winssen, Rumpt and Schelluinen samples were taken from sandy fluvial channel deposits within the Holocene Rhine–Meuse Delta. The Elden and Leidschendam samples were taken from Pleistocene, predominantly fluvial channel deposits underlying the Holocene delta. Sample location (local coordinates) and depth (meters below the surface): Winssen (168.485/435.475, −2.75 m), Rumpt I-3 (139.355/433.880, −4.35 m), Rumpt IV-2 (139.920/433.330, −2.95 m), Schelluinen II-3 (123.290/429.875, −3.15 m), Schelluinen II-6 (123.290/429.875, −5.55 m), Elden (187.985/440.000, −6.25 m), Leidschendam (87.540/454.380, for sample depths see [20]).

spread in situ organics interbedded with fluvial clastics, thus enabling highly accurate ^{14}C chronologies to be established. We collected OSL samples from three Holocene channel belts (Waal, Linge, and Schaik systems) and one Late Weichselian channel belt (Elden core), and additionally from Weichselian to Saalian (OIS 2 and older), fluvial and estuarine channel deposits (Leidschendam core) (Figs. 1 and 2).

According to historical maps, the deposits at the sampling site of the Waal (Winssen core) were formed between AD 1688 and 1723 [12], which corresponds to an age of ∼300 yr (note that we use AD 2000 as a reference for calendar ages). The Winssen sample was collected to assess the potential effects of poor bleaching. We selected a 300-yr-old sample rather than a modern one to avoid the influence of locks and waterworks that have altered sediment transport in the present-day river, and thus the bleaching environment.

The ages of the other two Holocene channel belts are well constrained by ^{14}C dating (Table 1) with periods of activity no longer than 1700 yr (Linge system) and 800 yr (Schaik system). Radiocarbon dating [13] employed accelerator mass spectrometry (AMS) of terrestrial botanical macrofossils [14]. For both the Linge and the Schaik systems, a larger number of ^{14}C ages are available; we used those that are located closest to the sites of our OSL samples. The beginning of activity of both channel belts was determined by ^{14}C dating the top of peat directly underlying

Table 1
Independent age constraints of OSL-dated fluvial channel deposits

Fluvial system	OSL samples	^{14}C age (yr BP)		Calendar age[b] (yr before AD 2000) (median and 2σ confidence interval)	
		Beginning	End	Beginning	End
Waal	Winssen			312[c]	277[c]
Linge	Rumpt I-3, Rumpt IV-2	2 235 ± 35[a1]		2 300[d1] (2 190–2 370)	∼700[e]
Schaik	Schelluinen II-3, Schelluinen II-6	5 050 ± 85[a2]	4 605 ± 45[a3]	5 850[d1] (5 670–6 030)	5 360[d2] (5 210–5 470)
Late Weichselian	Elden	11 063 ± 12[a4]		13 155[f] (12 990–13 320)	

[a] Laboratory numbers: (1) UtC-1481/1482; (2) UtC-1144/1300; (3)UtC-1128/1129/1130/1141/1142 (all based on AMS ^{14}C measurements of terrestrial plant macrofossils, except for samples UtC-1128, 1141 and 1481 [13]); (4) Hd-19607, Hd-18648, Hd-19098, Hd-19092, Hd-18622, Hd-19037, Hd-18438 (AMS ^{14}C measurements of decadal samples of poplar buried by the Laacher See Tephra [16]).
[b] As derived from historical sources or calibrated ^{14}C ages. For the latter 50 yr was added to each age (initially determined in yr BP = AD 1950) to enable direct comparison with OSL ages.
[c] Obtained from historical maps [12].
[d] Calibration of ^{14}C ages according to the Groningen CAL25 program [18] using smoothed curves [19]; smoothing parameters used: (1) 100; (2) 200.
[e] Historical age of damming of the river at its upstream bifurcation [15].
[f] Following calibration by Friedrich et al. [16], including a systematic uncertainty of 70 yr.

overbank deposits (sampling strategy is extensively discussed by Törnqvist and Van Dijk [13]). The end of activity of the Linge system was a result of artificial damming at its upstream bifurcation around AD 1300 [15]. The end of activity of the Schaik system was derived from the age of the base of peat overlying overbank deposits and basal peat in the residual channel [13].

We also sampled channel deposits of Late Weichselian age, which underlie the Holocene deltaic deposits at Elden (Figs. 1 and 2). The fluvial sands at this location are extremely rich in pumice that originated from the Laacher See volcanic eruption in the Eifel (Germany), ^{14}C dated at $11\,063 \pm 12$ yr BP [16]. Pumice from the Laacher See eruption blocked the River Rhine for several days [17], and was transported downstream when the dam collapsed. The pumice has been found throughout Late Weichselian fluvial deposits in The Netherlands [15], and the high concentration (up to 25% by volume) at our sampling site makes it highly likely that these sands were deposited shortly after the volcanic eruption.

In addition to the sites with independent age control (summarized in Table 1), samples were taken from a core retrieved from Saalian (OIS 6–8) to Weichselian (OIS 2–4) fluvial and estuarine channel deposits near Leidschendam [20] (Figs. 1 and 2). OSL dating of the quartz and feldspar separates from this core allows us to compare the results obtained on both mineral fractions up to an age of $\sim$200 ka.

