

**Luminescence and Infrared-Radiofluorescence Dating of Fluvial  
Deposits from the Rhine System – Methodological Aspects and  
new Insights into Quaternary Geochronology**

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<b>Abstract</b>	Xiii
<b>Kurzfassung</b>	XV
<b>Chapter 1: Introduction</b>	
1.1 Purpose	1
1.2 Study areas	1
1.2.1 Heidelberg Basin	2
1.2.2 Lower Rhine Embayment (LRE)	3
1.3 Optical dating of fluvial deposits – an introduction	5
1.4 Outline of the thesis	7
References	8
<b>Chapter 2: Fluvial aggradation phases in the Upper Rhine Graben—new insights by quartz OSL dating</b>	
Abstract	12
2.1 Introduction	13
2.2 Regional geology and new insights from the Heidelberg Basin Drilling Project	15
2.3 Luminescence dating	17
2.3.1 Principles	17
2.3.2 Sampling and preparation	18
2.3.3 Dosimetry	19
2.3.4 equivalent dose measurements	20
2.3.5 Purity of the quartz OSL signal	23
2.4 Discussion of dating results	24
2.4.1 Equivalent dose distribution and bleaching	24
2.4.2 Age models	24
2.5 Interpretation of the OSL ages and comparisons with other available OSL data from the Rhine Graben	27
2.6 Conclusion	29
References	30

### **Chapter 3: Infrared Radiofluorescence (IR-RF) dating of Middle Pleistocene fluvial archives of the Heidelberg Basin (Southwest Germany)**

Abstract	34
3.1 Introduction	35
3.2 Geological setting	37
3.3 Principles of luminescence and infrared radiofluorescence dating	38
3.4 Sampling	40
3.5 Sample preparation	43
3.6 Dose rate	44
3.6.1 External dose rate	44
3.6.2 Internal potassium content	44
3.7 Infrared radiofluorescence dating procedure	45
3.8 Results	46
3.8.1 Dose distribution and statistical treatment	46
3.8.2 IR-RF ages and stratigraphic interpretation	47
3.9 Discussion and Conclusion	49
3.9.1 Chronology of the Upper Interlayer	49
3.9.2 Control mechanisms for fluvial dynamics	50
3.10 Acknowledgements	52
References	54

### **Chapter 4: Luminescence dating of Last Glacial and Early Holocene fluvial deposits from the Lower Rhine – Methodological aspects and chronological framework**

Abstract	59
4.1 Introduction	60
4.2 Geological overview	61
4.3 Sampling sites	62
4.3.1 Choice of sites	62
4.3.2 Sites with Laacher See Tephra	63
4.3.3 Older Lower Terrace sites	65
4.4 Luminescence dating	68
4.4.1 Principles	68
4.4.2 Sample preparation	70

4.4.3	Dose rate	70
4.4.4	Equivalent dose measurements	71
4.4.4.1	Quartz measurements	71
4.4.4.2	Feldspar measurements	74
4.5	Results and discussion	76
4.5.1	Equivalent dose distribution	76
4.5.2	Luminescence age estimates	78
4.5.2.1	Rheinberg section	78
4.5.2.2	Monheim-Hitdorf section	79
4.5.2.3	Aloysiushof/Dormagen section	80
4.5.2.4	Niederkassel and Libur sections	80
4.6	Summary and conclusion	81
4.7	Acknowledgements	83
	References	83

## **Chapter 5: Geoarchaeological studies on Roman time harbour sediments in Cologne – Comparison of different OSL dating techniques**

	Abstract	89
5.1	Introduction	90
5.2	Archaeological Background – Cologne during Roman times	91
5.3	Sediments at the sampling site	92
5.4	Radiocarbon dating	93
5.5	Luminescence dating	94
5.5.1	Sample preparation for OSL dating	94
5.5.2	Dose rate	94
5.5.3	Equivalent dose measurements	95
5.5.3.1	Single aliquot regenerative dose measurements for sample SW-I	95
5.5.3.2	Measurements of the post-IR blue-stimulated luminescence	95
5.6	Results of Luminescence dating	97
5.6.1	Equivalent dose distribution of sample SW-II and statistical treatment	97
5.6.2	Post IR-measurements on sample SW-I	100
5.7	Conclusion	103
	References	103

**Chapter 6: Some aspects on bleaching behaviour and stability of the pIR-YOSL signal**

6.1	Introduction	106
6.2	Tests concerning the bleaching behaviour	106
6.3	Signal stability	108
6.4	Conclusion	108
	References	109

**Chapter 7: Conclusion**

7.1	New insights into responses of the Rhine to tectonics and changes in climate	110
7.2	Methodological aspects	111
	7.2.1 Statistical treatment of skewed equivalent dose distributions	111
	7.2.2 Problem of feldspar contaminated quartz	112
	7.2.3 Comparison of quartz and feldspar dating methods applied to samples with age control	112
	References	113

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	Acknowledgements	115
	Curriculum Vitae	116
	Publication list	117

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1.1	Map of Germany with the study areas in the Upper Rhine Graben and Lower Rhine Embayment.	2
1.2	Drilling sites of the Heidelberg Basin drilling project (map modified after Gabriel & Frechen, 2008).	3
1.3	Map showing the location of the sampled open pits in the Lower Rhine Embayment (red dots). Further samples were taken from an archaeological site (Roman harbour, marked with blue dot) in Cologne (modified after Lauer et al., accepted a; Source of Map: Geological Survey NRW).	4
1.4	Graph showing the principles of luminescence dating. The signal is reset during transport due to daylight exposure. After burial, the signal is generated mainly due to alpha, beta and gamma rays emitted from decaying radionuclides (uranium, thorium, potassium) being present in the sediment.	6
2.1	Study area showing the three drilling sites within the Heidelberg Basin Drilling Project at Heidelberg/Uni Nord, Ludwigshafen Parkinsel and Viernheim.	13
2.2	Description of sedimentological features from the upper 40 metres of the Viernheim core (after Hoselmann 2008) including the sampling points for OSL dating. OSL samples were taken from aeolian and fluvial sands above the first intercalated finer grained horizon. Samples for OSL dating were taken from the upper 33 metres of the sediment core, which was drilled to a depth of 340 metres.	14
2.3	The Viernheim core, showing well-sorted aeolian sands covering more heterogeneous fluvial sand and gravel.	19
2.4	Results of dose recovery test for samples VH-LM II (2.4a) and VH-LM VII (2.4b). The ratio of the measured/given dose including the standard error on the mean was determined out of four aliquots used for each temperature. The line at 1.0 (y-axis) was added for orientation and represents a measured to given dose ratio equal to unity.	21
2.5	Representative growth- and decay-curves from samples VH-LM I, V, VII. The curves show the decay from the natural OSL signal, the first	22

- regenerated OSL signal and from the testdose OSL signal (left to right).
- 2.6** Radial plot (2.6a) and frequency histograms (2.6b and 2.6c) showing the 25  
distribution of equivalent doses from fluvial samples VH-LM V, VH-LM  
VI and VH-LM IX. The class-width of the frequency histograms was  
defined by the Median value from all dose errors. Incomplete bleaching  
of some of the grains is assumed to be the most likely reason for the  
scattering of equivalent doses. The adaptability and reliability of the  
chosen SAR protocol was checked by applying dose recovery tests. The  
frequency histogram from sample VH-LM VI (2.6b) suggests that there  
is one insufficiently bleached population of grains (cluster of dose values  
on the right side of the x-axis).
- 2.7** OSL age estimates of fluvial samples plotted against the Weichselian 28  
Pleniglacial temperature curve, based on the mean annual temperature in  
Western Europe (following Vandenberghe et al., 2004). The OSL age  
estimates for samples VH-LM III, VH-LM V, VH-LM VI, VH-LM VII  
and VH-LM IX (fluvial samples) are plotted into the time scale. The  
dotted lines are the error bars. OSL ages of samples VH- LM IV and VH-  
LM VIII are not included (see discussion in section 2.4.2). The OSL ages  
indicate that all periods of sedimentation occurred during periods of  
warming. Increasing temperatures may have yielded an increased  
sediment supply. At the same time the deposits have only been preserved  
due to tectonic subsidence.
- 3.1** Study area showing the three drilling sites within the Heidelberg Basin 36  
Drilling Project at Heidelberg/Uni Nord, Ludwigshafen Parkinsel and  
Viernheim.
- 3.2** Two term energy band model explaining the principle of IR-RF (modified 39  
after Erfurt and Krbetschek, 2003a). The characteristic fluorescence light  
emission at 865 nm is linked to an electron transition into the IR-OSL trap  
(trapping). The IR-RF is therefore independent from recombination  
centres. This electron transition is going via the conduction band (CB)  
whereas the IRSL is linked to a localized transition in which the electron  
is recombining with a neighbouring recombination site, but not via the CB  
(Trautmann, 2000).
- 3.3** Viernheim core (left side) and Ludwigshafen core (core P34, right side) 41

modified after Hoselmann (2008) and Rolf et al. (2008), respectively. The IR-RF age estimates are inserted into the graph. The quartz OSL dating results from Lauer et al. (2010) are also inserted into the Viernheim core. The dotted lines mark core sections that can most likely be correlated.

<b>3.4</b>	This photograph shows the core sections of the Viernheim core from 39 m – 42 m core depth. This core part represents the transition (marked by the bin) from the coarse-grained and gravel rich upper fluvial units and the Upper Interlayer which is characterized by finer material and a high content of organic material. Sample VH-RF I was taken directly on top of the Upper Interlayer as marked by the circle.	43
<b>3.5</b>	Response of the IR-RF signal to irradiation (sample VH-RF I). The signal is decreasing with irradiation time. A stretched exponential function was fitted to the IR-RF data (red line). The green line marks the residual of the IR-RF dose curve.	46
<b>3.6</b>	Distributions of measured equivalent doses from samples VH-RF II and VH-RF III. The values inside the black boxes were included for age calculations following the statistical method described in chapter 3.8.	47
<b>3.7</b>	Marine Isotope record with plotted IR-RF ages from samples VH-RF I, II, IV & V (red dots) and samples LH-RF I-III (blue squares). The IR-RF age from sample VH-RF III was not inserted because it is most likely overestimating the true age. The Isotope record graph was taken out of Litt (2007) and the shown chronostratigraphy is based on the chronostratigraphical table of Hesse established by the Hessian Agency for Environment and Geology (HLUG) and by the Agency for Geology and Mining Rheinland-Pfalz (LGB).	48
<b>3.8</b>	IR-RF ages plotted against the core depth.	52
<b>4.1</b>	Map showing the study area and the position of sampling sites (source: Geological Survey NRW / Krefeld).	60
<b>4.2</b>	Schematic profile showing fluvial units at the Rheinberg section.	64
<b>4.3</b>	Schematic profile showing fluvial units at the Monheim-Hitdorf section.	64
<b>4.4</b>	Reworked pumice from the Laacher See Volcano in the gravel pit at Monheim-Hitdorf.	65
<b>4.5</b>	Schematic profile representing fluvial deposits at Niederkassel.	66
<b>4.6</b>	Photo of fluvial deposits exposed at the Libur section.	67

<b>4.7</b>	Photo of cryoturbation feature found at the Libur section.	67
<b>4.8</b>	Aloysiushof/Dormagen section with sampling points for luminescence dating.	68
<b>4.9</b>	Results of dose recovery tests from samples RB-I, ALH-I, MHT-III and NK-I at various preheat and cutheat temperatures. The discs were at each temperature hold for 10 seconds. For measurements five aliquots were used respectively.	72
<b>4.10</b>	A dose response curve for quartz of sample MHT-III. After measuring the natural signal, five regenerative doses (D1-D5) were given. The natural signal was then interpolated into the growth curve to estimate the equivalent dose (De). The natural luminescence signal (Ln) and the responses to the artificial doses (Lx) were sensitivity-corrected by the response to a test dose (Tn, Tx). In the final cycle, the measurements for the first regeneration dose (D1) were repeated (D6, red triangle). The ratio of the two measurements (D6/D1) yields the recycling ratio which is at $1.05 \pm 0.08$ for this aliquot. This indicates, that sensitivity changes were successfully corrected.	73
<b>4.11</b>	Decay curves showing the IRSL (50°C) and the pIRIR (225°C) signal for potassium rich feldspar of samples RB-II and MHT-III. Both signals were individually used for equivalent dose calculations. The pIRIR signal was expected to be less affected from anomalous fading. However, the pIRIR signal is less light sensitive and therefore more difficult to bleach.	74
<b>4.12</b>	Stimulation and detection window of the pIR-YOSL signal. To detect the 410 nm feldspar emission, a BG3 + D410-30 filter combination was used.	76
<b>4.13</b>	Equivalent dose distributions of samples RB-I (4.13a) and MHT-III (4.13b). The class-width of the frequency histogram (4.13b) was defined by the median of dose errors. The distributions show that the samples seem to be well bleached. For calculating the equivalent dose, the central age model (CAM) was applied.	77
<b>5.1</b>	Map of the Roman time city of Cologne after Dietmar & Trier (2006). The red line marks the course of the new subway. The quarry Kurt Hackenberg Platz were samples where taken from is marked by the arrow. The map shows also the lateral side channel that could be used as a harbour during Roman times.	91

- 5.2** Sampling points for OSL samples SW-II and SW-I. For SW-II material 93  
was taken from an intercalated fluvial sand layer. From the more  
heterogeneous material from above and below the sand layer, material for  
radiocarbon dating was taken (samples RC-1, RC-2, RC-3). Sample SW-I  
was taken in a distance of about 5 meters west from SW-II.  
The strong deformation structures around SW-I were assumed to have  
been caused by anthropogenic movements in shallow water.
- 5.3** Results from the dose recovery test applied to sample SW-II using 95  
different preheat temperatures. The cutheat was always at 180°C, a  
hotbleach at 280°C was inserted into the protocol at the end of each SAR  
cycle. It can be seen, that a preheat at 220°C yielded the best dose  
recovery. For the measurements 4 aliquots were used at each preheat  
temperature.
- 5.4** Frequency Distribution of measured equivalent doses from sample SW-II. 98  
For the measurements 3mm sized aliquots were used. The class-width  
was defined by the median off all De-errors.
- 5.5** OSL age estimates from sample SW-II obtained by using different 99  
statistical approaches. The vertical line marks the beginning of the Roman  
settlement in the year 19/18 BC. The dated sediments can not be older and  
should correlate to the 1st century AD.
- 5.6** Comparison of decay curves of the quartz OSL signal from sample SW-I. 100  
The CW quartz OSL signal is affected by feldspar impurities (5.6a) what  
can be seen by the shape of the decay curve. The feldspar signal can be  
reduced by applying pulsed OSL (5.6b) or by measuring the CW blue-  
stimulated quartz OSL after exposing the material to infrared light for 100  
s at 50°C and/or 225°C (5.6c,d). A prior IR-bleach at 225°C or pulsed  
OSL yield the highest purity for the quartz signal in that case.
- 5.7** Distribution of measured equivalent doses from sample SW-I based on the 102  
post-IR quartz signal measured after IR exposure at 50°C (5.7a) and at  
225°C (5.7b). For measurements 6mm sized aliquots had to be used due to  
the dim quartz OSL signal. The use of medium aliquots means that it is  
not possible to point out scatter among dose distribution in an effective  
way.
- 5.8** Equivalent dose values from sample SW-I obtained by applying different 102

luminescence dating protocols.

- 6.1** Bleaching behaviour of the IRSL and pIR-YOSL signal, tested on a 107  
fluvial sample derived from the Viernheim core. The pIR-YOSL signal is  
slightly less light sensitive than the IRSL signal.
- 6.2** Growth curve of the IRSL- and pIR-YOSL (260°C) signal obtained from 108  
a Tertiary fluvial sample taken from the Ludwigshafen core (URG). It is  
shown that the pIR-YOSL signal seems to be characterized by a higher  
stability (less fading) than the IRSL signal.

