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INFRARED RADIOFLUORESCENCE (IR-RF) DATING OF MIDDLE PLEISTOCENE FLUVIAL ARCHIVES OF THE HEIDELBERG BASIN (SOUTHWEST GERMANY)

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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 ± 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 ± 19 ka and 302 ± 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.

Keywords: Infrared Radiofluorescence, Heidelberg Basin, Rhine System, Fluvial archives, Middle Pleistocene.

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1. INTRODUCTION

The fluvial and limnic-fluvial archives of the Upper Rhine Graben (URG) host important information about the fluvial history of the Rhine system and the palaeo-landscape development of southwest Germany. The thickest successions of unconsolidated Tertiary and Quaternary fluvial sediments can be found in the Heidelberg Basin, located in the northern URG.

This study is within the framework of Heidelberg Basin Drilling Project (Ellwanger *et al.*, 2005; Gabriel *et al.*, 2008) which intends to obtain detailed information on the structure of the Heidelberg Basin, on basin dynamics as well as on the influence of tectonics and climate forcings on sediment supply. The three drilling sites (**Fig. 1**) are located at Ludwigshafen-Parkinsel (cores P34 and P35, both with 300 m drilling depth), Viernheim (350 m drilling depth) and Heidelberg UniNord (cores Uni Nord I and II with 190 m and 500 m depth, respectively). The cores have been analysed by a multidisciplinary approach (Gabriel *et al.*, 2008) using methods such as palynology, paleontology, heavy mineral analyses, applied geophysics, sedimentology and palaeomagnetic studies. The facies structure of the sediments as well as palaeobotanical information and mineral components provide important information for the environmental conditions in the past. However, to deliver a chronological framework for the palaeolandscape- and environmental evolution, numerical dating of the sediments is mandatory. Luminescence and infrared radiofluorescence dating have the possibility to determine the time elapsed since the last exposure of mineral grains to daylight (Aitken, 1985,1998; Trautmann *et al.*, 1998; Krbetschek *et al.*, 2000) and hence the time of deposition. This information is very important to bring more light into the complex sedimentation cycles of the Rhine system.

A first coarse-grained quartz OSL chronology was recently provided by Lauer *et al.* (2010) for the Upper Pleistocene units of the Viernheim core. Based on this chronology it was possible to recognize three aggradation phases for the last glacial (Weichselian) period. The lowermost sample dated by Lauer *et al.* (2010) derived from 33 m core depth and yielded a quartz OSL age estimate of 56.2 ± 6.3 ka based on the minimum age model (MAM3) after Galbraith *et al.* (1999). If the mean value of all measured equivalent dose (D_e) values is used, it results in a maximum age of 94.2 ± 10.9 ka for this sample.

In a core depth of 39 m the quartz OSL signal was already in saturation. We therefore investigated further material, taken between 39 and 100 m core depth, by measuring the infrared radioluminescence signal of potassium feldspar for age determinations. The saturation dose of the IR-RF is at about 1200-1500 Gy (Erfurt and Krbetschek, 2003a). The method therefore enables to date much older sediments which helps us to further complete the chronological study for Quaternary aggradation periods of the Heidelberg Basin.

2. GEOLOGICAL SETTING

Due to the long term subsidence of the Upper Rhine Graben (URG) since the mid-Eocene, very thick Tertiary and Quaternary sediments have deposited and preserved (Bartz, 1974; Cloetingh *et al.*, 2005; Haimberger *et al.*, 2005; Hagedorn and Boenigk, 2008; Preusser, 2008). The source areas of the unconsolidated deposits can be found in the Alps and in the eastern and western Graben margins (for example Buntsandstein-Odenwald, see **Fig. 1**). The Quaternary deposits of the URG are characterized by different components of heavy minerals if compared to the Tertiary sediments and show a higher carbonate-content (Ellwanger *et al.*, 2005; Hoselmann, 2008). This can be explained with the connection of the Rhine to the Alpine source areas, which has happened during Pliocene (Kemna, 2005).