3. Methods

Most samples were taken using a simple hand-operated suction corer, enabling us to obtain 30-cm-long samples in opaque PVC tubes [21]. The Leidschendam core was obtained using a mechanized bailer drilling unit [22] of the Netherlands Institute of Applied Geoscience TNO, yielding an undisturbed core with 10 cm diameter. The cores were opened in subdued red light, after which samples for OSL dating were taken. All samples were water-washed and treated with 10% HCl and 30% H_2O_2 to remove carbonates and organic material. After drying, the samples were sieved and subsequently density-separated using an aqueous solution of sodium polytungstate to extract the potassium-rich feldspar fraction lighter than 2.58 g/cm^3. The denser fraction was treated with concentrated hydrofluoric acid for 40 min to obtain a clean quartz sample and to etch away the outer 10 μm of the quartz grains. No hydrofluoric acid etching was used for the feldspar.

Measurements were made on an automated Risø TL/OSL reader, using an internal $^{90}Sr/^{90}Y$ β-source [23]. The sample grains were mounted on aluminum or stainless steel discs using silicone spray. Blue light emitting diodes were used for stimulation of quartz (at 125°C) and the resulting luminescence signal was detected through 9 mm of Schott U-340 filters (detection window 250–390 nm). The single-aliquot regenerative dose (SAR) protocol [24] was used for estimation of the equivalent dose. A relatively low preheat (200°C for 10 s) was used to avoid thermal transfer effects [1,25] that were shown to affect the equivalent dose of the Winssen sample when more stringent preheats were used (Fig. 3a). For the Leidschendam samples a more usual 10 s preheat at 260°C was used because here thermal effects are expected to be negligible. This was confirmed by the preheat plateau obtained for sample Leidschendam I (Fig. 3b). The test-dose response was measured after heating to 160°C for all samples.

For the feldspar separates, we used Schott BG-39 and Corning 7-59 filters, giving a transmission window between 320 and 480 nm. The single-aliquot additive dose (SAAD) procedure [26,27], with a 10 min preheat at 220°C, was used for estimation of the equivalent dose. Optical stimulation was provided by infrared diodes (emitting round 880 nm). The equivalent dose was also estimated using the SAR protocol for feldspar [28], using a 10 s preheat at 290°C for natural and regenerative doses, and heating to 210°C for the test doses. An infrared laser diode (emitting at 830 nm) was used for stimulation.

The natural dose rate was estimated in the laboratory using high-resolution γ-spectrometry [29] on bulk samples (results in Table 2), that were taken from around the sample used for equivalent-dose determination. It is fair to assume that the samples have been saturated with water

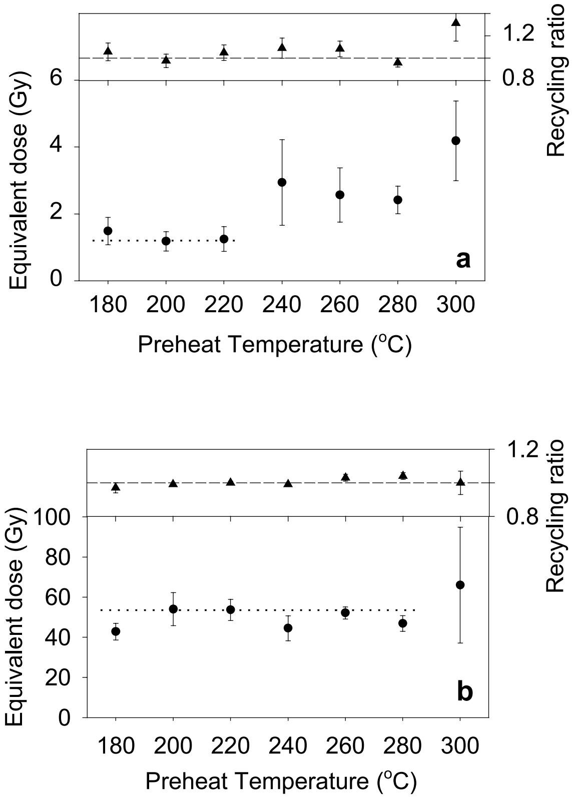

Fig. 3. Equivalent doses obtained for a range of preheat temperatures (each held for 10 s) for aliquots of quartz separate from the Winssen (a) and Leidschendam I (b) samples. Each estimate is the average from three aliquots, and error bars indicate the standard error on the mean. The equivalent dose (circles), and the recycling ratio (triangles) [24] are both shown. The equivalent-dose estimate used for age determination (Table 2; Winssen preheat 200°C, Leidschendam I preheat 260°C) is indicated by the dotted line.

throughout their lifetimes, which diminishes the dose rate [38].

4. OSL dating results and discussion

4.1. Quartz OSL

The quartz OSL dating results and independent ages are presented in Table 2 and Fig. 4. For the youngest sample (Winssen), the OSL age slightly overestimates the known historical age of the sample. For the other samples with independent age control all OSL ages are in excellent agreement with the radiocarbon-dated periods of activity. The slight offset (600 yr) found for the Winssen sample is likely caused by incomplete bleaching, and is comparable to that found for modern channel deposits in other parts of the world [2,4,5]. We observed that application of higher preheat temperatures (e.g. 260°C) resulted in a greater offset for the Winssen sample (Fig. 3a), and also in a slight overestimation of age for the other samples with independent age control (not shown). These results are in accordance with those of Rhodes [25] and indicate that stringent preheats should be avoided when dating young samples that might not have been thoroughly exposed to light prior to deposition.