<b>2.1</b>	Concentrations of radionuclides and calculated dose rates.	20
<b>2.2</b>	Measurements steps for the applied SAR protocol.	23
<b>2.3</b>	$D_e$ values and OSL age estimates of Viernheim samples using different age models.	26
<b>3.1</b>	Legend to sedimentological units of the Viernheim core after Hoselmann (2008).	42
<b>3.2</b>	Overview about dosimetry and IR-RF ages: The nuclide concentrations of $^{40}\text{K}$ , Th and U within the sampled material as well as the internal potassium content of feldspar samples define the total dose rate.	53
<b>4.1</b>	Sampling locations for luminescence dating.	61
<b>4.2</b>	Preheat (PH) and cutheat (CH) temperatures used in the SAR protocol (quartz OSL dating).	72
<b>4.3</b>	Measurement steps for luminescence dating of potassium rich feldspar (IRSL and pIRIR).	75
<b>4.4</b>	Measurement steps for the new feldspar dating protocol including the detection of the pIR-YOSL signal.	76
<b>4.5</b>	Overview over equivalent doses, quartz OSL age estimates and dose rates for all measured samples. For age calculations, the equivalent doses given by the central age model (CAM) were used. The $D_e$ mean values are quoted with the standard error on the mean. The error for the $D_e$ CAM values is given by the statistic age model.	77
<b>4.6</b>	IRSL and pIRIR age estimates and fading rates for samples RB-II and MHT-III. The given $D_e$ values (Gy) are based on the CAM (non fading corrected). Fading rates (g-values) were calculated after Huntley and Lamothe (2001). The quoted g-values show, that using the pIRIR (225°C) signal yields lower fading rates than using the IRSL (50°C) signal.	81
<b>4.7</b>	Luminescence age estimates using the common IRSL (50°C) signal and the pIR-YOSL signal measured at 260°C. The results show that the pIR-YOSL dating results yield in each case higher equivalent doses than the IRSL results and this gives a hint that the signal might yield a higher stability (less fading).	81
<b>5.1</b>	Radiocarbon ages.	93

<b>5.2</b>	Nuclide concentrations and dose rates.	94
<b>5.3</b>	Different luminescence dating protocols applied to sample SW-I.	96
<b>5.4</b>	$D_e$ -values and the OSL age estimates from sample SW-II using different statistical approaches. The standard deviation of sample SW-II (based on all counted $D_e$ -values) is at 70%. The recovered $D_e$ -values from the DR test have a standard deviation of only 8%. The finite mixture model distinguished 6 components.	99
<b>5.5</b>	$D_e$ -values from sample SW-I obtained by applying different luminescence dating protocols (protocol I, II and III as described in Table 3). $D_e$ -values were also calculated using the signal deriving from the prior IR-bleach (50°C and 225°C).	103

## **Abstract**

Fluvial aggradation and erosion is triggered by mechanisms like climate variations, tectonics, sea-level change and human impact. The Rhine system is one of the largest drainage systems in Europe and its sediments therefore provide important information about the palaeo-climate and tectonic evolution of Central Europe. To understand at what time for instance tectonic impulses or changes in climate, regulating sediment supply and sediment preservation, occurred, a reliable chronology for the fluvial sediments is mandatory.

In this thesis Luminescence and Infrared Radiofluorescence (IR-RF) dating were applied to fluvial deposited collected from the Heidelberg Basin (northern Upper Rhine Graben) and Lower Rhine Embayment, Germany. Optical dating of fluvial deposits is still challenging because in many cases the luminescence or IR-RF signal was not reset completely before burial. Further problems like feldspar impurities disturbing the quartz-OSL signal or a weak luminescence signal (bad signal to noise ration) of quartz can occur. Potassium feldspar gives much more luminescence (higher signal intensity) but it is effected by anomalous fading which has to be corrected. Fading corrections are problematic especially for old sediments. One aim of this thesis was to better overcome these problems and to contribute to the methodological progress among optical dating with focus on fluvial deposits. To do so, different dating approaches were tested on fluvial samples for which age control is available. Furthermore it was intended to establish a better chronological framework for Holocene, Upper and Middle Pleistocene fluvial sediments from the Rhine system.

The sediments in the Heidelberg Basin are characterized by heterogeneous, gravel-rich layers (cold stage deposits) and intercalated fine-grained layers hosting organic material (so called Interlayer). The latter were deposited during warmer climate periods. It was intended to obtain a reliable chronology for both, the warm stage and cold stage deposits. The quartz OSL ages demonstrate that the upper fluvial units (sediments above the Upper Interlayer) were deposited during the Last Glacial period (Weichselian).

To frame the sedimentation age of the Upper Interlayer and sediments below, IR-RF was used. For the Upper Interlayer the IR-RF ages point to a sedimentation age of  $\sim 300$  ka. This shows that there is a huge chronological gap between the Weichselian fluvial sediments and this interlayer. For the fluvial units below the Upper Interlayer it was possible to date up to  $\sim 640$  ka (100 m core depth at the Viernheim drilling site). For the Middle Pleistocene differences in the intensity of subsidence of the Heidelberg Basin mainly regulated the fluvial aggradation. During times of increased subsidence, accumulation space was created and the

sediments could be preserved. Hence, the IR-RF ages help now to better estimate the timing of subsidence of the Heidelberg Basin.

For the Lower Rhine the luminescence ages now yield a higher chronological resolution for the studied sections (mainly Lower Terrace) and help to better understand the past fluvial dynamics of the Rhine. It could for instance be shown that fluvial aggradation of many meters of sediments can happen within a very short time period. Samples which were taken with a vertical distance of > 5 meters from a section at Monheim-Hitdorf all yield equal OSL ages.

For some of the samples taken at the LRE independent age control was provided by Laacher See pumice (age ~ 12.9 ka). For these samples quartz OSL and feldspar measurements were conducted and the results were checked against the age control. For feldspar dating, the IRSL at 50°C was measured and after this, the post-IRSL signal was detected stimulated with red LED at 225°C (pIRIR signal). Next to this a new protocol was applied which includes the detection of a feldspar signal stimulated with yellow LED at 260°C after depleting the IRSL (50°C) signal (pIR-YOSL). The latter protocol was developed within this thesis.

It turned out that quartz dating worked well for the fluvial samples under study. The ages are in agreement with the age control and also the feldspar dating results agree with the quartz ages.

Further samples were taken from a Roman harbour exposed at Cologne. For these samples Roman artefacts gave a very precise age control. This gave the opportunity to test different statistical approaches for these incompletely bleached sands. Furthermore, different protocols were applied to minimize the feldspar signal in contaminated quartz and it was shown that pulsed OSL but also an IRSL bleach at 225°C prior to the detection of the quartz (blue stimulated) signal have very good potential to obtain a purer quartz signal.

The results which are presented in this thesis show that luminescence and IR-RF dating are powerful tools to establish a reliable chronological framework for fluvial deposits. There are still challenges (e.g. which statistical approach one should apply if samples are incompletely bleached). Nevertheless, the here applied dating approaches yielded reliable ages as for example demonstrated by quartz and feldspar ages from the Monheim-Hitdorf site (Lower Rhine). It was also of high relevance to point out that IR-RF dating could successfully be applied to samples being older than 600 ka.

## Kurzfassung

Fluviatile Sedimentation und Erosion wird durch Mechanismen wie Klimaänderungen, Tektonik, Änderungen des Meeresspiegels oder menschliche Einflüsse kontrolliert. Das Rheinsystem ist eines der größten europäischen Entwässerungssysteme und die rheinischen Sedimente speichern daher wichtige Informationen über die Entwicklung des Paläoklimas und der Tektonik Mitteleuropas. Um zu verstehen, wann beispielsweise tektonische Impulse oder Klimawechsel, welche die Sedimentzufuhr und die Sedimenterhaltung regulieren, auftraten, ist eine zuverlässige Chronologie der Flusssedimente unabdingbar.

In dieser Doktorarbeit wurden Flusssedimente vom Heidelberger Becken (nördlicher Oberrheingraben) und der Niederrheinischen Bucht (Deutschland) mittels Lumineszenz und Infrarot Radiofluoreszenz (IR-RF) datiert. Die optische Datierung von Flusssedimenten ist noch immer eine Herausforderung weil in vielen Fällen das Lumineszenz oder IR-RF Signal vor der Abdeckung nicht vollständig zurückgestellt worden ist. Weitere Probleme wie Feldspatverunreinigungen, welche das Quarz OSL-Signal stören oder ein schwaches Quarz-Lumineszenzsignal (niedrige Signal/ Hintergrund Rate) können auftreten. Kalifeldspat gibt deutlich mehr Lumineszenz (höhere Signalintensität). Problematisch ist hier jedoch das Phänomen des *Anomalous Fading*, das korrigiert werden muss. Fading-Korrekturen sind jedoch besonders für alte Sedimente schwierig.

Ein Ziel dieser Doktorarbeit war es, dazu beizutragen, bessere Lösungsmöglichkeiten für diese Probleme zu finden und einen Beitrag zur methodischen Weiterentwicklung der optischen Datierung von Flusssedimenten zu leisten. Auf Grund dessen wurden verschiedene Datierungsansätze an Flusssedimenten getestet, für die eine Alterskontrolle gegeben ist. Außerdem sollte ein besserer chronologischer Rahmen für Holozäne, Ober- und Mittelpleistozäne Flusssedimente des Rheins erarbeitet werden.

Die Sedimente des Heidelberger Beckens sind durch heterogene, schotterreiche Lagen (kaltzeitliche Ablagerungen) und zwischengeschaltete feinkörnige Lagen mit organischem Material charakterisiert (so genannte Zwischenhorizonte). Letztere wurden während wärmerer Klimaperioden abgelagert. Ziel war es eine zuverlässige Chronologie sowohl für die warmzeitlichen als auch für die kaltzeitlichen Ablagerungen zu erarbeiten. Die Quarz OSL Alter zeigen, dass die oberen Flussablagerungen (Sedimente oberhalb des Oberen Zwischenhorizontes) während des letzten Glazials (Weichsel) sedimentiert wurden.

Um das Ablagerungsalter des Oberen Zwischenhorizontes und der darunter liegenden Sedimente zu ermitteln, wurden IR-RF Datierungen durchgeführt. Die IR-RF Alter deuten auf

ein Sedimentationsalter des Oberen Zwischenhorizontes von  $\sim 300$  ka hin. Dies zeigt, dass zwischen den Weichselzeitlichen Flusssedimenten und diesem Zwischenhorizont eine deutliche chronologische Lücke auftritt.

Für die Flusssedimente unterhalb des Oberen Zwischenhorizontes war es möglich bis zu  $\sim 640$  ka zu datieren (100 m Kerntiefe bei der Viernheimbohrung). Während des Mittelpleistozäns kontrollierten vor allem unterschiedliche Subsidenzraten des Heidelberger Beckens die fluviatile Sedimentation. Zu Zeiten erhöhter Absenkung wurde Ablagerungsraum geschaffen und die Sedimente konnten erhalten werden. Die IR-RF Alter helfen daher den zeitlichen Ablauf der Absenkung des Heidelberger Beckens besser abschätzen zu können.

Für die untersuchten Aufschlüsse am Niederrhein (vor allem Niederterrasse) bieten die Lumineszenzalter nun eine bessere chronologische Auflösung und tragen zu einem besseren Verständnis über die Fluviatildynamik des Rheins in der Vergangenheit bei. Es konnte beispielsweise gezeigt werden, dass die Ablagerung von vielen Metern mächtigen Flusssedimenten innerhalb einer sehr kurzen Zeitspanne von statten gehen kann. Proben, die mit einer Vertikaldistanz von  $> 5$  Metern von einem Aufschluss bei Monheim-Hitdorf genommen wurden, ergaben alle einheitliche OSL Alter.

Für einige der Proben, die in der Niederrheinischen Bucht genommen wurden war über Laacher See Tephra (Alter  $\sim 12.9$  ka) eine Alterskontrolle gegeben. Für diese Proben wurden Quarz OSL und Feldspatmessungen durchgeführt und die Ergebnisse wurden mit der Alterskontrolle abgeglichen. Bei den Feldspatdatierungen wurde zunächst das IRSL Signal bei  $50^\circ\text{C}$  gemessen und anschließend das post-IRSL Signal aufgezeichnet, das mit einer roten LED bei  $225^\circ\text{C}$  (pIRIR Signal) angeregt wurde. Außerdem wurde ein neues Protokoll angewandt bei dem nach dem Bleichen des IRSL ( $50^\circ\text{C}$ ) Signals ein Feldspat Signal aufgezeichnet wird, das mit einer gelben LED bei  $260^\circ\text{C}$  angeregt wird. Das letztere Protokoll wurde im Rahmen dieser Doktorarbeit entwickelt. Die Ergebnisse zeigen, dass die Datierung an Quarzen für die untersuchten Flusssande gut funktioniert hat. Die Quarzalter stimmen mit der Alterskontrolle überein und auch die Ergebnisse der Feldspatdatierungen passen zu den Quarzaltern.

Weitere Proben wurden von einem römischerzeitlichen Hafen entnommen, der in Köln aufgeschlossen war. Über römischerzeitliche Artefakte war für diese Proben eine sehr genaue Alterskontrolle gegeben. Dies bot die Möglichkeit verschiedene statistische Ansätze an diesen unvollständig gebleichten Sanden zu testen. Des Weiteren wurden unterschiedliche Protokolle angewandt, um das Feldspatsignal in verunreinigtem Quarz zu minimieren und es konnte gezeigt werden, dass gepulste OSL aber auch eine IRSL-Bleichung bei  $225^\circ\text{C}$  vor der

Aufzeichnung des Quarzsignals (blaue Stimulation) sehr gut funktionieren, um ein reineres Quarzsignal zu erhalten.

Die in dieser Doktorarbeit gezeigten Ergebnisse verdeutlichen, dass Lumineszenz und IR-RF Datierungen sehr gute Methoden sind, um für Flusssedimente einen zuverlässigen chronologischen Rahmen zu erarbeiten. Zwar gibt es noch Herausforderungen (z.B. welches statistische Verfahren sich am besten eignet, wenn die Proben unvollständig gebleicht sind). Die hier angewandten Datierungsverfahren lieferten aber zuverlässige Alter, was beispielsweise an Hand der Quarz und Feldspatalter vom Aufschluss bei Monheim-Hitdorf (Niederrhein) gezeigt werden konnte. Von großer Relevanz ist auch, dass es möglich war mittels IR-RF Proben, die älter als 600 ka sind, erfolgreich zu datieren.

# Chapter 1

## Introduction

### 1.1 Purpose

In this PhD-thesis luminescence and infrared radiofluorescence (IR-RF) dating were applied to fluvial deposits deriving from the River Rhine System. The methods are described and potentials and challenges are demonstrated.