The thickest successions of Quaternary deposits should be found in the parts with the highest subsidence rates. In the Heidelberg Basin, Bartz (1974) expected successions of Quaternary deposits being more than 300 m thick. One of the aims of the Heidelberg Basin Drilling Project is to estimate the depth of the Pliocene-Pleistocene boundary and the first results of the project showed that Bartz (1974) even underestimated the thickness of the Quaternary in the northern Rhine Graben. Based on the results of some first pollen spectra, the coring at Heidelberg Uni Nord (basin depocentre) did not reach the Pliocene-Pleistocene boundary at a core depth of 500 m (Ellwanger *et al.*, 2008). For the Viernheim Drilling site the Pliocene-Pleistocene boundary was found at 225 m depth (Hoselmann, 2008) and for the

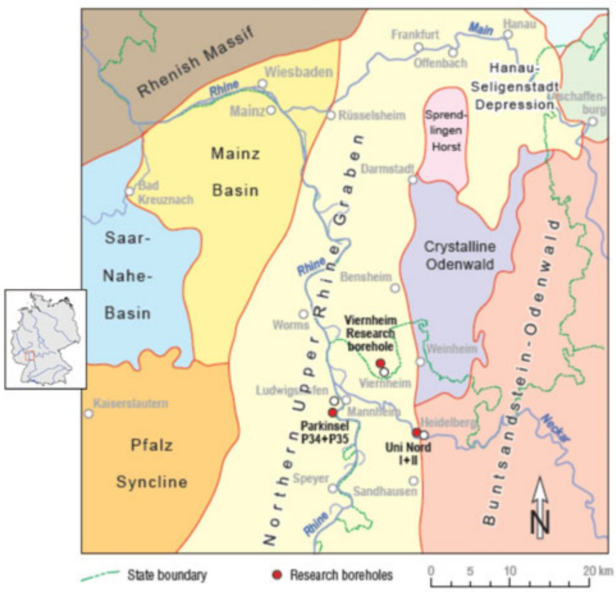

Fig. 1. *Study area showing the three drilling sites within the Heidelberg Basin Drilling Project at Heidelberg/Uni Nord, Ludwigshafen Parkinsel and Viernheim (after Gabriel et al., 2008).*

Ludwigshafen drilling sites (P34 and P35) it was found at between 170 and 180 and at 220 m depth, respectively (Weidenfeller and Knipping, 2008).

From the Ludwigshafen core (P34) the results of the palaeomagnetic study are available (Rolf *et al.* 2008). The upper 70 m of sediments show a normal polarity indicating that these sediments correlate with the Brunhes chron. A larger scatter in inclination with reverse and normal polarities within the sediments below 70 m core depth gives evidence that these sediments had been deposited before the Matuyama/Brunhes polarity change (780 ka).

As mentioned above, Lauer *et al.* (2010) investigated the upper sediment records of the Viernheim core up to 33 m by OSL dating. The results showed that these fluvial sediments had been deposited during the Weichselian and were accumulated in different aggradation periods interrupted by phases of fluvial erosion consequently leading to clear chronological gaps between the different units. It can be assumed that the interplay between deposition and erosion is controlled by tectonic subsidence which provides a space for the sedimentary input and by climatic variations which determine the transport dynamics of the Rhine system. Lauer *et al.* (2010) found that the Weichselian aggradation periods correlate with phases of increasing temperatures (cold to warm transitions) inducing an increase of sediment supply.

Between these coarser-grained upper fluvial units and the finer-grained and organic rich Upper Interlayer ("Oberer Zwischenhorizont"), a clear hiatus was expected. Knipping (2008) investigated pollen assemblages from this layer, and concluded that this layer cannot be correlated with the Eemian interglacial. Following the pollen contents as well as the molluscs identification (Rähle, 2005; Wedel, 2008), a Cromerian age was suggested for the Upper Interlayer. Absolute dating was however necessary to confirm this assumption and provide a more precise chronological framework.

3. PRINCIPLES OF LUMINESCENCE AND INFRARED RADIOFLUORESCENCE DATING

The methods of luminescence and infrared radiofluorescence dating are based on similar physical processes because both use mineral grains as natural dosimeters. In this section, we describe the basic background of these methods.

Minerals like quartz or potassium feldspars can be used as natural dosimeters. Following the energy band model electrons are lifted from the valence band (VB) up to the higher energetic conduction band (CB) while the minerals are exposed to ionizing radiation (Aitken, 1985). The electrons either recombine directly with connected holes or are trapped in defect structures (traps) located below the CB. The number of occupied traps increases with burial time. The electrons are detrapped again when the mineral is exposed to heat or light (bleaching or zeroing). During the recombination, the emission of photons occurs (= luminescence).