In addition to our test of the validity of quartz OSL dating, we applied OSL dating to older deposits with limited age constraints to allow comparison of quartz OSL and feldspar IR-OSL dating results for a wider age range. The geological context and the quartz OSL dating results on these samples (Leidschendam core) are discussed by Törnqvist et al. [20]; a brief summary is given here. The OSL data suggest that the base of the succession (samples VIII–X; Table 2) was deposited during the Saalian glaciation (OIS 6). The flattening of the OSL dose–response curve at the relatively high doses these samples have absorbed amplifies the scatter in the OSL measurements, and this is reflected in the relatively large uncertainties in the age estimates. The OSL age of sample VII suggests an Eemian or Early Weichselian (OIS 5) origin, which is supported by the first occurrence of transgressive marine shell remains at this level [20].

OSL ages for the upper part of the succession (samples I–VI) point to deposition around OIS 4, i.e. during a period with relatively low sea level. However, mud drapes containing warm pollen and estuarine diatoms were encountered near the base of this unit (sample VI, see also [20]), suggesting that these sediments must have been deposited during a period of relatively high sea level. Hence, we cannot preclude the possibility that these deposits were formed during the preceding sea-level highstand, i.e. OIS 5a ($\sim$80 ka), and that they are possibly older than the quartz OSL ages suggest. The slight age reversal for samples

Table 2
Quartz OSL dating results

Sample	Grain size (μm)	Radionuclide concentration[a] (Bq/kg)				Dose rate[a,b] (Gy/ka)	Equivalent dose (Gy)	OSL age (ka)	Independent age (ka)
		^{238}U	^{226}Ra	^{232}Th	^{40}K				
Winssen	180–212	12 ± 2	10.8 ± 0.2	10.9 ± 0.2	362 ± 7	1.27 ± 0.05	1.17 ± 0.12[c]	0.92 ± 0.10	0.3
Rumpt I-3	180–212	15 ± 2	10.1 ± 0.4	12.3 ± 0.4	404 ± 13	1.36 ± 0.05[d]	1.67 ± 0.13[d]	1.23 ± 0.10[d]	0.7–2.4
Rumpt IV-2	180–212	35 ± 5	31.7 ± 0.5	33.0 ± 0.5	482 ± 10	2.10 ± 0.14	3.7 ± 0.2	1.75 ± 0.10	0.7–2.4
Schelluinen II-3	180–212	12 ± 3	9.9 ± 0.3	10.7 ± 0.3	398 ± 8	1.33 ± 0.05	6.9 ± 0.5	5.1 ± 0.4	5.2–6.0
Schelluinen II-6	180–212	11 ± 2	9.3 ± 0.2	9.9 ± 0.2	363 ± 7	1.22 ± 0.09[d]	7.5 ± 0.2[d]	6.1 ± 0.5[d]	5.2–6.0
Elden	180–212	13 ± 2	14.3 ± 0.4	16.4 ± 0.4	415 ± 12	1.51 ± 0.05	20.0 ± 1.0	13.3 ± 0.8	13.0–13.3
Leidschendam I	180–212	10 ± 3	9.5 ± 0.5	9.6 ± 0.4	297 ± 13	1.11 ± 0.06	54 ± 3	48 ± 4	–
Leidschendam II	180–212	6 ± 3	9.5 ± 0.3	10.8 ± 0.3	249 ± 7	0.99 ± 0.05	54 ± 5	55 ± 6	–
Leidschendam III	180–212	6 ± 2	9.5 ± 0.4	7.5 ± 0.3	200 ± 10	0.78 ± 0.05	64 ± 6	82 ± 9	–
Leidschendam IV	180–212	12 ± 3	12.8 ± 0.3	14.1 ± 0.3	341 ± 7	1.29 ± 0.06	92 ± 7	71 ± 6	–
Leidschendam V	180–212	6 ± 3	6.9 ± 0.5	8.2 ± 0.4	253 ± 13	0.92 ± 0.05	56 ± 3	61 ± 5	–
Leidschendam VI	180–212	8 ± 3	9.8 ± 0.2	10.7 ± 0.2	314 ± 7	1.13 ± 0.05	66 ± 3	58 ± 4	–
Leidschendam VII	180–250	12 ± 3	6.8 ± 0.3	6.7 ± 0.2	237 ± 6	0.86 ± 0.04	104 ± 6	120 ± 9	–
Leidschendam VIII	180–250	10 ± 2	6.5 ± 0.4	8.2 ± 0.4	201 ± 10	0.79 ± 0.05	124 ± 7	158 ± 13	–
Leidschendam IX	180–212	11 ± 2	5.5 ± 0.3	7.4 ± 0.2	190 ± 7	0.74 ± 0.04	107 ± 10	145 ± 16	–
Leidschendam X	180–212	7 ± 3	6.9 ± 0.3	8.2 ± 0.2	240 ± 6	0.87 ± 0.04	156 ± 23	180 ± 28	–

[a] Spectral data from high-resolution γ-spectroscopy converted to activity concentrations and infinite matrix dose rates using the conversion data given by Olley et al. [30].
[b] The natural dose rate was calculated from the infinite matrix dose rate using attenuation factors given by Mejdahl [31], and includes a contribution from cosmic rays [32]. A contribution from internal α dose was calculated based on U and Th contents reported by Mejdahl [33], using an a-value of 0.04 ± 0.01 [34], which resulted in an internal dose rate of 0.028 ± 0.013 Gy/ka. All dose rates calculated for a water content of 20 ± 2% (based on a porosity of 34 ± 3% [35]) using attenuation factors given by Zimmerman [36].
[c] Four outliers ($D_e > 6$ Gy) from a total of 34 aliquots were not incorporated.
[d] Values differ slightly from those reported by Wallinga and Duller [37] due to a small shift in the source calibration, and an improved water content estimation.