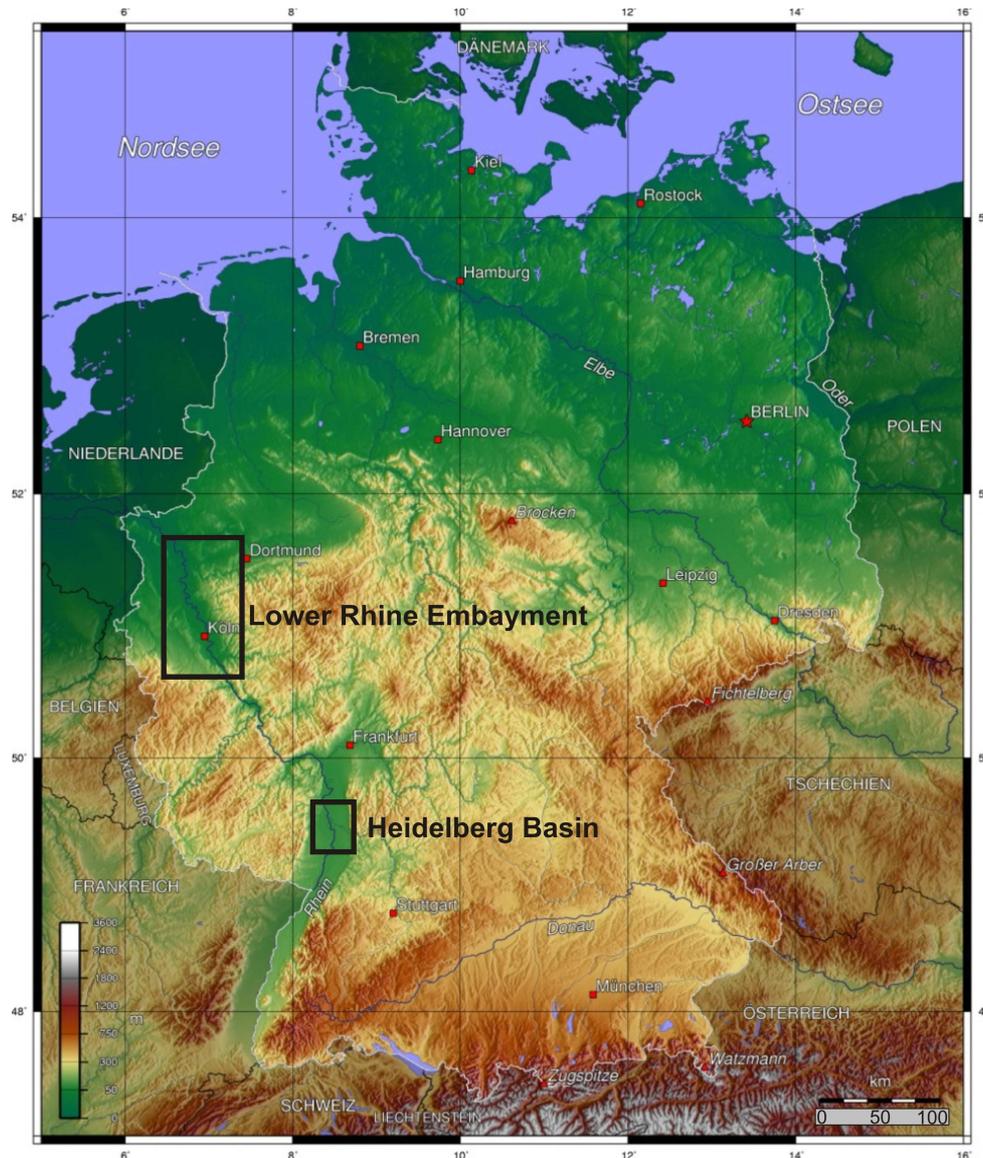
One aim of this thesis was to get a better chronological framework for Quaternary Rhine deposits. It is important to better understand these fluvial archives, since they record information about landscape evolution, climate changes and human activity.

Next to this, it was a concern to contribute to methodological progress in optical dating: Although during the last years, new methodological approaches made it possible to date fluvial deposits with higher precision than before, precision is still not sufficient for many questions. Thus, there is a need to further develop and improve optical dating methods for fluvial deposits, and to get information about the accuracy of various methodological approaches. This can be achieved, as applied in this thesis, by using different luminescence dating techniques for one sample and compare with independent dating.

### 1.2 Study areas

Samples were taken from gravel pits and an archaeological site located in the Lower Rhine Embayment (LRE) and from sediment cores drilled into the Heidelberg Basin which is located in the northern part of the Upper Rhine Graben (URG) (Fig. 1.1).

Both, the LRE and URG are part of the European Cenozoic Rift system (Preusser, 2008). Due to their long-term subsidence both areas are filled with large amounts of Tertiary and Quaternary sediments that contain important information about the Late Cenozoic landscape evolution of western Germany.



*Fig. 1.1: Map of Germany with the study areas in the Upper Rhine Graben and Lower Rhine Embayment (source of map: downloaded from [www.mygeo.info/landkarten\\_deutschland.html](http://www.mygeo.info/landkarten_deutschland.html) and modified).*

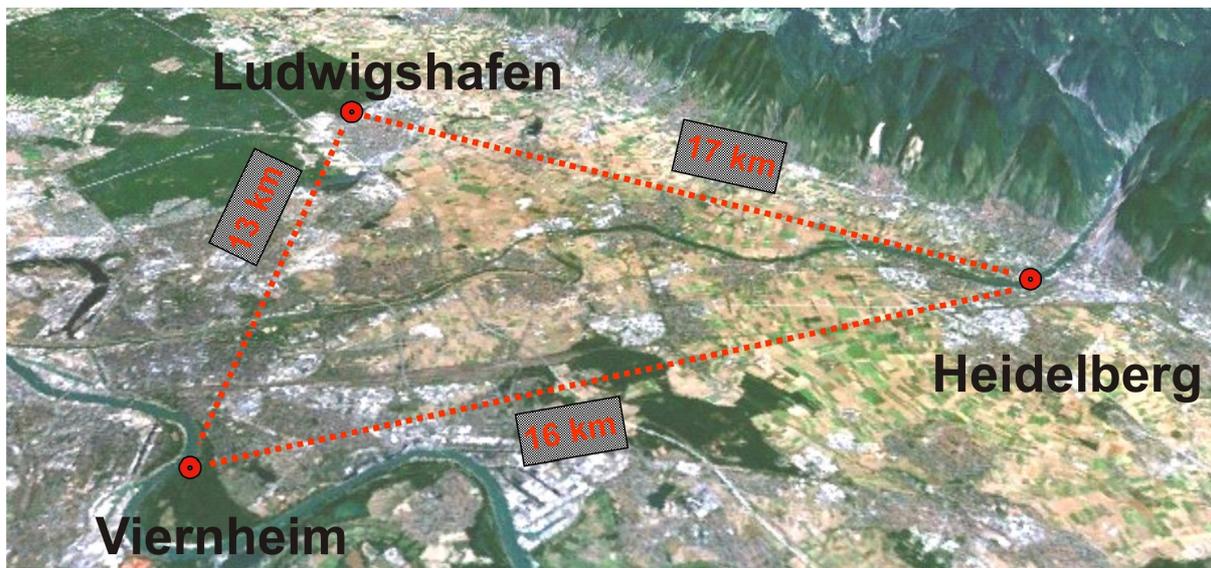
### 1.2.1 Heidelberg Basin

Due to long-term subsidence thick successions of Tertiary and Quaternary sediments were deposited and preserved within the sediment trap of the Heidelberg Basin (also called “Heidelberger Loch”). Therefore it is an important geological archive containing information about the interaction of tectonic and climatic factors, influencing the landscape evolution of Southern Germany. Due to this importance, the “Heidelberg Basin Drilling Project” (Gabriel et al., 2008) got started in the year 2002 in order to bring more light into the complex

architecture of unconsolidated fluvial and limnic sediments of this subsiding zone, to learn more about sediment supply and its forcing in space and time and to establish a chronological framework of the sediment sequences. To do so, boreholes were drilled into the unconsolidated Tertiary and Quaternary sediments at three drilling sites (Viernheim, Ludwigshafen and Heidelberg, see Fig. 1.2).

Luminescence and infrared radiofluorescence (IR-RF) dating were chosen as the tools to build up a more precise chronology for the Upper- and Middle Pleistocene deposits of the Heidelberg Basin. Samples for dating were taken from two sediment cores drilled near Viernheim and at Ludwigshafen.

In this PhD-thesis methodological background, dating results and interpretations in terms of fluvial dynamics being regulated by tectonic impulses and climate change are presented (chapter 2 and chapter 3).



*Fig. 1.2: Drilling sites of the Heidelberg Basin drilling project (map modified after Gabriel & Frechen, 2008).*

### **1.2.2 Lower Rhine Embayment (LRE)**

In the LRE samples were taken from five open pits (mainly Lower Terrace; Klostermann, 1992) located between Niederkassel and Rheinberg (Fig. 1.3, chapter 4).

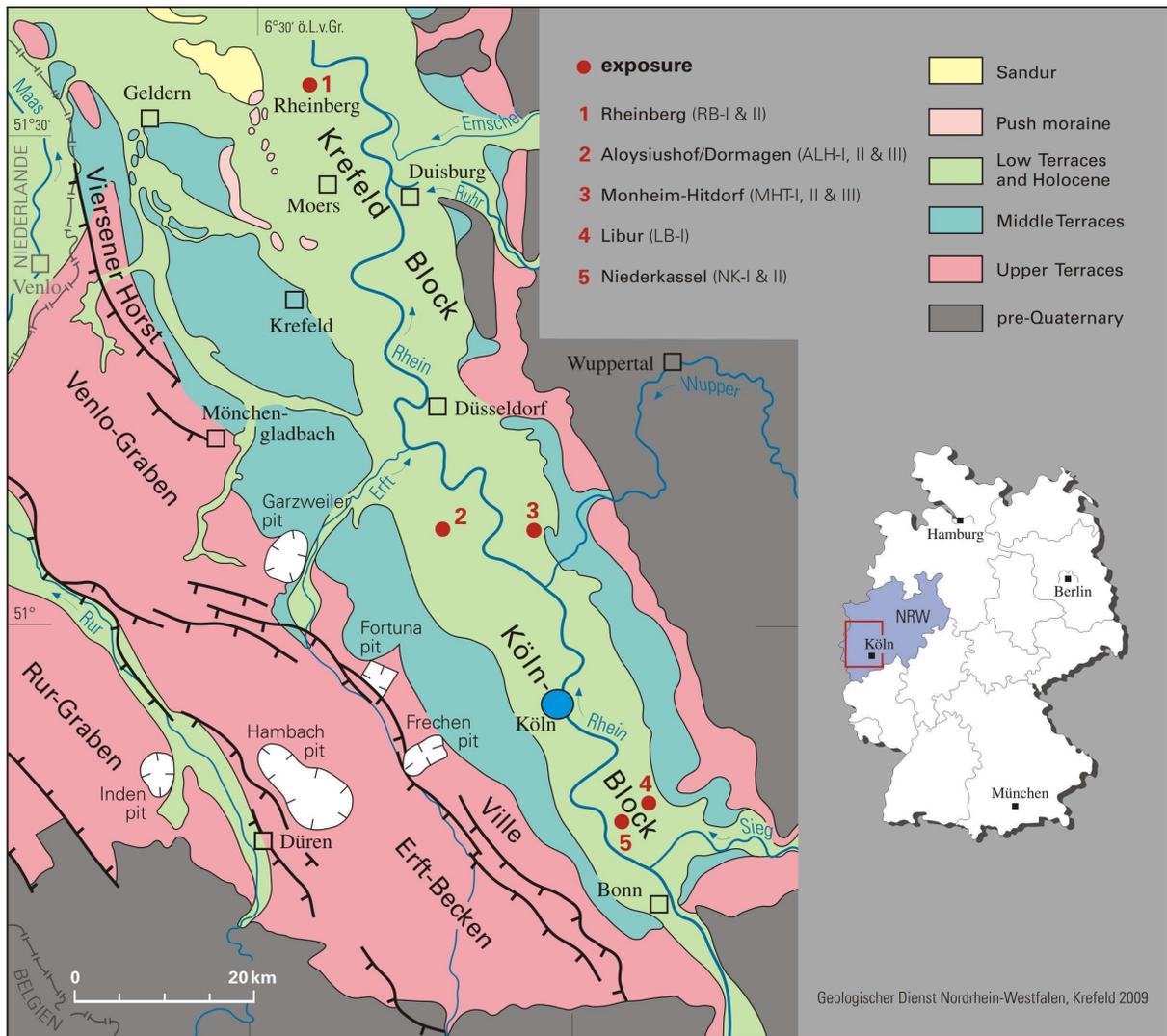


Fig. 1.3: Map showing the location of the sampled open pits in the Lower Rhine Embayment (red dots). Further samples were taken from an archaeological site (Roman harbour, marked with blue dot) in Cologne (modified after Lauer et al., accepted a; Source of Map: Geological Survey NRW).

Selected sections were of interest for luminescence dating due to two reasons:

Firstly, for two sections (Rheinberg and Monheim-Hitdorf) independent age control is available due to intercalated pumice originating from the Laacher See Volcano with an age of 12.9 ka (Bogaard, 1995). This gives a maximum sedimentation age for these fluvial sands and gravels. Based on this, luminescence dating results obtained from different methodological approaches (quartz and feldspar) could be checked against the age control.

The second aim was to obtain a higher chronological resolution for the studied Lower Terrace deposits. Although there exists already a large amount of literature about terrace stratigraphy and external forcing on fluvial dynamics of the Lower Rhine (e.g. Klostermann,

1992; Schirmer, 1990), it was often not possible to obtain a reliable chronological framework for the fluvial archives. Hence, there is still a strong demand on luminescence dating of fluvial units from the Lower Rhine system. Based on our dating results we present new information about sedimentation patterns at the sites.

Next to this, two fluvial samples of Roman age were taken from a former harbour exposed in Cologne (chapter 5). This harbour was discovered during the construction of a new public subway-tunnel. For these samples very good age control was available due to Roman artefacts which were found in layers above and below the sampling points.

### **1.3 Optical dating of fluvial deposits – an introduction**

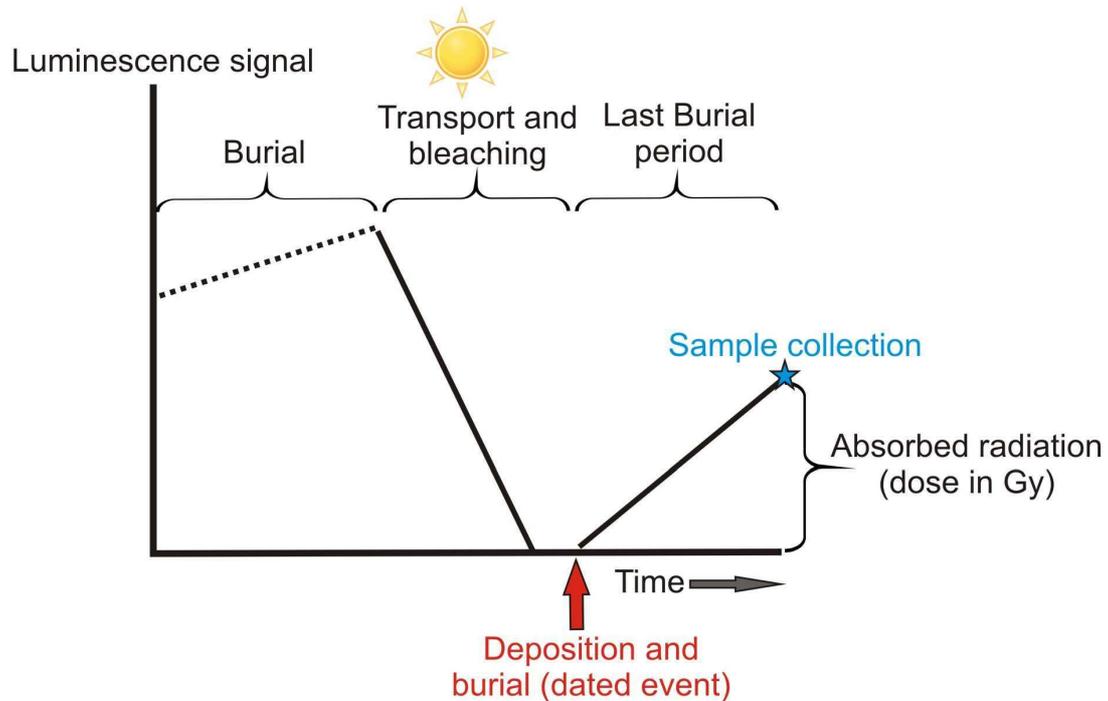
Luminescence and IR-RF dating are methods to determine the time passed since the last sunlight exposure of sediments. This correlates with the time of deposition followed by the burial period during which the luminescence signal is generated (Fig. 1.4).

Quartz or potassium-feldspar are normally the minerals of choice for luminescence dating (IR-RF dating only works on K-feldspars) as both minerals act as natural dosimeters and are ubiquitously found in natural environments. Theoretically also other minerals as e.g. volcanic glass could be used as natural dosimeters (Aitken, 1998), however, luminescence properties of these minerals are not well understood yet.

The main challenge of dating fluvial deposits is to point out if sediment grains were sufficiently bleached during transport to completely reset the luminescence signal. If this was not the case for all grains, statistical methods have to be applied to include only those grains for age calculation where the luminescence signal was completely bleached by sunlight (e.g. Rodnight et al., 2006; Galbraith et al., 1999).