The datable age range for optically stimulated luminescence (OSL) is thought to be restricted by the quantity of luminescence centres. The OSL signal of quartz reaches to saturation at about 300 Gy (Wallinga, 2002) which covers an age range up to about 150 ka if the dose rate is at ~ 2 Gy/ka. Feldspar saturates at much higher doses and hence, the mineral can be used to date much older sediments by infrared stimulated luminescence (IRSL) dating.

One disadvantage of applying IRSL to potassium feldspar is the phenomenon of anomalous fading (Wintle, 1973; Vasil´chenko *et al.*, 2005). The fading rate has to be quantified (Huntley and Lamothe, 2001; Kars *et al.*, 2008) to avoid an age underestimation but this is still problematic especially for older samples showing non-linear dose response curves.

Infrared Radiofluorescence (IR-RF, also knows as infrared radioluminescence) provides an alternative possibility to date K-feldspar (Trautmann *et al.*, 1999b). Various studies showed that this method is a powerful tool to date sediments from the last glacial-interglacial cycles (Krbetschek *et al.*, 2008). The IR-RF is based on the emission of fluorescent light in the wavelength range of 865nm (Erfurt and Krbetschek, 2003a) occurring during the irradiation of the K-feldspar grains (Trautmann *et al.*, 1998). The emission is linked to the process of electron trapping (**Fig. 2**) and derives most likely from electron transitions in Pb^+ centres (Erfurt, 2003).

The intensity of the IR-RF signal correlates with the amount of free optically active traps. The process is therefore independent from recombination phenomena (Krbetschek *et al.*, 2000) and the density of free traps can be measured. The radioluminescence signal decreases

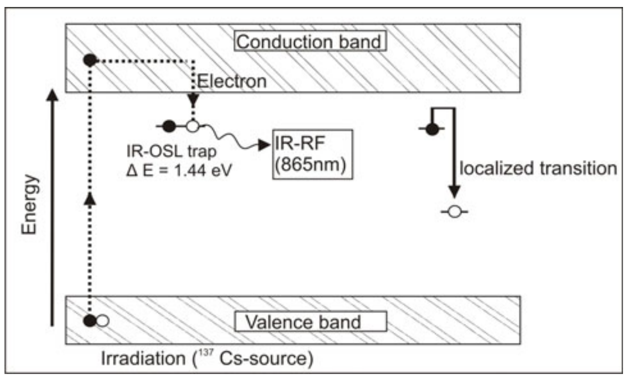

Fig. 2. *Two term energy band model explaining the principle of IR-RF (modified after Erfurt and Krbetschek, 2003a). The characteristic fluorescence light emission at 865 nm is linked to an electron transition into the IR-OSL trap (trapping). The IR-RF is therefore independent from recombination centres. 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).*

with irradiation time whereas it is growing when the material is exposed to daylight. For old samples which are closed to saturation, the detectable natural IR-RF signal is therefore dim (contrary to the OSL signal). Trautmann *et al.* (2000) found out that the physical process for the IR-RF, regarding the interaction of free electrons and electron traps, are different from the kinetic properties for the IRSL. Whereas the IRSL signal can be bleached by IR-light exposure (880nm), the IR-RF signal is only increased by exposing the sample material to higher energy light (< 500 nm; > 2.5 eV). To explain this phenomenon Trautman *et al.* (2000) introduced a modified energy level model assuming a localised transition of electrons during IR-light exposure (see also Templer, 1986). Following this model, the electrons are only exited into a higher energetic level during IR-stimulation but not lifted into the CB. However, the IR-RF emission should derive from the electron transition via the CB into the IR-RF trap.

4. SAMPLING

Five samples for IR-RF dating were taken from the Viernheim core (samples VH-RF I – V) and further three samples from the Ludwigshafen core (samples LH-RF I - III). The sampling positions are marked in **Fig. 3** and a more detailed description of the sampled fluvial units of the Viernheim core (Hoselmann, 2008) is provided in **Table 1**.

From the Viernheim core material for IR-RF dating was taken out of sandy layers in between ~ 39.5 m to ~ 100.5 m core depth with the intention to obtain the chronological framework for the Upper Interlayer and also for the fluvial units below.