Leidschendam III–VI might also indicate that the OSL ages obtained on samples V and VI (∼60 ka) could be too young. It is interesting to note that slight age underestimation was also reported for quartz OSL ages of Eemian (OIS 5e) deposits from Denmark [39].

This study for the first time rigorously compares OSL ages and independent age control for fluvial deposits over a relatively wide age range (up to 13 ka), and the results underline the validity of quartz OSL dating for establishing absolute chronologies for fluvial deposits. However, the controversy over the age of the Weichselian deposits in the Leidschendam core stresses the need for comparisons of OSL ages with independent age control for pre-Holocene deposits. Future research should focus on such comparisons to further increase the confidence in quartz OSL ages.

4.2. Feldspar IR-OSL

The feldspar IR-OSL dating results obtained by the SAAD protocol [26,27] are presented in Table 3 and Fig. 4. For the Winssen sample the IR-OSL age overestimates the independent age and is also slightly higher than that obtained by OSL dating of the quartz separate. For the Rumpt samples IR-OSL ages are found in agreement with the independent age range and with the quartz OSL dating results. However, for the Schelluinen and Elden samples an IR-OSL age underestimation is found, both compared to independent ages and compared to the quartz OSL ages. An increasing deviation with increasing age (Fig. 4b) suggests that the underestimation is a relative effect, possibly masked in the younger samples by poor bleaching. This underestimation of IR-OSL age

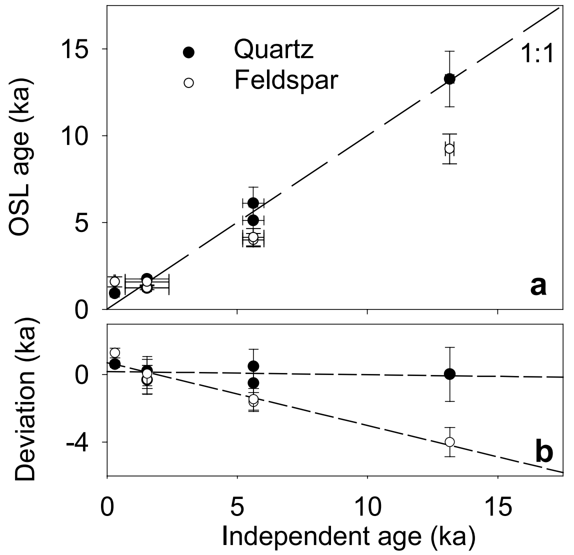

Fig. 4. (a) OSL ages obtained on quartz separates using the SAR protocol [24] with a 10 s 200°C preheat (filled circles), and IR-OSL ages obtained on potassium-rich feldspar separates using the SAAD procedure [26,27] (open circles) plotted against the independent age of the samples. All errors indicate 2σ confidence intervals. (b) Difference between the luminescence ages and the independent age control. Errors include uncertainties in both the independent ages and the OSL ages.

is unlikely to be caused by errors in determination of the external dose rate; this would also affect the quartz ages. It is improbable that the differences between OSL ages on quartz and IR-OSL ages on feldspar are caused by differences in bleaching, as the quartz signal is more readily reset by sunlight [1], and because the quartz ages agree with the independent chronology.

To investigate whether the age underestimation could be caused by sensitivity changes during the measurement procedure, a SAR protocol for feldspar [28] was employed. Using this protocol, ages similar to those obtained using the SAAD procedure were obtained (Table 3, see also [28]). An additional advantage of the SAR procedure is that it is straightforward to test whether preheating removes all unstable trapped charge caused by laboratory irradiation. Using the SAR protocol, we found equivalent doses to be independent of preheat temperature for 10 s preheats above 200°C (Fig. 5).

The IR-OSL age of feldspar separates from the Leidschendam core was measured using the SAR protocol. The average equivalent dose of three to six aliquots of each sample is shown in Table 3. In Fig. 6 the IR-OSL ages obtained on the samples are plotted as a function of the quartz OSL age. All but two samples follow a trend where the feldspar IR-OSL age is only half that of the quartz OSL age from the same sample. For the two samples that do not follow this trend (Leidschendam VII and VIII), an atypically large scatter was observed between equivalent doses obtained on different feldspar aliquots, suggesting that the IR-OSL signal was not completely reset for all grains at the time of deposition [41].