Quartz is the mineral of choice when applying luminescence dating to fluvial deposits in most cases (Wallinga, 2002). Reasons for this are that its luminescence signal bleaches faster than that of feldspar as it is more light-sensitive, and furthermore it shows no so-called anomalous fading (a significant loss of signal during time) as in the case of feldspar. Nevertheless, there are several obvious limitations in connection with optically stimulated luminescence (OSL) dating of quartz: For instance, a low luminescence sensitivity (narrow signal to noise ratio) or feldspar micro-inclusions contaminating the quartz OSL-signal can be problematic. However, the most serious limitation of quartz is its quite low saturation limit, reaching saturation at values < 300 Gy (Wallinga, 2002). In some cases, quartz saturates even

at doses below 100 Gy (Fitzsimmons et al., 2010). Hence, the period datable with quartz-OSL is generally limited to the last glacial cycle, depending on the dose rate (natural radioactivity) of the sediment.



*Fig. 1.4: Graph showing the principles of luminescence dating. The signal is reset during transport due to daylight exposure. After burial, the signal is generated mainly due to alpha, beta and gamma rays emitted from decaying radionuclides (uranium, thorium, potassium) being present in the sediment.*

To date older sediments feldspar can be used as it saturates at much higher doses. However, as mentioned above there are some disadvantages of this mineral: Firstly, its luminescence signal is bleaching more slowly, and secondly it is affected by the phenomenon of anomalous fading (Wintle, 1973) which can be explained by quantum mechanical tunnelling (Vasil'chenko et al., 2005). In order to minimize this unwanted effect, recent studies tried to isolate more stable components of the feldspar signal than those measured before. For example, the signal is measured at elevated temperatures after having reset the infrared stimulated luminescence (IRSL) signal measured at 50°C (e.g. Thomsen et al., 2008; Buylaert et al., 2009; Thiel et al., 2009; Lauer et al., accepted a).

Infrared radiofluorescence (IR-RF) dating is another tool appropriate to date fluvial sediments. Although the IR-RF signal is less bleachable than the IRSL signal measured at

50°C making it problematic for young insufficiently bleached samples, this emission is very stable and recent studies show that IR-RF can be used to obtain reliable ages for fluvial deposits up to > 600 ka (Wagner et al., 2010).

#### **1.4 Outline of the thesis**

This PhD-thesis is composed of seven chapters. Chapters 2, 3, 4 and 5 are papers that were submitted to peer-reviewed journals.

Chapters 2 and 3 present and discuss results obtained from OSL and IR-RF dating applied to fluvial deposits collected from the Heidelberg Basin. The papers were published in “*Proceedings of Geologists Association*” (chapter 2, Lauer et al., 2010) and “*Geochronometria*” (chapter 3, Lauer et al., 2011 a). Chapter 4 presents dating results from open pits located in the Lower Rhine Embayment and is accepted in the journal “*Zeitschrift der Deutschen Gesellschaft für Geowissenschaften*” (Lauer et al., 2011 b). Chapter 5 is a mainly methodological study on OSL dating of fluvial sands sampled at the Roman harbour in Cologne. It is accepted in the journal “*Geochronometria*” (Lauer et al., 2011 c). Chapter 6 presents extracts from a submitted paper. This paper was sent to the journal “*Geomorphology*” and summarizes the most relevant results from the other papers of this thesis. Additionally *some new results* are integrated and only these are presented in chapter 6.

*The chapters deal with the following topics:*

**CHAPTER 2:** FLUVIAL AGGRADATION PHASES IN THE UPPER RHINE GRABEN—NEW INSIGHTS BY QUARTZ OSL DATING (LAUER ET AL., 2010).

In this chapter a single aliquot regenerative (SAR) dose protocol (Murray and Wintle, 2000; 2003) was used to establish a chronological framework for insufficiently bleached fluvial sands from the Heidelberg Basin originating from the early Holocene and the Last Glacial (Viernheim drilling site). It was possible to apply quartz OSL dating up to 33 meter core depth (~ 56 ka). For some samples pulsed OSL (POSL) was applied, in order to minimize the effect of feldspar contamination. To calculate age estimates, different statistical approaches (mean, minimum age model, central age model) were applied onto skewed palaeodose-distributions.

**Chapter 3:** INFRARED RADIOFLUORESCENCE (IR-RF) DATING OF MIDDLE PLEISTOCENE FLUVIAL ARCHIVES OF THE HEIDELBERG BASIN (SOUTHWEST GERMANY) (LAUER ET AL., 2011 A).

The infrared-radiofluorescence (IR-RF) dating technique on coarse-grain K-feldspar was used to establish a chronological framework for Middle Pleistocene sediment successions from the Viernheim and Ludwigshafen drilling sites (Heidelberg Basin). Based on IR-RF ages it was possible to obtain a better chronology for Middle Pleistocene aggradation periods at the Heidelberg Basin up to ~ 640 ka (100 m core depth at the Viernheim site). These results show that aggradation periods were mainly driven by tectonic influence.

**CHAPTER 4:** LUMINESCENCE DATING OF LAST GLACIAL AND EARLY HOLOCENE FLUVIAL DEPOSITS FROM THE LOWER RHINE – METHODOLOGICAL ASPECTS AND CHRONOLOGICAL FRAMEWORK (LAUER ET AL., 2011 B).

In this chapter, a numerical chronology was established for the five open gravel-pits located at the LRE using quartz OSL. Additionally, feldspar dating was applied to two samples for which age control was available due to the presence of pumice from the eruption of the Laacher See Volcano (~ 12.9 ka). For the latter samples we measured the IRSL signal at 50°C as well as the pIRIR signal at 225°C (Buylaert et al., 2009). Fading correction was conducted following Huntley and Lamothe (2001). Furthermore, we applied a new dating approach, measuring the luminescence signal of potassium feldspar stimulated with yellow light at 260°C after having annealed the IRSL signal measured at 50°C. The results obtained by the different methods were subsequently checked against stratigraphic age control.

**CHAPTER 5:** GEOARCHAEOLOGICAL STUDIES ON ROMAN TIME HARBOUR SEDIMENTS IN COLOGNE – COMPARISON OF DIFFERENT OSL DATING TECHNIQUES (LAUER ET AL., 2011 C).

Different OSL dating techniques were applied to quartz samples from the former harbour in Cologne originating from the Roman period. The precise archaeological age control gave the opportunity to test and compare different statistical approaches (e.g. minimum age model, finite mixture model, leading edge method) for an insufficiently bleached sample. For another quartz sample contaminated with feldspar, different dating protocols were tested in order to minimize the contribution of the feldspar signal. Thus, it was tried to deplete the feldspar signal by using pulsed OSL or by detecting the quartz signal after an IRSL stimulation at 50°C or at 225°C.

## CHAPTER 6: SOME ASPECTS ON BLEACHING BEHAVIOUR AND STABILITY OF THE pIR-YOSL SIGNAL

In chapter 4 it is demonstrated that the dating approach using the yellow stimulated feldspar signal after depleting the IRSL (50°C) signal (pIR-YOSL) yielded reliable age estimates for the studied fluvial sands from the LRE. In chapter 6, the signal stability of the pIR-YOSL signal was tested on a fluvial sample of Tertiary age where the natural luminescence signal was completely saturated. For another sample tests concerning the bleaching behaviour of the IRSL (50°C) signal and the pIR-YOSL signal were conducted (Lauer et al., submitted).

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## Chapter 2

### Fluvial aggradation phases in the Upper Rhine Graben—new insights by quartz OSL dating

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#### Abstract

The Upper Rhine Graben (URG) is characterized by a thickness of up to 500 m of unconsolidated Quaternary sediments, providing excellent records of the Rhine river system and its responses to tectonic and climatic changes. The most complete Quaternary sequence of fluvial and limnic-fluvial deposits is found in the Heidelberg Basin, due to its long-term subsidence since the mid-Eocene. The aim of this study is to provide a chronological framework using optically stimulated luminescence (OSL) dating of aeolian and fluvial sands derived from the upper 33 m of a sediment core, which was drilled into the Heidelberg Basin infill close to the village of Viernheim, Germany. The OSL ages demonstrate that the dated fluvial sediments were deposited during the last glacial period (Weichselian) and that there were at least three aggradation periods during this episode. The coversands that cap the sequence were emplaced during the early Holocene.

## 2.1 Introduction

The sediment archives from the Heidelberg Basin, located in the northern part of the Upper Rhine Graben (URG), provide excellent terrestrial climate records of supra-regional importance. Climatic forcing and tectonic impulses initiated the erosion and the aggradation of the river deposits. The sedimentological features and the lithological components within the sediments provide information on the landscape development and fluvial dynamics of the Rhine system.

In this study, optically stimulated luminescence (OSL) dating was applied to fluvial and aeolian sands in a sediment core from the Heidelberg Basin, sampled near Viernheim, Germany. The Viernheim core is part of the Heidelberg Basin Drilling Project, which is also including drilling sites at Heidelberg-Nord and at Ludwigshafen (Fig. 2.1).

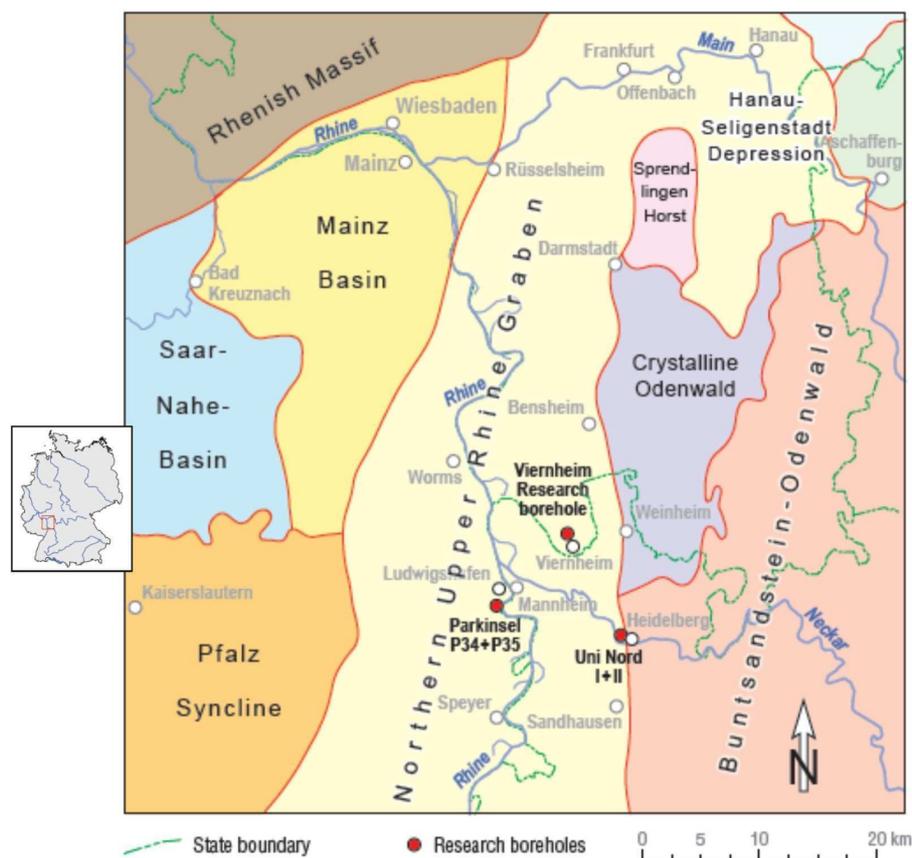
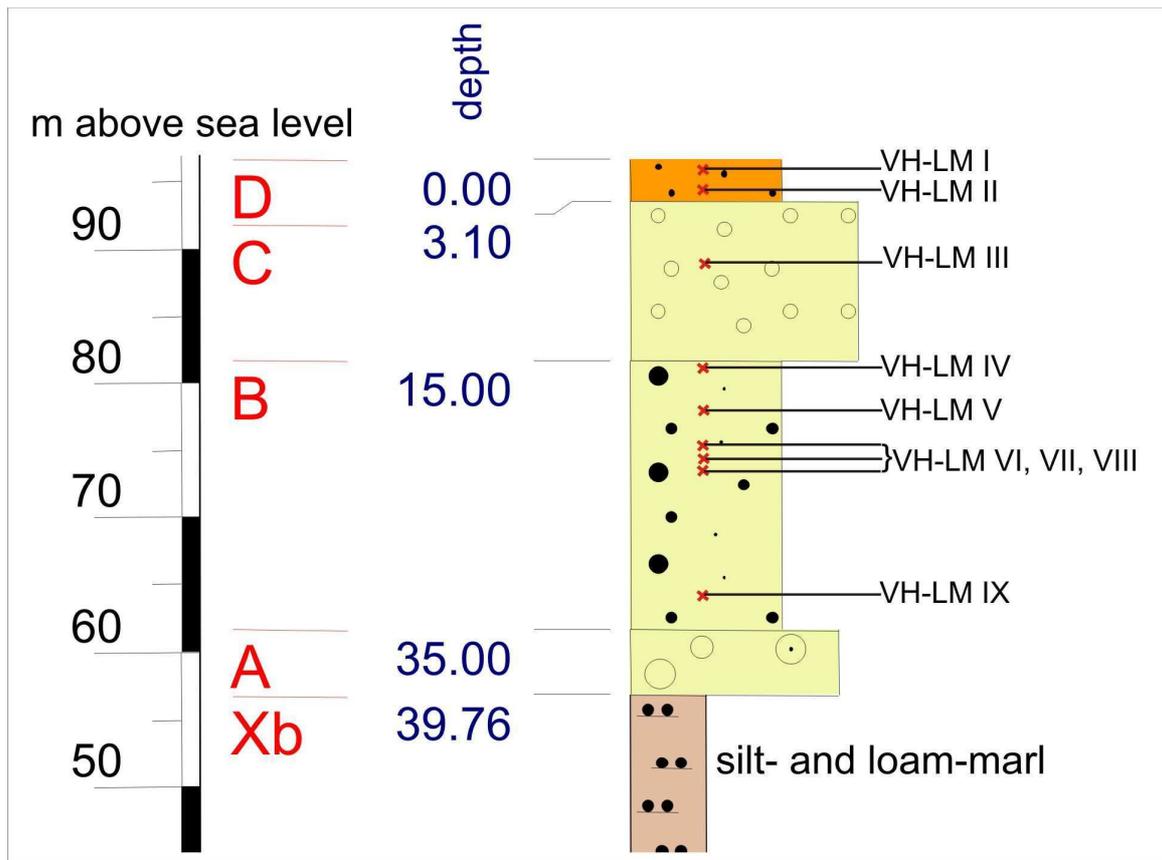


Fig. 2.1: Study area showing the three drilling sites within the Heidelberg Basin Drilling Project at Heidelberg/Uni Nord, Ludwigshafen Parkinsel and Viernheim (after Gabriel et al., 2008).

This project was conducted by the Leibniz Institute for Applied Geophysics (LIAG) and the three Geological Surveys of Baden-Württemberg, Hessen and Rheinland-Pfalz. The cores have been investigated using a multi-methodological approach (Gabriel et al., 2008), aiming to provide new insights into the complex sediment architecture of the Heidelberg Basin and to yield information about subsidence/uplift rates, basin dynamics, neo-tectonics, provenance, palaeo landscape evolution and sediment supply in space and time.