The finer-grained Upper Interlayer is characterized by high contents of organic material (Hoselmann, 2008, see also **Table 1** and **Fig. 4**) and should correlate with a warmer climate period. So far, there were no absolute dating results available for this layer. Therefore one sample (VH-RF I) was taken from directly above the finer-grained and organic-rich Upper Interlayer (**Fig. 4**) and one other sample (VH-RF II) from the layer (unit Xb) itself. Samples VH-RF III and VH-RF IV were taken from the coarse-grained unit below the Upper Interlayer (unit Xa) and sample VH-RF V from the lower part of unit IXa2 ranging from 77 m – 101.5 m core depth (see **Fig. 3**).

Samples from the Ludwigshafen core (P34) were all taken from sediments below the Upper Interlayer in between ~ 40.5 m-49.5 m core depth with the aim to figure out if a correlation of the fluvial units from below the Upper Interlayer is possible between the Viernheim and the Ludwigshafen cores.

5. SAMPLE PREPARATION

The radioluminescence signal is emitted from K-feldspars (particularly orthoclase and microcline) and it is therefore important to obtain a high purity of the K-feldspar extracts for measurements. For mineral preparation the sediment was first dried and sieved to isolate the 100-300 µm grain size fraction. Organic matter was removed by H_2O_2 treatment. The quartz and heavy mineral fractions were isolated from the feldspars by flotation (Miallier *et al.*, 1983) followed by a separation of the potassium feldspar fraction from other feldspars using sodium polytungstate (2.58 g/cm^3). The K-feldspars were etched using 10% HF for 40 min and 10% HCL for 30 min to remove the alpha ray-effected outer rim of the grains. After the etching procedure, the material was resieved to separate the < 90 µm, 90-160 µm and > 160 µm grain size fraction. For measurements either the 90-160 µm or the > 160 µm fraction was used.

To check the purity of the K-feldspar extracts, the material was analysed using a raster electron microscope. For all analysed samples a K-feldspar content of > 90% was obtained. This demonstrates that the flotation technique is a powerful tool for obtaining high quality of feldspar extracts. For sample VH-RF I the mineral separation was first done by just using sodium polytungstate solutions (2.58, 2.62 and 2.70 g/cm^3) without flotation. For this sample the percentage of feldspar grains was at only ~ 80%.

6. DOSE RATE

External dose rate

The external dose rate was quantified by measuring the contents of the radionuclides uranium and thorium and potassium (^{40}K) which are present within the natural sediment. For measurements 50 g of the material was taken from each sample. The material was first dried and then stored for four weeks to avoid any radon specific disequilibrium. Gamma spectrometry was then carried out on a high purity germanium gamma detector. To obtain the concentrations of uranium, thorium and potassium the activities of ^{238}U, ^{232}Th and ^{40}K were measured (Aitken, 1998).

The water content was estimated to be at 18 ± 6%. This moisture content was calculated by Lauer *et al.* (2010) for the upper fluvial units of the Viernheim core and it is based on the assumption that the unconsolidated sediments were water saturated during almost the whole burial period. Due to the fact that there is no big difference in grain sizes between the layers sampled for IR-RF dating and the fluvial sands that were dated by Lauer *et al.* (2010), the same water content was used.