From the results for the samples with independent age control, we deduced that the accuracy of the quartz OSL ages is superior to that of the feldspar IR-OSL ages. Combining the results obtained on the samples with independent age control, and that from the Leidschendam samples, we conclude that the IR-OSL ages obtained on the potassium-rich feldspar separates severely underestimate the age of our samples, but that in some

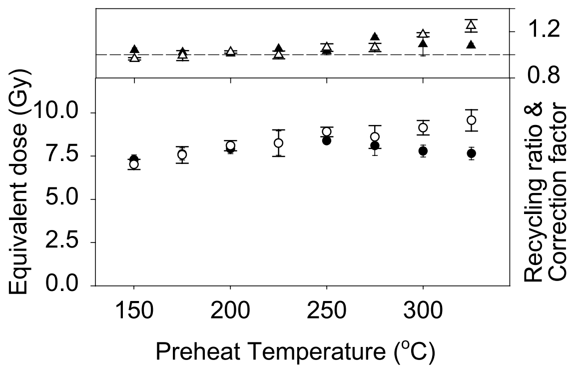

Fig. 5. Equivalent doses obtained for a range of preheat temperatures (each held for 10 s) for aliquots of feldspar separate from sample Schelluinen II-3. The sample was heated to 150°C after the test dose was administered. Each estimate is the average of three aliquots. The equivalent dose obtained by the SAR procedure (filled circles) and the recycling ratio (filled triangles) are indicated, as are the correction factor for different preheat temperatures (open triangles) and the equivalent dose after correction with this factor (open circles). The derivation of the correction procedure is described in the main text.

Table 3
Feldspar IR-OSL dating results

Sample	Internal K[a] (%)	Dose rate[b] (Gy/ka)	Equivalent dose (Gy)		IR-OSL age (ka)		Independent age (ka)
			SAAD[c]	SAR[d]	SAAD[c]	SAR[d]	
Winssen	8.5	1.88 ± 0.08	3.0 ± 0.2	2.9 ± 0.2	1.58 ± 0.14	1.52 ± 0.14	0.3
Rumpt I-3	7.4	1.92 ± 0.08[e]	2.38 ± 0.05[e]	2.8 ± 0.4	1.24 ± 0.05[e]	1.5 ± 0.2	0.7–2.4
Rumpt IV-2	9.5	2.87 ± 0.13	4.53 ± 0.13	4.2 ± 0.3	1.58 ± 0.08	1.47 ± 0.13	0.7–2.4
Schelluinen II-3	7.9	1.93 ± 0.07	7.7 ± 0.2	7.4 ± 0.5	4.0 ± 0.2	3.8 ± 0.3	5.2–6.0
Schelluinen II-6	9.8	1.91 ± 0.11[e]	7.9 ± 0.2[e]	7.6 ± 0.4	4.2 ± 0.3[e]	4.0 ± 0.3	5.2–6.0
Elden	8.0	2.12 ± 0.08	19.6 ± 0.5	16.8 ± 1.2	9.2 ± 0.4	7.9 ± 0.6	13.0–13.3
Leidschendam I	9.2	1.68 ± 0.09	–	46.9 ± 1.8	–	28 ± 2	–
Leidschendam II	8.2	1.51 ± 0.09	–	47.6 ± 1.5	–	32 ± 2	–
Leidschendam III	9.2	1.35 ± 0.08	–	59 ± 4	–	44 ± 4	–
Leidschendam IV	9.1	1.87 ± 0.09	–	71 ± 5	–	38 ± 3	–
Leidschendam V	9.2	1.49 ± 0.08	–	52 ± 3	–	35 ± 3	–
Leidschendam VI	9.0	1.69 ± 0.08	–	65 ± 3	–	38 ± 3	–
Leidschendam VII	8.9	1.47 ± 0.12	–	146 ± 17	–	100 ± 15	–
Leidschendam VIII	8.5	1.36 ± 0.12	–	191 ± 13	–	140 ± 16	–
Leidschendam IX	10.1	1.36 ± 0.08	–	96 ± 6	–	71 ± 6	–
Leidschendam X	9.1	1.43 ± 0.07	–	112 ± 4	–	78 ± 5	–

[a] The potassium content of the potassium-feldspar separates was determined by β-counting in a GM multicounter system [40]. A calibration uncertainty of 5% was assumed.
[b] Calculations similar to quartz, apart from a contribution from internal potassium [33]. An a-value of 0.08 ± 0.02 [34] was used to calculate the contribution from internal α-radiation (0.06 ± 0.03 Gy/ka). As the separates were not HF-etched a small contribution from external α-radiation was also included (typically ~0.02 Gy/ka).
[c] SAAD protocol [26,27] used for equivalent-dose estimation.
[d] SAR protocol [28] used for equivalent-dose estimation.
[e] Values differ slightly from those reported by Wallinga and Duller [37] due to a small shift in the source calibration, and an improved water content estimation.

cases the underestimation is masked by incomplete bleaching. The quartz OSL ages of the samples from the Leidschendam core might slightly underestimate the true age, as was discussed in Section 4.1. If this is the case, the feldspar IR-OSL age underestimation is even more severe than indicated by the comparison of quartz OSL and feldspar IR-OSL results. In the following sections, the causes for the feldspar IR-OSL age underestimation are considered.

5. Possible reasons for age underestimation in feldspar IR-OSL

5.1. Anomalous fading

Anomalous fading is the loss of electrons from traps on a time scale that is short compared with the lifetime predicted on the basis of their trap depth. This phenomenon is only known to affect feldspars and gives rise to an age underestimation [42–44]. We carried out fading tests in three different ways.