*Fig. 2.2: Description of sedimentological features from the upper 40 metres of the Viernheim core (after Hoselmann 2008) including the sampling points for OSL dating. OSL samples were taken from aeolian and fluvial sands above the first intercalated finer grained horizon. Samples for OSL dating were taken from the upper 33 metres of the sediment core, which was drilled to a depth of 340 metres.*

Unit	Depth in m below ground level	Description summary	Special features	OSL samples incl. sampling depth (m.b.s)
D	0-3.1	Markedly coarse, pedogenically overprinted aeolian sand	0-1 m core loss	VH-LM I 1.31-1.38 VH-LM II 2.64-2.75
C	3.1-15	Gravelly sands and gravels with fining-upward and coarsening-upward sequences, Rhenish portion strongly reduced	Neckar gravels	VH-LM III 8.51-8.61 VH-LM IV 15.58 - 15,68
B	15-35	Four fining-upward sequences, inhomogeneous, 27-32 m gravel	With Rhenish Facies in the fine grained parts, Neckar gravel	VH-LM V 18.28 - 18,36 VH-LM VI 22.69 - 22,78 VH-LM VII 23.41 - 23,49 VH-LM VIII 24.27 - 24,35 VH-LM IX 32.65 - 32,75
A	35-39.76	Xb is discordantly overlain by gravelly sands to gravels in three fining upward sequences	With Rhenish Facies in the fine grained parts, Neckar gravels	

Numerical age estimates have not yet been available for most of the fluvial units of the northern URG and, therefore, it has often been impossible to establish a precise stratigraphical framework for these sediments. The aim of this study is to deliver a chronological framework for the Upper Pleistocene units of the Heidelberg Basin by OSL dating of the upper part (33 m) of the Viernheim core. A detailed chronology is needed to answer questions about the timing of aggradation and erosion periods triggered by climate and tectonic impulses and to provide a better correlation of sediment units between the southern and northern parts of the Graben. The latter remains problematic because of the variable lithofacies, the differences in thickness of the formations (Hagedorn and Boenigk, 2008) and the lack of reliable chronologies.

## 2.2 Regional geology and new insights from the Heidelberg Basin Drilling Project

The Rhine system is one of the largest drainage systems in the European continent (Boenigk and Frechen, 2006; Preusser, 2008). With a catchment area of 185,000 km<sup>2</sup> (Westerhoff, 2008), it plays a central role in long-term sediment re-organization in NW Europe (Busschers, 2008). In its 1320 km long course from the Alps to the North Sea, the Rhine passes through various geological settings. The main controls on the preservation and formation of terraces have been tectonic activity and climatic variation. Whereas the uplift of the Rhenish shield caused 150 m of incision by the Rhine into the mainly Palaeozoic bedrock, forming a series of terraces (Boenigk and Frechen, 2006; Hoselmann, 1996), the long-term

tectonic subsidence of the Lower Rhine Embayment and the Upper Rhine Graben, which are part of the European Cenozoic rift system, created a major accommodation space for the accumulation and preservation of thick Tertiary and Quaternary deposits (Cloetingh et al., 2005; Frisch and Meschede, 2005).

The 35–45 km wide, NNE-trending Upper Rhine Graben is a failed rift valley of Tertiary age that extends for about 300 km between Basel (Switzerland) and Frankfurt (Germany). The infill of Pliocene–Quaternary sediments, which covers a complex fault system, is thought to be up to 1000 m thick (Ellwanger et al., 2005). A significant lithostratigraphic boundary within this sequence is defined by the first occurrence of Alpine components, characterized by a higher carbonate content within the sediments and a higher percentage of Alpine heavy minerals (Hagedorn and Boenigk, 2008; Hoselmann, 2008). In the southern part of the URG the Quaternary sediments are up to 270 m thick in the Geiswasser Basin (Bartz, 1974). In the Heidelberg Basin, located in the north-eastern part of the Rhine Graben, earlier boreholes have suggested a thickness of about 380 m of unconsolidated Quaternary sediments (Bartz, 1953). New studies support an estimate of > 500 m thickness of Quaternary deposits in the Basin depocentre (Ellwanger et al., 2008). The above-average thickness of unconsolidated sediments is related to the increased subsidence in parts of the southern and northern URG. The Heidelberg Basin or “Heidelberger Loch” is a depression within the Graben providing a sediment trap that hosts one of the most complete successions of Pliocene and Quaternary deposits in central Europe (Ellwanger et al., 2005; Gabriel et al., 2008). The Quaternary successions are characterized by thick gravel and sand layers that can be correlated with cold periods, and intercalated fine grained layers and (in part) peat horizons, representing warmer periods. Transitions from coarser grained, higher dynamic river facies to finer grained oxbow facies and clay and organic rich lacustrine facies are preserved in all the cores obtained from the Heidelberg Basin Drilling Project. Based on the characteristic Pleistocene geological formations, the establishment of a correlation between the drilling sites up to a certain depth is possible but chronological data is required for its verification. The thickness and facies characteristics of correlative cold- and warm-stage deposits vary between the different drilling sites. The above-mentioned thickness of Quaternary rocks of > 500 m, which was documented for the Heidelberg core (Ellwanger et al., 2008), is not present in the Viernheim core. Hoselmann (2008) provided evidence for the Pliocene–Pleistocene transition occurring at 225 m depth in the Viernheim sediment successions. The Ludwigshafen drilling site has yielded two cores (P35 and P34, see Fig. 2.1) 500 m apart. The Plio–Pleistocene boundary in P34 is estimated to be between 170 and 180 m

depth, whereas core P35 indicates a much thicker succession of Quaternary deposits of around 220 m. This suggests the importance of neo-tectonics in the Heidelberg Basin, controlling the thickness of this single stratum.

## **2.3 Luminescence dating**

### **2.3.1 Principles**

Optically stimulated luminescence (OSL) dating allows the determination of the time of the last exposure to sunlight of sediments and is a powerful tool for estimating the ages of sediments from the last glacial and interglacial periods. The method is based on photon emission during the recombination of electron–hole pairs (Aitken, 1998; Preusser et al., 2008). Following burial, minerals like quartz and feldspars accumulate radiation damage caused by alpha, beta and gamma rays from uranium, thorium and potassium ( $^{40}\text{K}$ ) and by cosmic rays. The signal is reset by exposure to sunlight during the transportation of the grains by wind, water or gravity. The time passed since the last exposure to sunlight can be calculated from the amount of accumulated dose in the minerals (palaeodose,  $D_e$ ) divided by the annual dose rate.

If the exposure of the sand to sunlight during transportation is insufficient, the resetting or zeroing of the radiometric clock is not completed before deposition (Wallinga, 2002), which is often the case for fluvial sands (Rodnight et al., 2006). This condition is strongly linked to the transport mechanism, with parameters like water depth, transport distance and sediment load regulating the efficiency of sunlight exposure (Murray et al., 1995; Jain et al., 2004). Applying the single-aliquot regenerative dose (SAR) protocol (Murray and Wintle, 2003) for numerous single grains or for small aliquots with only a small number of grains on each disc makes it possible to check whether bleaching was complete before burial. If a sample is only partly bleached, statistical approaches have to be applied after the measurements of  $D_e$  values to calculate ages using only those grains that received most sunlight (Wallinga, 2002; Arnold et al., 2007; Rodnight, 2008).

### 2.3.2 Sampling and preparation

Nine samples for OSL dating (VH-LM I–VH-LM IX) were taken from the Viernheim core between 1 and 33 m below surface (Fig. 2.2). Samples VH-LM I and VH-LM II were derived from well sorted aeolian sands on top of the fluvial sands and gravels (Fig. 2.3). The coversands reach a thickness of about 3 m at the drilling site. Samples VH-LM III–VH-LM IX were collected from more heterogeneous fluvial deposits. The fluvial sediments are characterized by calcareous sands, indicating a distal Alpine source area (Rhenish Facies), and intercalated reddish sandstone gravels that were delivered from local tributaries, mainly the Neckar (Hoselmann, 2008). A detailed description of the investigated Viernheim core can be found in Hoselmann (2008).

The samples were taken from core sections showing relatively homogeneous sand layers to guarantee higher precision in dose rate estimation. It was not possible to sample at equal intervals due to the lack of suitable material in those parts of the core dominated by gravel, with the consequence that chronological data are not available for depths of 9–15 and 25–32 m. The samples were first sieved to separate the 100–200  $\mu\text{m}$  grain-size fractions, and treated with 10% HCl and 30% hydrogen peroxide to remove carbonates and organic matter, respectively. The sand samples were then treated with sodium oxalate to remove clay particles, followed by heavy liquid separation using sodium polytungstate solutions (2.62 and 2.70  $\text{g}/\text{cm}^3$ ). Finally, the quartz-rich fraction was etched with 40% hydrofluoric acid (HF) to remove remaining feldspar and to etch the alpha-ray affected outer parts of the quartz grains. The HF etching was carried out for 60 min, and after the etching the grains were sieved again with a 100  $\mu\text{m}$  mesh. The quartz extract was then mounted onto stainless steel discs (aliquots) using silicon spray. For the fluvial sand samples VH-LM IV–VH-LM IX, for which insufficient bleaching was anticipated, small aliquots (diameter = 3 mm) with only a limited number of grains were used to investigate the distribution of  $D_e$  values (Wallinga, 2002). For the uppermost three samples (VH-LM I–VH-LM III) medium aliquots (diameter = 6 mm) were used.



Fig. 2.3: The Viernheim core, showing well-sorted aeolian sands covering more heterogeneous fluvial sand and gravel.

### 2.3.3 Dosimetry

Obtaining an age by luminescence dating is based on the following equation, after Aitken (1998):

$$Age = \frac{D_e}{w \cdot (D_\beta + D_\gamma) + D_{cosmic}} \left( \frac{Gy}{Gy/a} \right)$$

The dose rate is determined by the quantity of radionuclides such as uranium, thorium and potassium ( $^{40}\text{K}$ ) present within the natural sediment. Those nuclides emit  $\alpha$ -,  $\beta$ - and  $\gamma$ -rays, leading to the accumulation of radiation damage within the minerals. Cosmic rays ( $D_{cosmic}$ ) also contribute to a minor part to the ionisation of the grains. As noted above, the  $\alpha$ -ray affected layers of the grains were removed by HF etching. The energy derived from the  $\beta$ - and  $\gamma$ -rays ( $D_\beta + D_\gamma$ ) is attenuated by the factor  $w$ , related to the moisture and the attenuation of  $\beta$ -rays.

The concentrations of uranium, thorium and potassium (Table 2.1) were obtained by measuring the activity of  $^{238}\text{U}$ ,  $^{232}\text{Th}$  and  $^{40}\text{K}$  (Aitken, 1998) using a high purity germanium detector. The decay of  $^{40}\text{K}$  to  $^{40}\text{Ar}$  implies the emission of detectable gamma rays. This is not the case for  $^{238}\text{U}$  and  $^{232}\text{Th}$ , this involving an alpha decay and thus the activity was obtained by measuring the gamma emission of daughter nuclides:  $^{210}\text{Pb}$ ,  $^{214}\text{Pb}$ ,  $^{234}\text{Th}$  and  $^{214}\text{Bi}$  were

used for  $^{238}\text{U}$  and  $^{212}\text{Pb}$ ,  $^{208}\text{Tl}$  and  $^{228}\text{Ac}$  for  $^{232}\text{Th}$ . The equal activities of the measured radionuclides show that there is a radioactive equilibrium.

For the dose rate calculations, the cosmic dose rate was corrected to account for sediment thickness and altitude, following the recommendations of Prescott and Hutton (1994). The cosmic dose rates range from 177 mGy/ka (sample VH-LM I) to 14 mGy/ka (sample VH-LM IX). The correction factor for water content was calculated following Aitken (1985). For all fluvial samples a water content of 18% was presumed. This value is based on the knowledge that all sediment units below 4 m depth were water saturated, so that this approximate mean value could be calculated by determining the dry and water saturated weight of representative core parts. Possible variations within the palaeo-water content were not integrated for calculations. For the aeolian sands (VH-LM I and II) the natural moisture content was assumed to be at 6% and 10% respectively. To obtain this approximate value we measured the natural water content of aeolian sand exposed in the northern Upper Rhine Graben close to the drilling site.

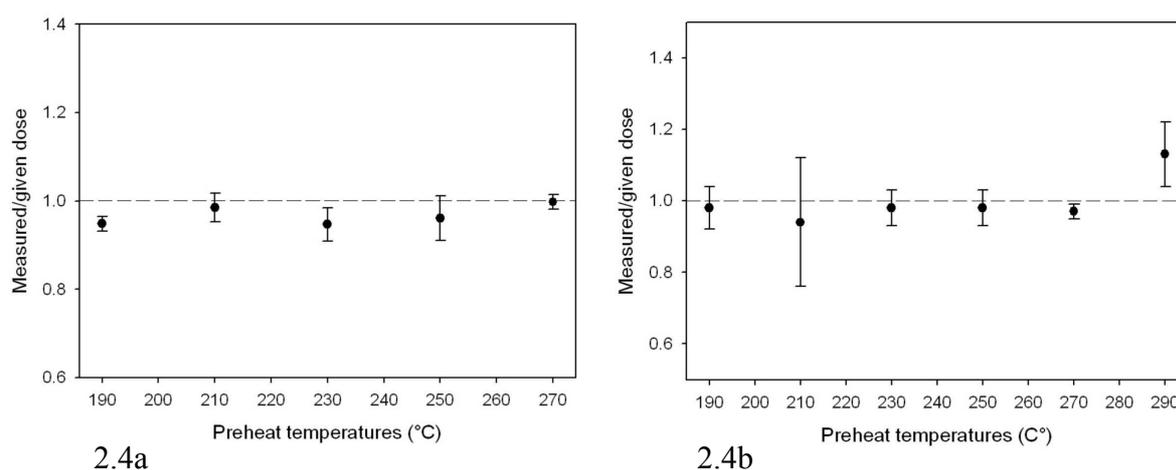
Sample code	% K	ppm Th	ppm U	Dose rate Gy/ka
VH-LM I	$1.17 \pm 0.02$	$7.69 \pm 0.13$	$2.06 \pm 0.04$	
VH-LM II	$1.63 \pm 0.03$	$2.49 \pm 0.08$	$0.78 \pm 0.04$	$1.83 \pm 0.21$
VH-LM III	$1.49 \pm 0.02$	$3.02 \pm 0.07$	$1.01 \pm 0.04$	$1.49 \pm 0.17$
VH-LM IV	$1.46 \pm 0.03$	$2.52 \pm 0.09$	$0.83 \pm 0.04$	$1.62 \pm 0.41$
VH-LM V	$1.51 \pm 0.03$	$3.39 \pm 0.08$	$1.04 \pm 0.04$	$1.70 \pm 0.22$
VH-LM VI	$1.52 \pm 0.02$	$4.41 \pm 0.08$	$1.14 \pm 0.03$	$1.57 \pm 0.20$
VH-LM VII	$1.36 \pm 0.03$	$4.32 \pm 0.10$	$1.14 \pm 0.05$	$1.64 \pm 0.20$
VH-LM VIII	$1.52 \pm 0.03$	$3.59 \pm 0.09$	$0.96 \pm 0.04$	$1.58 \pm 0.25$
VH-LM IX	$1.65 \pm 0.03$	$3.22 \pm 0.09$	$1.06 \pm 0.05$	$1.68 \pm 0.21$

Table 2.1: Concentrations of radionuclides and calculated dose rates.