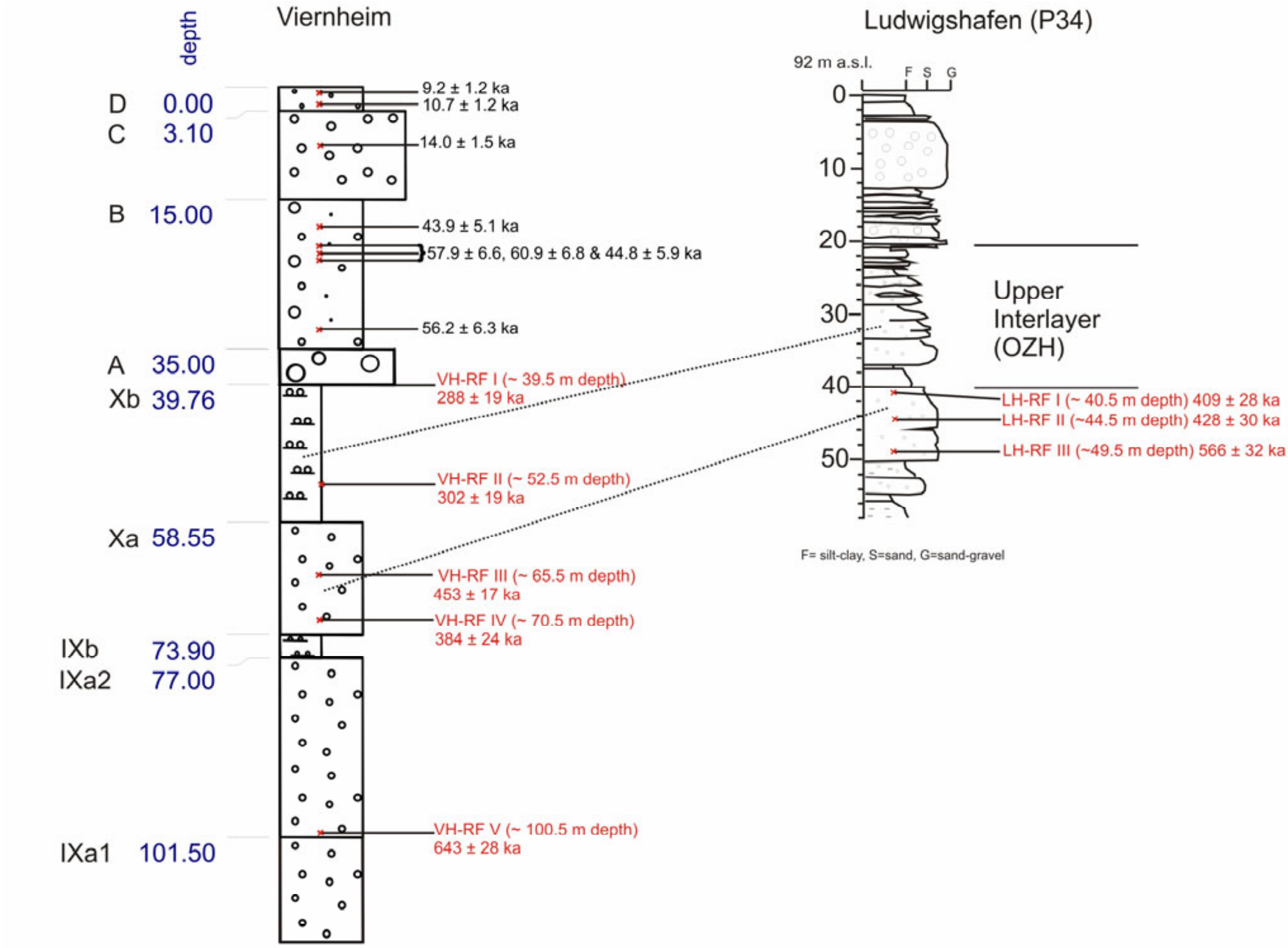

Fig. 3. *Viernheim core (left side) and Ludwigshafen core (core P34, right side) modified after Hoselmann (2008) and Rolf et al. (2008), respectively. The IR-RF age estimates are inserted into the graph. The quartz OSL dating results from Lauer et al. (2010) are also inserted into the Viernheim core. The dotted lines mark core sections that can most likely be correlated.*

Table 1. *Legend to sedimentological units of the Viernheim core after Hoselmann (2008).*

Unit	Depth in m below ground level	Description summary	Special features
D	0 – 3.1	Markedly coarse, pedogenically overprinted aeolian sand	0-1 m core loss
C	3.1 – 15	Gravely sands and gravels with fining-upward and coarsening-upward sequences, Rhenish portion strongly reduced	Neckar gravels
B	15 – 35	Four fining-upward sequences, inhomogeneous, 27-32 m gravel	With Rhenish Facies in the fine grained parts, Neckar gravel
A	35 – 39.76	Xb is discordantly overlain by gravely sands to gravels in three fining upward sequences	With Rhenish Facies in the fine grained parts, Neckar gravels
Xb	39.76 – 58.55	Dominantly fine-clastic with sandy intercalations, partially in Rhenish Facies, at 49 and 53.5 m strongly humic, five partial sequences in total, humic clays and peat in the upper section	51.9-52.5 m in typical Rhenish Facies
Xa	58.55 – 73.9	The cycle begins with sand and some gravel, coarsening upwards, from 67-69 m reddish because of the stronger Neckar influence, then fluctuating fine gravel portions	Concretionary in part due to calcareous content
IXb	73.9 – 77	Fine clastic sediments, reduced at the bottom, then humic up to 75 m, followed by strong humic peat and finally by silt	70-80 m, 87.2-88.2 and 101,8-103 m in typical Rhenish Facies
IXa2	77 – 101.5	Marked change in colour from grey to more reddish colours up to 98 m Coarsening-upward sequence, from 88.2 m again sediments in Rhenish Facies, becoming more fine-grained upwards.	Notable colour change IXa2 to IXa1. IXa2 with strong influence from Neckar sediments?
IXa1	101.5 – 122.09	VIIIb is discordantly overlain by sandy zone with subordinate interbedded gravely horizons	