Firstly, the decay in IR-OSL was monitored following laboratory irradiation of previously unmeasured natural samples. Doses similar to the natural dose of the sample were used for this experiment. After irradiation, the samples were preheated to 220°C for 10 min and measurements were made using short exposure to infrared light. To correct for the decay due to this measurement, the same measurements were made on natural samples that did not receive a laboratory dose but were otherwise treated identically. The fading ratio is given by the ratio of irradiated to natural

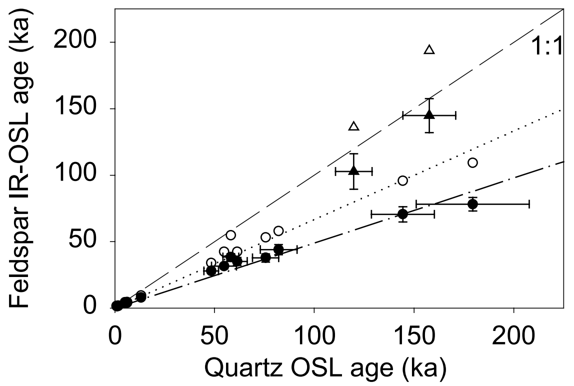

Fig. 6. Feldspar IR-OSL ages as a function of the quartz OSL ages of the same samples. The SAR procedure was used for equivalent-dose determination in both (see Tables 2 and 3 for sample names, equivalent doses and ages). Nearly all samples follow a trend where the feldspar IR-OSL age is only half the quartz OSL age (filled circles, dash–dot trend line). The two samples that are indicated by triangles show unusually wide scatter in the IR-OSL equivalent-dose determinations and were not incorporated in the regression. Also shown are the feldspar IR-OSL ages after application of the correction factor as determined for each sample (open symbols, dotted trend line; Table 5).

IR-OSL response before storage, divided by the same ratio after storage at ambient temperature for 6 months.

For the second and third tests, fading was checked using the SAR procedure [28]. We compared the sensitivity-corrected OSL signal after irradiation (~50 Gy) and storage with that measured immediately after irradiation (time between irradiation and measurement was a maximum of 4 h). These fading tests were performed on aliquots that had previously been used for equivalent-dose determination using the feldspar SAR procedure [28]. The samples were preheated to 290°C for 10 s prior to measurement of the IR-OSL signal. Samples were stored either for 4 months at ambient temperature (second test), or for 10 days at 100°C (third test). Due to a lack of material, it was not possible to apply all three methods to each sample.

Some fading was detected in the Elden sample using the third test (Table 4); unfortunately there was not enough material to check this fading with the other two methods. The fading in this sample might be associated with the recent volcanic origin of some of the feldspar, as the material was deposited shortly after the Laacher See volcanic

Table 4
IR-OSL fading tests on the feldspar separates

Sample	Fading ratio		
	Conventional 6 months ambient	SAR 4 months ambient	SAR 10 days 100°C
Winssen	–	–	1.00 ± 0.05
Rumpt I-3	0.95 ± 0.04	–	1.003 ± 0.013
Rumpt IV-2	1.01 ± 0.08	–	–
Schelluinen II-3	0.96 ± 0.02	–	1.06 ± 0.02
Schelluinen II-6	0.87 ± 0.03	–	1.00 ± 0.02
Elden	–	–	0.907 ± 0.010
Leidschendam I	–	1.003 ± 0.005	0.940 ± 0.007
Leidschendam II	–	0.98 ± 0.02	1.034 ± 0.002
Leidschendam III	–	1.00 ± 0.02	0.91 ± 0.03
Leidschendam IV	–	0.934 ± 0.012	0.970 ± 0.011
Leidschendam V	–	1.00 ± 0.03	0.962 ± 0.011
Leidschendam VI	–	0.957 ± 0.006	0.965 ± 0.007
Leidschendam VII	–	0.972 ± 0.008	0.962 ± 0.014
Leidschendam VIII	–	0.98 ± 0.02	0.97 ± 0.02
Leidschendam IX	–	0.978 ± 0.006	0.944 ± 0.009
Leidschendam X	–	0.996 ± 0.007	0.936 ± 0.008
Group average	0.95 ± 0.03	0.980 ± 0.007	0.970 ± 0.011
Overall average		0.970 ± 0.007	

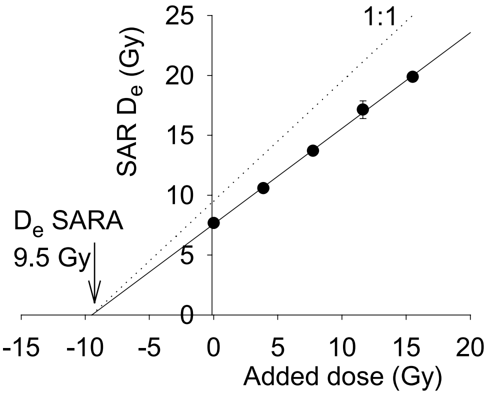

Fig. 7. Equivalent-dose determination on feldspar separates from sample Schelluinen II-6 using the SARA protocol [48]. The equivalent dose obtained by this procedure is not affected by changes in the trapping probability due to preheating, as the added doses are administered prior to any heating of the aliquots. In the absence of trapping sensitivity changes, the regression should follow the dotted line of unit slope.

eruption [16]. Based on the results of the fading tests (Table 4), we cannot completely rule out the existence of anomalous fading in our samples (average fading ratio 0.970 ± 0.007). In interpreting this fading ratio, one should also keep in mind that the laboratory time scale for fading tests is very short compared to the geological time scale. However, tests on samples that show an IR-OSL age underestimate as a result of anomalous fading tend to give an unambiguous indication of the presence of fading [45]. On balance we therefore think it is unlikely that anomalous fading is the main cause for the severe age underestimation found for our feldspar samples.