### 2.3.4 Equivalent dose measurements

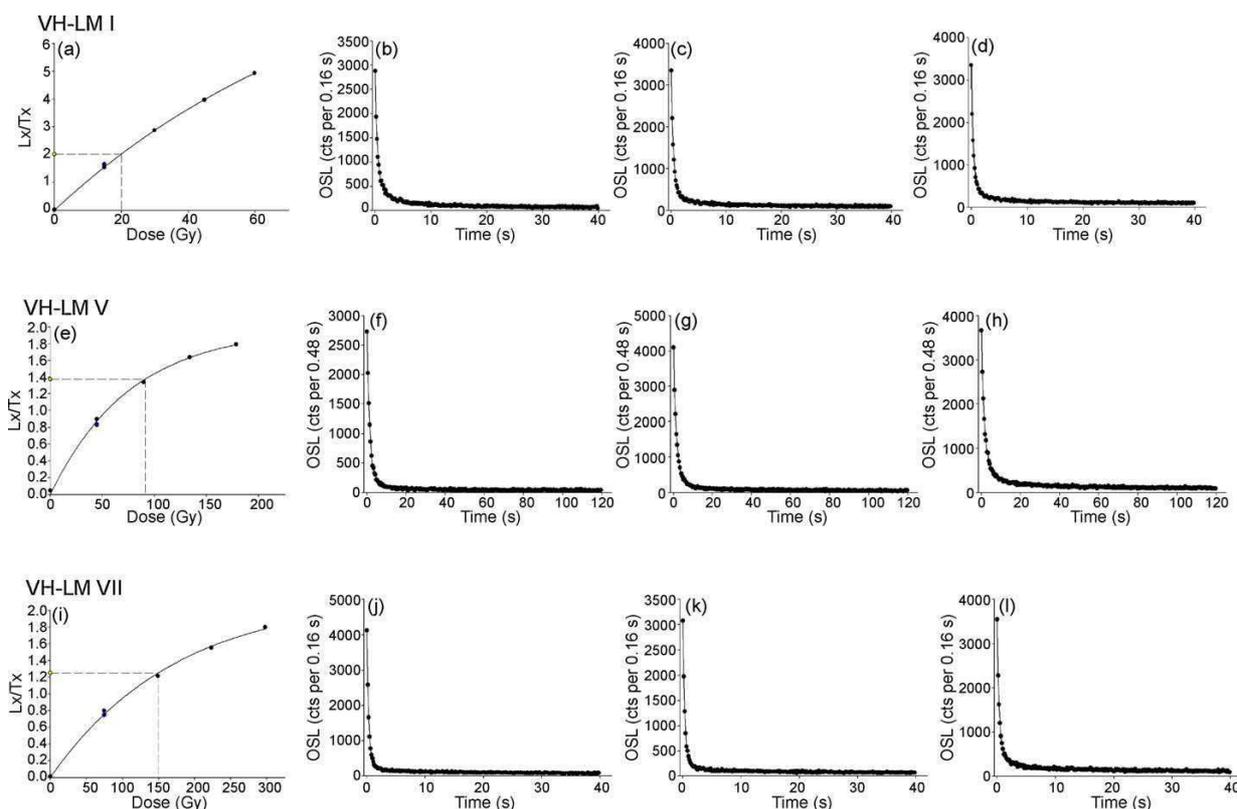
OSL measurements were made on a Risø TL/OSL DA-20 reader equipped with a  $^{90}\text{Sr}/^{90}\text{Y}$  beta source with a dose rate of  $\pm 0.12$  Gy/s and a pulsed LED attachment. The OSL signal was recorded at 125 °C during a 40 s readout using blue light emitting diodes (LEDs) at 470 nm in combination with an ultraviolet transmitting Hoya U-340 filter (7.5 mm).

Dose recovery tests including various preheat and cut-heat temperatures were applied to select appropriate parameters for the single-aliquot regenerative dose (SAR) protocol. Prior to the irradiation, all subsamples were bleached twice for 500 s at 125 °C using blue LED with a pause of 5000 s in between. The double bleach was used to make sure that there was no residual signal left before irradiation. The given dose was chosen to be close to the expected  $D_e$ . For samples VH-LM IV–VH-LM IX the initial part (0.8 s) of the OSL decay curve was integrated to be used as signal and the last 4 s were subtracted as background. For these samples the choice of background did not affect the results and so the approach yielding the most precise estimates of  $D_e$  was applied. For samples VH-LM I–III, early background subtraction (at 1.6–5 s) was used because it yielded the best dose recovery. This is presumably because the luminescence signal from samples VH-LM I–III is not dominated by the fast component.



*Figs. 2.4a and 2.4b: Results of dose recovery test for samples VH-LM II (2.4a) and VH-LM VII (2.4b). The ratio of the measured/given dose including the standard error on the mean was determined out of four aliquots used for each temperature. The line at 1.0 (y-axis) was added for orientation and represents a measured to given dose ratio equal to unity.*

For all samples SAR protocols with four or five regenerative doses were then applied, using a preheat for 10 s at 210 or 250 °C and a cut-heat at 190 or 210 °C, for young (VH-LM I–III) and old samples (VH-LM IV–IX) respectively (Fig. 2.4a and 2.4b and Table 2.2).



*Figs. 2.5a-l: Representative growth- and decay-curves from samples VH-LM I, V, VII. The curves show the decay from the natural OSL signal, the first regenerated OSL signal and from the testdose OSL signal (left to right).*

As a quality check, the recycling ratio for each SAR cycle was determined by re-measuring the first regenerative dose at the end of the cycles ( $D_x/D_1$ ). The IR depletion ratio was used to check if feldspar contamination has affected the quartz OSL signal (Duller, 2003). Therefore, two cycles with equal regeneration doses were inserted and the IR bleach for 40 s at 125 °C was added before OSL measurement in one of the two cycles. For the IR bleach, infrared light emitting diodes emitting at 870 nm were used. If there is feldspar in the quartz aliquots, the OSL signal is depleted by infrared (IR) bleaching. The aliquots that showed an IR depletion ratio and/or a recycling ratio of more than 10% were rejected.

Step	Treatment
1	Give dose, $D_i$ → irradiation of the sample
2	Preheat (250 °C or 210°C)
3	Stimulate for 40 s at 125°C using blue LED
4	Give test dose, $D_t$
5	Heat to 190/210°C
6	Stimulate for 40 s at 125 °C
7	Stimulate for 40 s at 280°C
8	Return to step 1

*Table 2.2: Measurements steps for the applied SAR protocol.*

### 2.3.5 Purity of the quartz OSL signal

Many aliquots showed significant IR depletion and thus were not taken into account for estimation of  $D_e$ . To check the purity of the quartz extracts, two samples (VH-LM IV and VH-LM VII) were investigated with a raster electron microscope. The geochemical scan of those subsamples indicated a high purity of the quartz extracts, with a feldspar contamination of < 0.5%. The presence of feldspar emission can be explained by the much higher sensitivity of feldspar in comparison to quartz, so that a single feldspar grain or feldspar impurities within the quartz crystals can significantly contribute to the OSL signal.

The strongest feldspar contamination was detected for samples VH-LM IV and V, for which > 50% of the aliquots had to be rejected when the luminescence properties were tested with a standard SAR protocol using 8 aliquots respectively. For these two samples, pulsed OSL was applied (Thomsen et al., 2008) and a modified SAR protocol was used, adopting the blue pulsed OSL (120 s) after IR exposure for 120 s at 125 °C (Wallinga et al., 2002; Zhang et al., 2007). The luminescence intensity was recorded only during the 100  $\mu$ s ‘off time’ in between 50  $\mu$ s pulses of stimulation. This setting made actual stimulation time to be 40 s for both IRSL and OSL. Since most of the feldspar OSL signal has decayed away within  $\sim$  1  $\mu$ s after the pulse is switched off, the off time signal is dominated by quartz OSL. Fig. 2.5f–h shows that the decay curves of sample VH-LM V using pulsed OSL are clearly dominated by the quartz fast component. The quality of the protocol used for pulsing was checked for sample VH-LM IV by applying a dose recovery test using the same preheat and cut-heat conditions as for samples VH-LM VI–IX (250 and 210 °C) and the given dose could successfully be recovered with a ratio of  $0.95 \pm 0.08$  (measured/given dose out of 5 aliquots).

## **2.4 Discussion of dating results**

### **2.4.1 Equivalent dose distribution and bleaching**

It can be assumed that incomplete bleaching is not a problem for the covering aeolian sands, which would have had longer exposure to sunlight than fluvial deposits, due to the rearrangement of the material by the wind (which can be taken for granted due to the well-sorted nature of the sands). However, in the case of river deposits it is necessary to check the bleaching properties of the grains by measuring small aliquots and analysing the dose distribution (Fig. 2.6a–c).

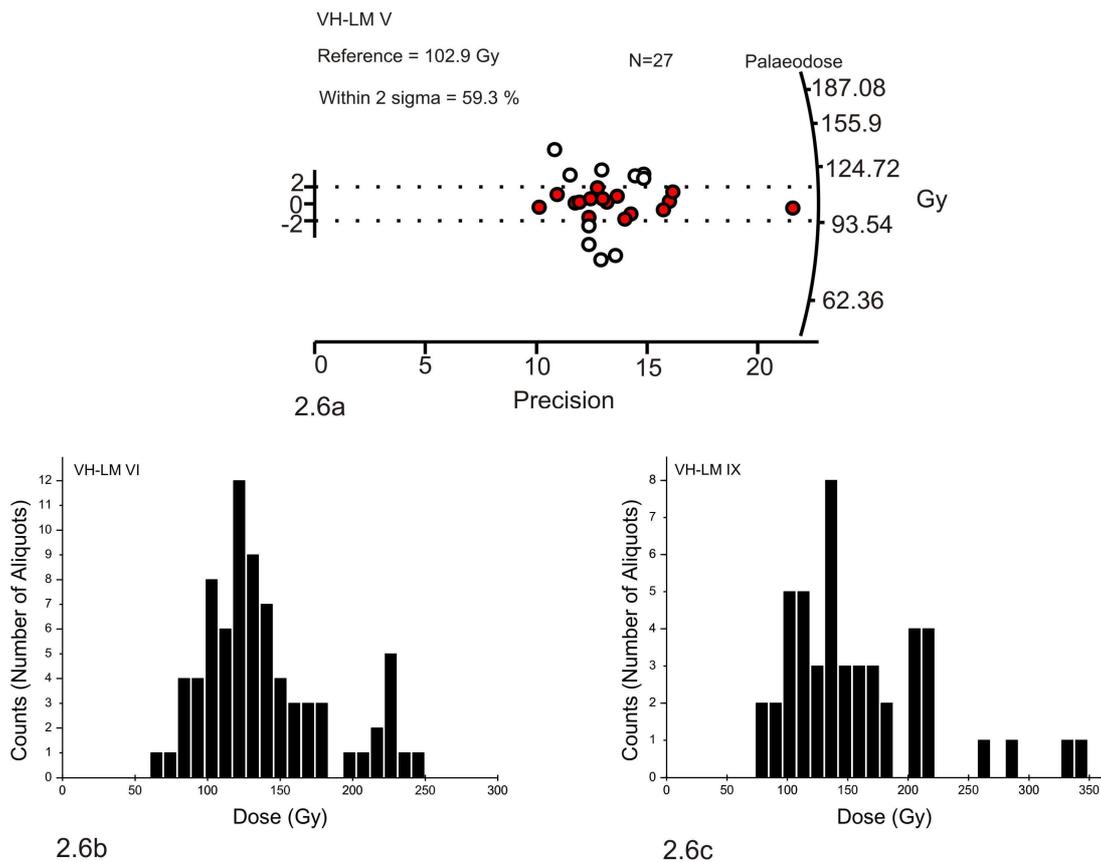
It can clearly be seen that the measured equivalent doses of fluvial samples are significantly skewed. It must be mentioned, however, that skewed dose distributions can also be linked to differences within micro-dosimetry, post-depositional mixing (Lomax et al., 2006) or instrumental error. In this case the most plausible reason for the broad dispersion of equivalent doses is insufficient sunlight exposure due to a sediment transport in a highly dynamic fluvial environment, with the consequence of there being a residual signal in some of the grains at time of sedimentation.

### **2.4.2 Age models**

The central age model (CAM) and the minimum age model (MAM3) were applied for all the fluvial samples (Galbraith et al., 1999). Table 2.3 shows the OSL age estimates calculated with the different statistical approaches. For the coversands, which are expected to be well bleached, the  $D_e$  values obtained from the CAM were used, which are indistinguishable with their mean  $D_e$  values. For fluvial samples, which showed significantly skewed dose distributions, the application of the MAM was necessary to avoid an age overestimation. For the youngest fluvial sample (VH-LM III) scattering among dose distribution could not be detected because medium aliquots were needed for measurements due to the dim OSL signal. For this sample, the CAM age fits well into the chronology obtained for the overlying sands. Nevertheless, partial bleaching also has to be assumed for this sample and, therefore, the CAM age has to be regarded as a maximum age. Special attention should be given to the OSL age estimates of samples VH-LM IV and V, which were measured by pulsed OSL. The CAM ages of those samples are similar ( $58.4 \pm 9.1$  and  $59.3 \pm 6.5$  ka respectively), whereas the MAM3 ages of  $26.1 \pm 5.2$  ka (VH-LM IV) and  $43.9 \pm 5.1$  ka

(VH-LM V) strongly diverge from each other. Hence, it can be assumed that the MAM3 age underestimates the true age for sample VH-LM IV.

Samples VH-LM VI, VII and IX show luminescence ages that are very close to each other ( $57.9 \pm 6.6 // 60.9 \pm 6.8 // 56.2 \pm 6.3$  ka, calculated by the MAM3). Considering the error limit, they can be interpreted as having been derived from one single fluvial unit. Sample VH-LM VIII was derived from this same unit but the age (MAM3 age =  $44.8 \pm 5.9$  ka) seems to underestimate the true age.



*Figs. 2.6a, 2.6b and 2.6c: Radial plot (2.6a) and frequency histograms (2.6b and 2.6c) showing the distribution of equivalent doses from fluvial samples VH-LM V, VH-LM VI and VH-LM IX. The class-width of the frequency histograms was defined by the Median value from all dose errors. Incomplete bleaching of some of the grains is assumed to be the most likely reason for the scattering of equivalent doses. The adaptability and reliability of the chosen SAR protocol was checked by applying dose recovery tests. The frequency histogram from sample VH-LM VI (2.6b) suggests that there is one insufficiently bleached population of grains (cluster of dose values on the right side of the x-axis).*

Sample code	Sampling depth (m)	Material	n/al-type*	Recycling ratio	D <sub>e</sub> -Mean (Gy)	D <sub>e</sub> -CAM (Gy)	D <sub>e</sub> -MAM3 (Gy)	Age-Mean (ka)	Age-CAM (ka)	Age-MAM3 (ka)	σ %
VH-LM I	1.31 – 1.38	coversand	17 m-al	1.023 ± 0.009	18.7 ± 0.7	18.5 ± 0.7	15.3 ± 2.8	9.3 ± 1.1	<b>9.2 ± 1.1</b>	7.6 ± 1.6	14
VH-LM II	2.64 – 2.75	coversand	23 m-al	1.004 ± 0.007	19.7 ± 0.7	19.6 ± 0.6	15.9 ± 2.8	10.8 ± 1.2	<b>10.7 ± 1.2</b>	8.7 ± 1.8	15
VH-LM III	8.51 – 8.61	fluvial	21 m-al	1.016 ± 0.009	21.2 ± 0.9	20.8 ± 0.8	16.6 ± 2.8	14.0 ± 1.5	<b>14.0 ± 1.5</b>	10.9 ± 2.2	17
VH-LM IV	15.58 – 15.68	fluvial	19 s-al	1.014 ± 0.014	106 ± 11	94.7 ± 9.4	42.3 ± 6.7	65.2 ± 10.2	58.4 ± 9.1	<b>26.1 ± 5.2</b>	42
VH-LM V	18.28 – 18.36	fluvial	27 s-al	0.970 ± 0.007	104 ± 5	101 ± 5	74.8 ± 4.3	61.3 ± 6.8	59.3 ± 6.5	<b>43.9 ± 5.1</b>	23
VH-LM VI	22.69 – 22.78	fluvial	76 s-al	1.022 ± 0.005	139 ± 5	135 ± 4	91.0 ± 4.5	88.3 ± 9.5	87.2 ± 9.2	<b>57.9 ± 6.6</b>	28
VH-LM VII	23.41 – 23.49	fluvial	45 s-al	1.054 ± 0.006	158 ± 8	149 ± 7	99.8 ± 4.6	96.5 ± 10.9	92.7 ± 10.2	<b>60.9 ± 6.8</b>	29
VH-LM VIII	24.27 – 24.35	fluvial	55 s-al	0.996 ± 0.005	139 ± 8	128 ± 7	70.7 ± 5.9	87.9 ± 10.2	82.8 ± 9.4	<b>44.8 ± 5.9</b>	38
VH-LM IX	32.65 – 32.75	fluvial	48 s-al	1.005 ± 0.008	158 ± 9	148 ± 7	94.4 ± 4.6	94.2 ± 10.9	89.8 ± 10	<b>56.2 ± 6.3</b>	33

\* m-al: medium aliquots, s-al: small aliquots

Table 2.3: D<sub>e</sub> values and OSL age estimates of Viernheim samples using different age models.