Internal potassium content

The internal potassium content of the K-feldspars is an important factor because the internal radioactive decay of potassium contributes to a significant part to the ionisation. For the internal dose rate a potassium content of 12.5 ± 0.5% is often used (Huntley and Baril, 1997). But it also has to be taken into account that this value can vary among feldspar types derived from different source areas. To estimate the internal potassium content more accurately, the concentrations of different elements (including K) were measured for all samples from the Viernheim core by electron probe microanalysis. The internal potassium concentrations were measured for 36 to 39 feldspar grains from each sample. For all samples the internal potassium content was calculated by using the mean of all measured internal K-contents from a sample. The error was calculated by the standard error on the mean. For the samples from the Ludwigshafen core (LH-RF I – III) the internal potassium value was estimated by taking the mean of all five values estimated for the Viernheim samples. The values are listed in **Table 2**.

Fig. 4. *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 contend of organic material. Sample VH-RF I was taken directly on top of the Upper Interlayer as marked by the circle.*

7. INFRARED RADIOFLUORESCENCE DATING PROCEDURE

Measurements were conducted on an automated multi-spectral radioluminesence (RL) instrument, equipped with ten ^{137}Cs sources (Erfurt *et al.*, 2003). For measurements ten aliquots (diameter = 5 mm) were used for each sample. The number of measured aliquots was restricted due to the extended length of measurement time. To obtain the D_e values, a single-aliquot regenerative-dose dating protocol (IRSAR) was applied (Erfurt and Krbetschek, 2003a). Following the IRSAR protocol, the IR-RF intensity of the natural dose was first measured. After this, the samples were bleached by an mercury lamp. After the relaxation of the phosphorescence which occurs during the bleaching, the natural IR-RF signal was regenerated. The IR-RF was then detected continuously during the irradiation and a few hundreds up to > 1000 data points were collected. Finally the data points were fitted to a single stretched exponential function (Erfurt and Krbetschek, 2003b) and the natural IR-RF signal was interpolated into the curve to obtain D_e (**Fig. 5**).

Table 2. *Overview about dosimetry and IR-RF ages: The nuclide concentrations of ^{40}K, Th and U within the sampled material as well as the internal potassium content of feldspar samples define the total dose rate.*

Sample	Depth (m)	% K	ppm Th	ppm U	Internal K%	Total dose rate (Gy/ka)	ED (Gy)	Age (ka)
VH-RF I	39.36 – 39.43	1.59 ± 0.03	5.21 ± 0.08	1.59 ± 0.03	13.36 ± 0.08	2.54 ± 0.19	732 ± 27	288 ± 19
VH-RF II	52.39 – 52.47	1.43 ± 0.02	8.27 ± 0.07	2.37 ± 0.03	13.15 ± 0.13	2.75 ± 0.19	829 ± 22	302 ± 19
VH-RF III	65.37 – 65.47	1.50 ± 0.02	3.00 ± 0.07	0.85 ± 0.03	12.77 ± 0.12	2.16 ± 0.12	977 ± 38	453 ± 17
VH-RF IV	70.43 – 70.52	1.45 ± 0.03	2.85 ± 0.09	1.01 ± 0.05	13.27 ± 0.08	2.16 ± 0.15	828 ± 23	384 ± 24
VH-RF V	100.31 – 100.39	1.45 ± 0.02	3.16 ± 0.07	1.05 ± 0.04	12.66 ± 0.11	2.22 ± 0.12	1425 ± 43	643 ± 28
LH-RF I	40.44 – 40.55	1.21 ± 0.02	3.39 ± 0.06	1.12 ± 0.03	13.04 ± 0.14	2.23 ± 0.17	911 ± 31	409 ± 28
LH-RF II	44.50 – 44.70	1.27 ± 0.02	3.22 ± 0.06	1.08 ± 0.03	13.04 ± 0.14	2.26 ± 0.18	966 ± 37	428 ± 30
LH-RF III	49.40 – 49.56	1.24 ± 0.01	2.99 ± 0.03	0.91 ± 0.01	13.04 ± 0.14	2.55 ± 0.18	1442 ± 63	566 ± 32