5.2. Sensitivity change

The SAR procedure was developed to overcome problems with sensitivity change during measurement [24,28,46]. However, Murray and Wintle [24] pointed out that if sensitivity changes occur between measurement of the natural OSL signal and measurement of the OSL from the test dose related to the natural, the SAR procedure will not detect or correct these. Wallinga et al. [47] have shown that heating of feldspar grains to temperatures higher than 200°C for 10 s can cause changes in the charge trapping probability, and thus can change the overall sensitivity. In both SAR and SAAD measurement procedures, such a change in trapping probability would occur during preheating of the aliquot (prior to the first measurement of the IR-OSL). Using samples from the same area as discussed here, Wallinga et al. [47] showed that the change in trapping probability resulted in underestimation of a known laboratory dose administered prior to any heating of the sample.

Changes in trapping probability because of heating can be avoided if laboratory doses are given prior to heating of the sample, as is the case in multiple-aliquot methods. To test for this, we applied the single-aliquot regeneration and added dose (SARA) procedure [48], which is (despite its name) a multiple-aliquot procedure. We used the SAR protocol for determination of the dose in those aliquots that just retained their natural signal and in those where a laboratory dose had been added to the natural dose, thereby slightly modifying the standard SARA procedure [48]. Using this protocol, higher equivalent doses are obtained than with direct SAR measurements (Fig. 7, Table 5). Although clearly an improvement for feldspar separates, the SARA procedure might not be the preferred protocol. Firstly, it is extremely time consuming, as the method needs equivalent-dose determinations to be carried out on a large number of aliquots. Secondly, the equivalent dose is obtained by extrapolation, which is not desirable, especially for older samples. Finally, a linear extrapolation might not be justified [3].

As an alternative to the use of a SARA procedure to circumvent the problem, a sample-dependent correction factor can be determined by measuring the extent of the change in trapping probability. For this purpose, three aliquots from each sample were bleached for at least 2 h in a Hönle SOL2 solar simulator, and subsequently given a dose similar to their natural dose. As this first dose is administered prior to any heating, it is expected to have the same trapping sensitivity as the natural dose. Hence, a correction factor for the natural equivalent dose can be derived by dividing the known laboratory dose

Table 5
Feldspar IR-OSL results corrected for sensitivity changes

Sample	Correction factor SAR[a]	Feldspar IR-OSL equivalent dose (Gy)			Feldspar IR-OSL age (ka)			Quartz OSL age (ka)
		SAR[b]	SARA[c]	Corrected SAR[d]	SAR[b]	SARA[c]	Corrected SAR[d]	
Winssen	0.94 ± 0.04	2.9 ± 0.2	4.2 ± 2.1	2.7 ± 0.2	1.52 ± 0.14	2.3 ± 1.1	1.44 ± 0.14	0.92 ± 0.10
Rumpt I-3	0.97 ± 0.02	2.8 ± 0.4	3.6 ± 0.6	2.7 ± 0.4	1.5 ± 0.2	1.9 ± 0.3	1.4 ± 0.2	1.23 ± 0.10[d]
Rumpt IV-2	1.217 ± 0.014	4.2 ± 0.3	6.9 ± 0.6	5.1 ± 0.4	1.47 ± 0.13	2.4 ± 0.2	1.8 ± 0.2	1.75 ± 0.10
Schelluinen II-3	1.16 ± 0.02	7.4 ± 0.5	11.2 ± 2.2	8.5 ± 0.6	3.8 ± 0.3	5.8 ± 1.2	4.4 ± 0.4	5.1 ± 0.4
Schelluinen II-6	1.13 ± 0.08	7.6 ± 0.4	9.5 ± 0.3	8.6 ± 0.8	4.0 ± 0.3	5.0 ± 0.3	4.5 ± 0.5	6.1 ± 0.5
Elden	1.20 ± 0.06	16.8 ± 1.2	–	20.2 ± 1.7	7.9 ± 0.6	–	9.5 ± 0.9	13.3 ± 0.8
Leidschendam I	1.22 ± 0.08	46.9 ± 1.8	52 ± 6	57 ± 4	28 ± 2	31 ± 4	34 ± 3	48 ± 4
Leidschendam II	1.34 ± 0.08	47.6 ± 1.5	–	64 ± 4	32 ± 2	–	43 ± 4	55 ± 6
Leidschendam III	1.32 ± 0.09	59 ± 4	–	78 ± 7	44 ± 4	–	58 ± 6	82 ± 9
Leidschendam IV	1.41 ± 0.13	71 ± 5	–	99 ± 11	38 ± 3	–	53 ± 7	71 ± 6
Leidschendam V	1.21 ± 0.13	52 ± 3	–	63 ± 8	35 ± 3	–	42 ± 6	61 ± 5
Leidschendam VI	1.43 ± 0.04	65 ± 3	–	93 ± 5	38 ± 3	–	55 ± 4	58 ± 4
Leidschendam VII	1.324 ± 0.007	146 ± 17	–	193 ± 23	100 ± 15	–	136 ± 18	120 ± 9
Leidschendam VIII	1.34 ± 0.07	191 ± 13	–	256 ± 22	140 ± 16	–	194 ± 20	158 ± 13
Leidschendam IX	1.36 ± 0.06	96 ± 6	137 ± 18	130 ± 10	71 ± 6	105 ± 8	96 ± 9	145 ± 16
Leidschendam X	1.40 ± 0.05	112 ± 4	–	156 ± 8	78 ± 5	–	109 ± 8	180 ± 28