The here quoted recycling ratios give the mean recycling ratio of those aliquots which fulfilled the quality criteria. Latter are aliquots which showed an IR depletion ratio and/or a recycling ratio of less than 10%, the number of accepted aliquots is listed in the column *n/al-type*. The mean recycling ratios and D<sub>e</sub>- mean values are quoted with the standard error on the mean. The errors of the D<sub>e</sub>-CAM and D<sub>e</sub>-MAM3 values were given by the statistic models. The OSL age estimates are quoted with random errors.

## 2.5 Interpretation of the OSL ages and comparisons with other available OSL data from the Rhine Graben

The obtained OSL ages provide new insights into the complex sedimentology of the URG. It is known that the impacts of climate and tectonics play key roles in determining the sediment budget of each fluvial environment. In the case of the URG, tectonic activity can be seen as the main force regulating the aggradation and preservation of sediments, although the amount of sediment carried by the Rhine system can first of all be associated with climatically controlled geomorphological weathering processes and the intensity of discharge. The OSL age estimates show that at least 33 m of sediments from the last glacial could be preserved. The dated 25 m of fluvial sediments were not deposited in one uninterrupted aggradation cycle, however, as it is clearly demonstrated by the non-linear age increase with depth. It must be taken into account that sampling at high resolution was not possible and we have no evidence for the ages of the core sections that were not dated, but the similar OSL age estimates from 22 to 32 m depths show that deposition of fluvial units that are several metres thick can occur during short time periods, perhaps during just a few flood events.

From the OSL ages at least three fluvial aggradation periods can be demonstrated for the Weichselian (Fig. 2.7):

I: The oldest quartz OSL ages ( $56.2 \pm 6.3$  to  $60.9 \pm 6.8$  ka) provide evidence for aggradation during the Early to Middle Pleniglacial transition. Sample VH-LM IX (32.65–32.75 m depth) was taken from 7 m above the first intercalated finer grained and organic rich horizon (“Oberer Zwischenhorizont”). This layer was previously investigated by pollen analyses (Knipping, 2008) from the Ludwigshafen core (P34) and the pollen spectrum indicates a Cromerian age. That means that there must be a clear chronological gap in between the interglacial deposits and the overlying fluvial units.

II: A second period of aggradation is indicated by the MAM3 age of sample VH-LM V ( $43.9 \pm 5.1$ ), representing the sedimentation of Rhine deposits during the Middle Pleniglacial (MIS 3). This can also be confirmed by quartz OSL ages obtained by Frechen et al. (2009) from fluvial samples taken at the Hertten section, which is located in the Hochrhein Valley between Constance and Basel. Two samples from here yielded age estimates of  $46.4 \pm 4.1$  and  $37.3 \pm 7.4$  ka, which should be regarded as maximum ages.

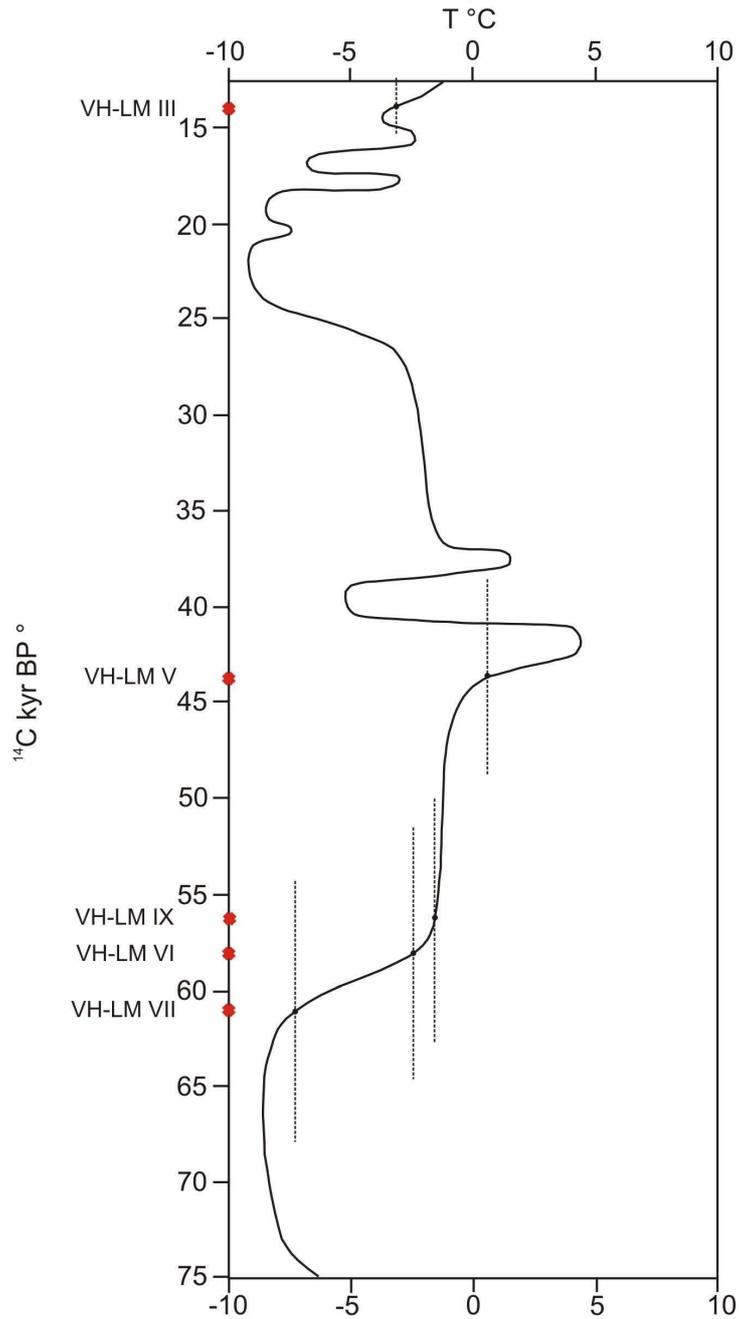


Fig. 2.7: OSL age estimates of fluvial samples plotted against the Weichselian Pleniglacial temperature curve, based on the mean annual temperature in Western Europe (following Vandenberghe et al., 2004). The OSL age estimates for samples VH-LM III, VH-LM V, VH-LM VI, VH-LM VII and VH-LM IX (fluvial samples) are plotted into the time scale. The dotted lines are the error bars. OSL ages of samples VH-LM IV and VH-LM VIII are not included (see discussion in section 2.4.2). The OSL ages indicate that all periods of sedimentation occurred during periods of warming\*\* (see comment at the end). Increasing\*\* temperatures may have yielded an increased sediment supply. At the same time the deposits have only been preserved due to tectonic subsidence.

III: The youngest unit is dated to the transition from the Late Pleniglacial to the Lateglacial period. It should again be mentioned that the CAM OSL age of  $14.0 \pm 1.5$  ka for sample VH-LM III, taken from  $\sim 8.5$  m depth, should be regarded as a maximum age. Also Erkens et al. (2009) postulate a terrace marking Late Pleniglacial to Lateglacial activity in the northern Upper Rhine Graben and in addition Frechen et al. (2009) established quartz OSL age estimates for fluvial sand from the Knobel section (southern URG) ranging from 11.6 and 15.9 ka.

At the Viernheim section the fluvial sediments are covered by early Holocene aeolian sands yielding OSL ages of  $10.7 \pm 1.2$  (VHLM II) and  $9.2 \pm 1.1$  (VH-LM I) ka. Kasse (2002) distinguished three major phases of aeolian sand accumulation in northwest and central Europe between the Last Glacial Maximum and the beginning of the Holocene. For the youngest phase Kasse (2002) claimed a time period between 13 and 10 ka cal BP but he also mentioned that the mobility of sand locally might have continued during the Preboreal period. Frechen et al. (2009) dated aeolian or fluvio-aeolian deposits at the Wyhlensection (Hochrhein Valley), obtaining quartz OSL age estimates that range from  $16.4 \pm 0.8$  to  $10.6 \pm 0.5$  ka, which are regarded as maximum ages. This fits well with the OSL age of 10.7 ka from Viernheim. However, the OSL age of 9.2 ka probably indicates that the remobilisation of aeolian sands on the drier parts of the floodplain in the northern Upper Rhine Graben perhaps continued until the Boreal period.

## 2.6 Conclusion

For the uppermost 33 m of the Viernheim core a chronology has been established by optically stimulated luminescence dating, which has been applied successfully to coarse-grained aeolian and fluvial quartz. It has to be mentioned that no independent age control is available for the investigated samples but the OSL ages should increase with core depth. The obvious age underestimation of sample VH-LM VIII underlines the methodological challenges. The problem of age overestimation caused by insufficient bleaching was addressed by applying statistical age models. Impurities within the quartz OSL signal from feldspar emissions were detected and pulsed OSL was successfully applied for samples VH-LM IV and V to isolate the quartz signal.

The OSL ages show that the studied fluvial sands were deposited during the last glacial period (Weichselian). Three aggradation periods could be demonstrated by OSL dating.

During periods of cold to warmer climate conditions the increased sediment supply provided an impulse for the aggradation of Rhine deposits that have been preserved thanks to tectonic subsidence creating accommodation space.

The age of the coversands was estimated to be 9–11 ka. For the early Holocene a period of strong morphodynamic activity, including significant movement of sand, can be assumed. Warmer climate conditions then led to increasing vegetation cover, consequently stabilizing the landscape. The coversands have not been reactivated for at least the past 9 ka, which thus implies that there was no remobilisation of the investigated sands due to human impact.

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**\*\*Comment on Figure 2.7:** The author of this thesis made some changes in the subtext of Figure 2.7 if compared to the Lauer et al. (2010) paper (here Figure 7).

In the published paper (Lauer et al., 2010, Proceedings of Geologists Association) it is written: “... The OSL ages indicate that all periods of sedimentation occurred during periods of *cooling*. *Decreasing* temperatures may have yielded an increased sediment supply. At the same time the deposits have only been preserved due to tectonic subsidence.”

As periods of sedimentation occurred during periods of *warming* (as rightly written in the Conclusion chapter of the Lauer et al., 2010 article) we changed this into: “...*The OSL ages indicate that all periods of sedimentation occurred during periods of warming. Increasing temperatures may have yielded an increased sediment supply. At the same time the deposits have only been preserved due to tectonic subsidence.*”

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## Chapter 3

This section has already been published. Please see below for corresponding information.

Title	Infrared Radiofluorescence (IR-RF) dating of Middle Pleistocene fluvial archives of the Heidelberg Basin (Southwest Germany)
Journal	Geochronometria
Volume/ Issue	2011: 38/1, 23-33
First author	Tobias Lauer
Co-authors	Matthias Krbetschek, Manfred Frechen, Sumiko Tsukamoto, Christian Hoselmann and Michael Weidenfeller
Date of acceptance	2.09.2010
Link (www)	<a href="http://www.springerlink.com/content/f722470128hgw261/">http://www.springerlink.com/content/f722470128hgw261/</a>
DOI	DOI 10.2478/s13386-011-0006-9
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**Abstract:** The infrared radiofluorescence (IR-RF) dating technique was applied to eight fluvial samples that were collected from two sediment cores at the Heidelberg Basin located near Viernheim and Ludwigshafen in southwest Germany. Based on the IR-RF derived ages of the samples it was possible to establish a chronological framework for the Mid-Pleistocene fluvial deposits of the Heidelberg Basin. The results allow us to distinguish between four main periods of aggradation. The lowermost sample taken from 100 m core depth lead to an IR-RF age of  $643 \pm 28$  ka pointing to a Cromerian period of aggradation (OIS 17–16). For the Elsterian it is now possible to distinguish between two aggradation periods, one occurring during the Lower Elsterian period (OIS 15) and a second during the Upper Elsterian period (OIS 12–11). For the so called Upper interlayer (or “Oberer Zwischenhorizont” — a layer of organic-rich and finer-grained deposits), the IR-RF results point to a deposition age of around 300 ka, with samples taken directly on top and out of this layer yielding IR-RF ages of  $288 \pm 19$  ka and  $302 \pm 19$  ka, respectively. Hence, the measured IR-RF ages clearly point to a deposition during the Lower Saalian period (OIS 9–8) whereas earlier studies assumed a Cromerian age for the sediments of the Upper Interlayer based on pollen records and also mollusc fauna. The new IR-RF dataset indicates that significant hiatuses are present within the fluvial sediment successions. In particular the Eemian and Upper Saalian deposits are missing in this part of the northern Upper Rhine Graben, as the 300 ka deposits are directly overlain by Weichselian fluvial sediments. It is obvious that time periods of increased fluvial aggradation were interrupted by time periods of almost no aggradation or erosion which should have been mainly triggered by phases of increased and decreased subsidence of the Heidelberg Basin.

## Chapter 4

This section has already been published. Please see below for corresponding information.

Title	Luminescence dating of Last Glacial and Early Holocene fluvial deposits from the Lower Rhine – Methodological aspects and chronological framework
Journal	Zeitschrift der deutschen Gesellschaft für Geowissenschaften (ZDGG),
Volume/ Issue	2011: 162/1, 47-61
First author	Tobias Lauer
Co-authors	Manfred Frechen, Josef Klostermann, Matthias Krbetschek, Georg Schollmayer and Sumiko Tsukamoto
Date of online-publication	1.3.2011
Link (www)	<a href="http://www.schweizerbart.de/papers/zdgg/detail/162/75689">http://www.schweizerbart.de/papers/zdgg/detail/162/75689</a>
DOI	DOI 10.1127/1860-1804/2011/0162-0047
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**Abstract:** Luminescence dating was applied to Last Glacial and Early Holocene fluvial deposits derived from the Lower Rhine. The aim was to obtain a robust chronology for the sections (open pits) under study in order to contribute to a better understanding of past fluvial dynamics of the River Rhine. Furthermore, different luminescence dating methods (quartz OSL, feldspar IRSL and pIRIR as well as pIR-YOSL) were compared and tested by applying them to sands sampled at Rheinberg and Monheim-Hitdorf where independent age control is provided by intercalated pumice originating from the eruption of the Laacher See Volcano, about 12 900 a ago. The obtained quartz ages are in agreement with the age of the marker tephra. Also the feldspar luminescence age estimates agree with the quartz OSL ages. For the Rheinberg and Monheim-Hitdorf sections the obtained ages now yield a very precise chronology. Based on this chronology a very rapid fluvial aggradation could be demonstrated for the sediment succession at the Monheim-Hitdorf site occurring during Younger Dryas. At Rheinberg it was shown that the Laacher See pumice was reworked for long time as the luminescence ages point to a Boreal period of aggradation (~ 4 ka after the eruption of the volcano). For the older Lower Terrace sites (Aloysiushof/Dormagen, Niederkassel, Libur) the ages now yield a reliable chronological framework for the fluvial aggradation helping to better understand the timing of changes in fluvial dynamics.

## Chapter 5

This section has already been published. Please see below for corresponding information.

Title	Geoarchaeological studies on Roman time harbour sediments in Cologne – Comparison of different OSL dating techniques
Journal	Geochronometria
Volume/ Issue	available online
First author	Tobias Lauer
Co-authors	Reiner Bonn, Manfred Frechen, Magret C. Fuchs, Marcus Trier and Sumiko Tsukamoto
Date of acceptance	10.09.2010
Link (www)	<a href="http://www.springerlink.com/content/u3615741v1x306x0/">http://www.springerlink.com/content/u3615741v1x306x0/</a>
DOI	DOI 10.2478/s13386-011-0020-y.
Copyright notice	Reproduction of this section only by permission of the rightholder.

### Abstract:

Due to the construction of a new North-South subway in Cologne, Roman time harbour sediments were exposed and were sampled for luminescence dating. A very good independent age control was given by the precise knowledge of the chronology of Roman activity and by radiocarbon ages of charcoal samples. Hence, different methodological approaches within luminescence dating were applied for Holocene heterogeneously bleached fluvial samples and were compared to the known ages. For one sample, optically stimulated luminescence (OSL) dating was applied to coarse-grained quartz using a single aliquot regenerative-dose (SAR) protocol. After  $D_e$ -measurements, different statistical approaches were tested (i.e. arithmetic mean, median, minimum age model, finite mixture model, leading edge method and the Fuchs and Lang approach). It is demonstrated that the Fuchs and Lang approach along with the leading edge method yielded the best matching OSL ages with respect to the known ages.

For the other sample which showed feldspar contamination within the quartz signal, the post-IR blue stimulated luminescence (double SAR protocol) was measured in three different ways to calculate the  $D_e$ -value: with continuous wave (CW) stimulation with an IR-bleach at 50°C and at 225°C for 100 s prior to the OSL, and pulsed OSL (POSL). It was demonstrated that the IR-stimulation at 225°C has very good potential to remove the feldspar signal contribution as well as pulsed OSL, but the former might deplete parts of the quartz OSL signal.

## Chapter 6

### Some aspects on bleaching behaviour and stability of the pIR-YOSL signal

#### 6.1 Introduction

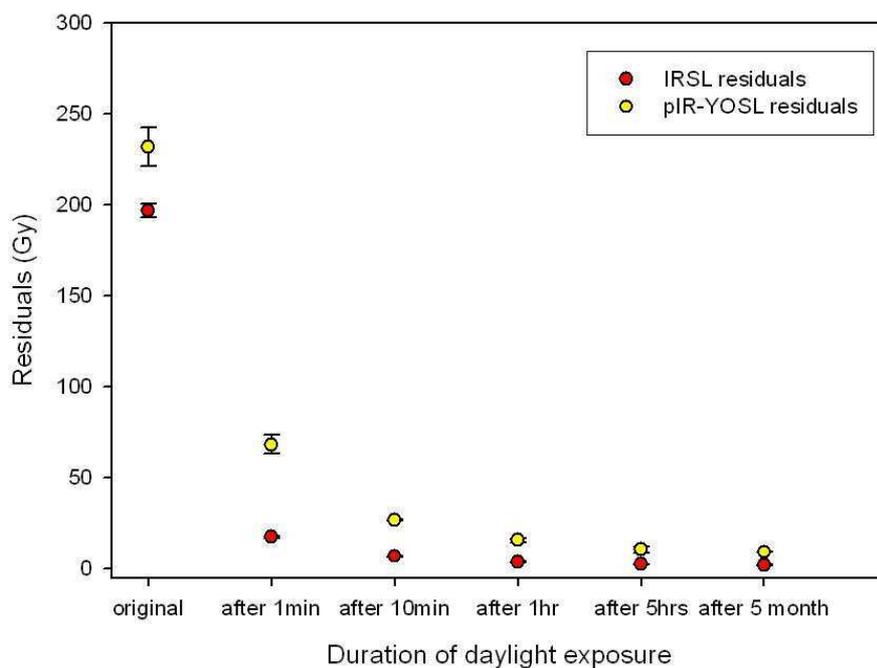
This chapter presents some new aspects concerning bleaching behaviour and stability of the yellow stimulated luminescence signal from potassium feldspar. Excerpts from a manuscript that was submitted to the journal “*Geomorphology*” (Lauer et al., submitted) are shown.

As the correction of anomalous fading is one of the major factors of uncertainty to obtain reliable feldspar ages, different studies within the last years tried to search for more stable feldspar signal components (e.g. Thomsen et al., 2008; Buylaert et al., 2009; Thiel et al., 2010). In chapter 4 of this thesis (Lauer et al., 2011) we applied the approach following Buylaert et al. (2009) to fluvial samples taken from Monheim-Hitdorf and Rheinberg (LRE) and measured the IRSL (50°C) signal and after the pIRIR signal at 225°C. Next, we tested the potential of first depleting the IRSL (50°C) signal and then detecting the K-feldspar OSL signal stimulated with yellow LED at 260°C (pIR-YOSL). We show that both, the pIRIR and the pIR-YOSL measurements yielded excellent results because the obtained ages are in agreement with available quartz OSL ages and also with the age control (Laacher See pumice).

#### 6.2 Tests concerning the bleaching behaviour

To obtain information on the light sensitivity of the pIR-YOSL signal, we conducted a bleaching test using a fluvial sample from the northern URG (sample Lum 1487, Viernheim core). Three aliquots of the material were exposed to daylight (cloudy day) for 1 min, 10 min, 1 hr and 5 hrs, respectively. Further material was bleached at daylight for 5 months. After this light exposure, remaining equivalent doses were measured using the pIR-YOSL dating approach as described in chapter 4. In Figure 6.1 those results and a comparison of the dose residuals of the IRSL (50°C) and the pIR-YOSL (260°C) signal are shown.

We demonstrate that the IRSL signal measured at 50°C is faster reset than the corresponding pIR-YOSL (260°C) signal. The originally measured equivalent dose value is 197 Gy for the IRSL and 232 Gy for the pIR-YOSL signal. After one minute of daylight exposure, > 90% of the natural IRSL related dose is depleted whereas only about 70 % of the pIR-YOSL corresponding dose is bleached down. After 10 minutes of daylight exposure, approximately 27 Gy of residual dose was measured for the pIR-YOSL signal (~ 12 % of the original natural dose). After one hour of light exposure the pIR-YOSL related dose is bleached down to > 90%, after 5 hrs to approximately 96 %.

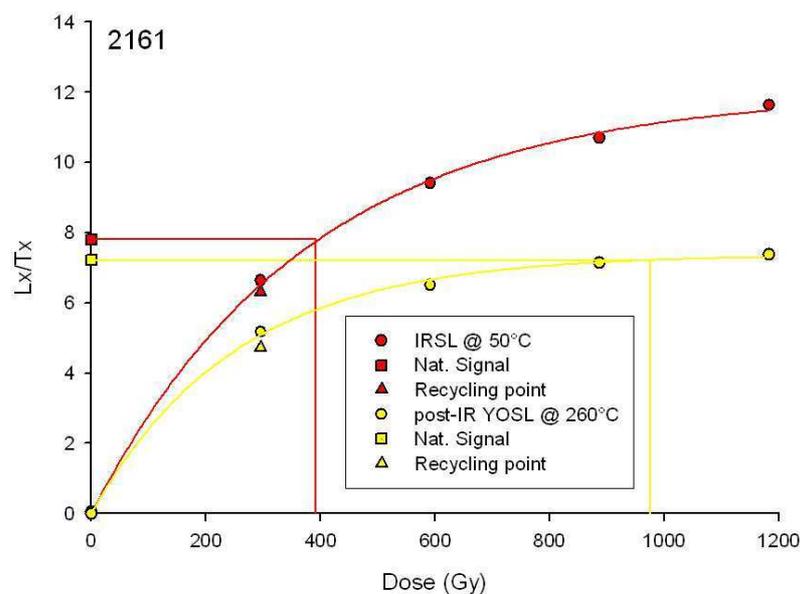


*Fig. 6.1: Bleaching behaviour of the IRSL and pIR-YOSL signal, tested on a fluvial sample derived from the Viernheim core. The pIR-YOSL signal is slightly less light sensitive than the IRSL signal (Lauer et al., submitted).*

The results indicate that it might be more problematic to use the pIR-YOSL approach for young, insufficient bleached samples as the signal is slightly less light sensitive than the IRSL (50°C) signal. However, for well bleached samples the protocol delivers reliable results as shown in chapter 4.

### 6.3 Signal stability

Concerning the stability of the pIR-YOSL signal we assume less fading for the pIR-YOSL signal (see chapter 4, Lauer et al., 2011). To gain further information on signal stability we measured the equivalent dose from a Tertiary fluvial sand (luminescence signal in saturation) sampled from the Ludwigshafen core (core number = P 34). The material (sample code = Lum 2161) was taken from below the Pliocene-Pleistocene boundary at 192 m core depth. The Pliocene-Pleistocene boundary was defined to be in between 170-180 m core depth at the drilling site (Weidenfeller and Knipping, 2008). The results are shown in Figure 6.2. When using the pIR-YOSL (260°C) signal, an equivalent dose of 950 Gy could be obtained. The IRSL signal yielded only 470 Gy. This is most likely due to higher fading of the IRSL (50°C) signal if compared to the pIR-YOSL signal.



*Fig. 6.2: Growth curve of the IRSL- and pIR-YOSL (260°C) signal obtained from a Tertiary fluvial sample taken from the Ludwigshafen core (URG). It is shown that the pIR-YOSL signal seems to be characterized by a higher stability (less fading) than the IRSL signal.*

### 6.4 Conclusion

The dating results shown in chapter 4 (Lauer et al., 2011) and this study underline that the pIR-YOSL approach has very good potential to date Quaternary sediments. Nevertheless,

it has to be taken into account that the pIR-YOSL signal is less light sensitive than the IRSL (50°C) signal and thus, the application for young, insufficiently bleached samples might be problematic. Dose residuals should become less relevant for older samples. The tests on signal stability showed that the pIR-YOSL signal seems to be characterized by a high stability and therefore the problem of anomalous fading becomes less relevant. As fading corrections are difficult for old samples with dose response curves in the non linear range (Huntley & Lamothe, 2001), the pIR-YOSL dating approach might provide a feasible alternative to the IRSL (50°C) dating approach.

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## Chapter 7

### Conclusion

#### 7.1 New insights into responses of the Rhine to tectonics and changes in climate

In this PhD thesis luminescence and infrared radiofluorescence (IR-RF) dating were applied to fluvial deposits which were collected from the Rhine river system.

For the fluvial successions at **Viernheim** (Heidelberg Basin, northern Upper Rhine Graben) a chronology based on optically stimulated luminescence dating on quartz was established up to a core depth of 33 m. By using a single aliquot regenerative (SAR) dose protocol (Murray and Wintle, 2000; 2003) it was shown that the upper fluvial units were all deposited during the Last Glacial (Weichselian) period. At 33 m depth the minimum age model-based quartz age is at  $56.2 \pm 6.3$  ka. When the quartz OSL signal was in saturation, feldspar was used for dating.

By using the IR-RF technique on potassium feldspar (Trautmann et al., 1998; Erfurt and Krbetschek, 2003) it was possible to establish a chronological framework for the fluvial deposits at Viernheim down to 100 meter core depth. The lowermost sample yielded an age of  $643 \pm 28$  ka (OIS 16). Further material was collected from the **Ludwigshafen** core, here the lowermost sample derived from 49.5 meters core depth and yielded an IR-RF age of  $566 \pm 32$  ka.

The new OSL and IR-RF ages from the Heidelberg Basin help to better understand the internal and external forcing on the Rhine fluvial dynamics. It was pointed out that in between the single fluvial units clear hiatuses occur. This discontinuity shows that periods of fluvial aggradation had been interrupted by erosion periods. The OSL age estimates of the upper part (33m) of the Viernheim core point to at least three aggradation periods during the Weichselian that occurred during periods of warming. The deposits could be preserved following tectonic subsidence creating accommodation space.

Between these upper fluvial units and the Upper Interlayer (a fine-grained and organic-rich layer correlating to a warmer climate period) the OSL and IR-RF ages show a clear chronological gap. The sample for IR-RF dating which was taken at 39 m core-depth (directly above the Upper Interlayer) yielded an age of  $288 \pm 19$  ka. The IR-RF age from sample VH-RF II, taken directly from this layer, is at  $302 \pm 19$  ka. This shows that Eemian sediments were not preserved at the drilling site but it could also be demonstrated that the Upper

Interlayer seems to be younger than Cromerian. A Cromerian age was before likely due to biostratigraphical evidence (e.g. Knipping, 2008; Weidenfeller and Knipping, 2008).

For the Middle Pleistocene Rhine sediments we can assume that differences in tectonics play the key role for the preservation of the fluvial archives. During times of increased subsidence the fluvial sands and gravel could be preserved and were not eroded again after deposition.

For the **Lower Rhine Embayment** (LRE) we mainly dated samples from the Lower Terrace (Klostermann, 1992). The new luminescence ages deliver a better chronological resolution for the studied sections and contribute to a better understanding on how fluvial sedimentation can occur. The ages obtained from the Monheim-Hitdorf section demonstrated that at least 8 m of fluvial sand and gravel were deposited within a very short time period, most likely during some single events. All taken samples from the section yielded equal ages. It was also of interest to show that the fluvial deposits hosting pumice from the Laacher See volcano can be much younger than the corresponding eruption (~ 12.9 ka, during Alleröd). At the Rheinberg section the OSL ages point for instance to an Early Holocene deposition age of the fluvial successions. This shows that the here intercalated Laacher See pumice was reworked for many times over a long time period.

## **7.2 Methodological aspects**

### **7.2.1 Statistical treatment of skewed equivalent dose distributions**

One often occurring problem with luminescence or IR-RF dating of fluvial deposits is an incomplete bleaching of the luminescence signal due to insufficient sunlight exposure during transport (e.g. Wallinga, 2002). Incomplete resetting of the OSL signal and solutions to deal with were addressed topics in this PhD thesis. For the sample set from the Viernheim and Ludwigshafen core the distribution of equivalent doses obtained from the quartz OSL and IR-RF measurements indicated that these fluvial sands have not been exposed to sunlight long enough during transportation. Thus, various statistical methods (e.g. central age model, minimum age model, values less than median) were applied to correct the quartz OSL and IR-RF ages. Nevertheless, for these samples no independent age control was available and the results based on the different age models could not be checked against any age control.

For samples collected from the Roman harbour at Cologne very good age control was available due to Roman artefacts. This gave us the possibility to apply various statistical

approaches to a skewed dose distribution and to compare the luminescence ages obtained from the different statistical methods with the known age. The results show that for the Roman harbour sample (sample SW-II) the age estimates which were calculated by using the statistical approach after Fuchs and Lang (2001) and Lepper and McKeever (2002) correspond best with the age control. Hence, these statistical approaches successfully corrected the equivalent dose values in that case.

### **7.2.2 Problem of feldspar contaminated quartz**

One other addressed topic was feldspar contamination in quartz. For some fluvial samples from the URG and the LRE the quartz OSL signal was disturbed by feldspar. Different approaches were tested to minimize the feldspar signal in quartz. For all these samples pulsed-OSL (POSL) helped to obtain a higher purity of the quartz signal. It was also pointed out that an IR-bleach at elevated temperatures (225°C) before measuring the quartz OSL has better potential to deplete the feldspar signal than an IRSL exposure at 50°C. But the 225°C IRSL pre-treatment might also deplete parts of the quartz signal (especially fast component) and this might be problematic for insufficient bleached samples. Therefore POSL should be the method of choice but as pulsing equipment is not always available the 225°C IRSL pre-treatment might provide an alternative, at least for well bleached sands.

### **7.2.3 Comparison of quartz and feldspar dating methods applied to samples with age control**

Different dating approaches were applied to samples from the LRE for which age control is available due to intercalated pumice from the Laacher See Volcano. The tephra gives us a maximum deposition age (< 12.9 ka, Boogard, 1995). For these samples a SAR protocol was applied on quartz. Additionally different feldspar dating protocols were applied. The results were then compared and checked against the age control. It could be demonstrated that the quartz OSL ages are in good agreement with the age control. But also the feldspar measurements yielded reliable results. We applied the approach described by Buylaert et al. (2009) and hence measured the IRSL (50°C) signal and the pIRIR (225°C) signal. Fading corrections were conducted after Huntley and Lamothe (2001). Next to this we tested for the

first time the potential of a yellow stimulated feldspar signal to date fluvial deposits. We first bleached the IRSL (50°C) signal and in the following we detected the pIR-YOSL signal measured at 260°C. These measurements yielded results fitting well with the quartz OSL ages and the age control. Hence, it could be demonstrated that the pIR-YOSL approach has good potential to date quaternary sediments. We also conducted test on the bleaching behaviour and stability of the pIR-YOSL signal. It was shown that the pIR-YOSL signal is more difficult to bleach than the IRSL 50°C signal. Hence, the application of the dating approach to young, insufficiently bleached samples might be problematic. Concerning the stability of the signal it could be demonstrated that the pIR-YOSL signal has a higher stability than the IRSL 50°C signal. All obtained equivalent doses using the pIR-YOSL signal were higher than the corresponding values of the IRSL 50°C signal and measurements on a fluvial sample in field saturation underlined this trend.

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## **Lebenslauf**

Der Lebenslauf ist in der Online-Version aus Gründen des Datenschutzes nicht enthalten

## Publication list

### *Peer-reviewed*

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