[a] Correction needed to account for sensitivity changes due to preheating. Details are given in the main text.
[b] The SAR protocol [28] was used for equivalent-dose estimation.
[c] The SARA protocol [48] was used for equivalent-dose estimation.
[d] As derived using the correction factors given in the first column.

by the dose estimated by the SAR procedure. Using SAR and these correction factors (Table 5), similar ages were obtained as by the SARA protocol (Table 5). An apparently more elegant approach would be to preheat only to temperatures below 200°C for 10 s, but this is outside the temperature range needed to remove unstable trapped charge after laboratory irradiation (indicated by the rising plateau in this region in Fig. 5).

Either the use of the correction factors or the application of the SARA procedure improves the feldspar IR-OSL ages, and brings them closer to the independent ages and the quartz OSL ages (Table 5). However, even after correction for trapping probability there is still a clear age underestimation (Fig. 6). It must be recognized that the correction factor may be underestimated as a consequence of accidental heating of the extracts prior to measurement. During sample preparation the extracts are exposed to temperatures above ambient during drying at 60°C, during treatment with H_2O_2 (some samples were heated significantly by exothermic reactions), and during bleaching in the solar simulator. It is possible that even at these relatively low temperatures the trapping probability changes. In radioluminescence studies of potassium feldspar, sensitivity changes up to 50% have been shown to occur at temperatures below 100°C [49]. We strongly recommend that temperatures above ambient should be avoided at any stage during sample preparation for IR-OSL dating of feldspar separates.

It is not yet known whether the change in trapping probability in feldspar as a consequence of heating is common. In a preliminary investigation, the correction factor was determined for three feldspar separates from Denmark (samples 989201–989203 [50]) and one feldspar separate from New Zealand (sample GDNZ6 [8]). The correction factors obtained for a 10 s, 290°C preheat were all less than 1.1, indicating that the effect does not produce an error greater than 10% for these samples. For the samples from Denmark, identical results were obtained from the OSL dating of the quartz fraction and IR-OSL dating of the feldspar separates (both used SAR procedures for equivalent-dose determination [50]). Although it is not clear how widespread this problem is, we recommend that testing for the phenomenon should be routine practice when single-aliquot IR-OSL dating is applied to feldspar separates.

It should be stressed that underestimation in the luminescence age obtained on feldspar separates has also been found when multiple-aliquot procedures were used [9]. Moreover, correcting for changes in trapping probability or application of multiple-aliquot techniques does not eliminate the age underestimation for our samples. This indicates that there must be additional reasons for the underestimation. A possible candidate would be incomplete removal of unstable charge from laboratory irradiation. Incorporation of the correction factor for different preheats, as shown in Fig. 4, suggests that a true preheat plateau does not exist for this sample; after correction for changes in trapping probability the 'plateau' rises continuously. This indicates that unstable charge is present up to temperatures where almost all trapped charge is removed (10 s preheat $> 325°C$). The apparent plateau shown for the uncorrected data (Fig. 4) appears to be a consequence of two competing phenomena: incomplete removal of unstable charge giving a rising trend with temperature, and changes in trapping probability causing a decreasing trend.

6. Conclusions

OSL ages, obtained using the SAR protocol on quartz from fluvial channel deposits in the Rhine–Meuse Delta, are in excellent agreement with tight independent age control for the range of 1–13 ka. Thermal transfer was shown to result in a small overestimation of age, but this unwanted effect was largely avoided by using a less stringent preheat regime. Our results confirm the applicability of quartz OSL dating to establish absolute chronologies for late Quaternary sedimentary records in general, and fluvial records in particular.

Single-aliquot IR-OSL dating of feldspar separates proved to be less successful. The IR-OSL age of the feldspar samples is underestimated by up to 50% in comparison with independent age control (up to 13 ka), and quartz OSL dating results (up to 200 ka). We show that part of this age underestimation is caused by changes in the charge trapping probability as a consequence of heating of the sample during single-aliquot procedures. This problem can be circumvented by using the SARA protocol, or by determining a sample-dependent correction factor. Both procedures produce results that are in better agreement with the independent age control, but they only partly solve the underestimation problem. Clearly, our results indicate that previously established luminescence chronologies based on coarse-grain feldspar may need re-evaluation.

Considering the problems encountered in the IR-OSL dating of feldspar, we suggest that quartz is the mineral of choice for OSL dating of these deposits, and probably of late Quaternary sediments in general. Nevertheless, it is important that the problems with coarse-grain feldspar dating continue to be investigated, in view of the potential of feldspars to extend luminescence dating to much longer time scales than quartz.

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