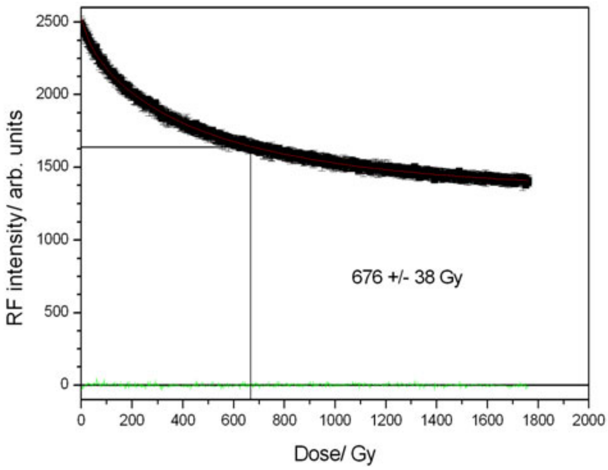

Fig. 5. *Response of the IR-RF signal to irradiation (sample VH-RF I). The signal is decreasing with irradiation time. A stretched exponential function was fitted to the IR-RF data (red line). The green line marks the residual of the IR-RF dose curve.*

8. RESULTS

Dose distribution and statistical treatment

Fig. 6 shows representative D_e distributions from samples VH-RF II and VH-RF III. The scattering among equivalent doses can be explained by a methodological error and incomplete bleaching of some of the grains due to insufficient light exposure during transportation (Arnold *et al.*, 2007; Jain *et al.*, 2004; Rodnight, 2008). The latter has to be regarded as very likely as it is known that the IR-RF signal is less light sensitive than the IRSL signal. Trautmann *et al.* (1999a) tested the bleaching behaviour of the IR-RF signal and found out that 2-3 hours of daylight exposure are needed for the radioluminescence signal to reach to the maximum level. Furthermore, Lauer *et al.* (2010) showed skewed D_e distributions

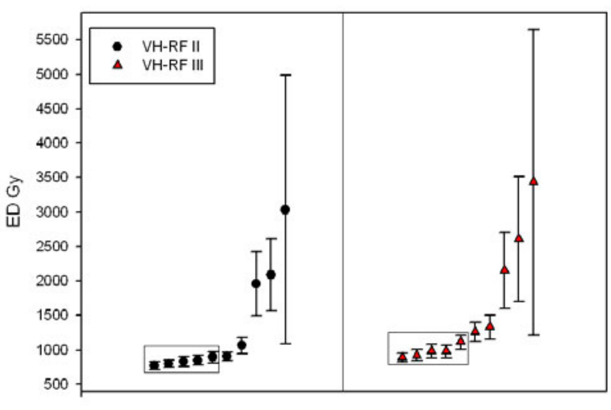

Fig. 6. *Distributions of measured equivalent doses from samples VH-RF II and VH-RF III. The values inside the back boxes were included for age calculations following the statistical method described in chapter 8.*

from quartz OSL measurements for the samples derived from the upper fluvial units of the Viernheim core. Hence, incomplete bleaching is very likely for the investigated samples and statistical treatment of the data is required.

Due to the small number of available D_e values (10 aliquotes), the application of statistical models like the minimum age model (Galbraith *et al.*, 1999) or the leading edge method after Lepper and McKeever (2002) was not possible. Therefore we decided to apply a simple statistical treatment to exclude outliers among D_e values in the high range. For age calculations, all D_e values being equal or less than the median D_e values were included. The mean value of these integrated D_e values was finally taken for age calculations and the D_e error was defined by the standard error on the mean.

IR-RF ages and stratigraphic interpretation

The IR-RF ages of the Viernheim core and Ludwigshafen core are shown in **Figs**. **3**, **7** and **8** and **Table 2**. Based on these results four main Middle Pleistocene periods of aggradation have been recognized:

- The IR-RF ages from samples VH-RF I (288 ± 19 ka) and VH-RF II (302 ± 19 ka) both point to a deposition of the Upper Interlayer during an early Saalian stage (OIS 9-8). The discrepancy between these ages and so far existing biostratigraphy based on pollen data and molluscs is discussed in chapter 9.
- A second aggradation period is indicated by the IR-RF ages from samples LH-RF I and LH-RF II (Ludwigshafen site), and sample VH-RF IV (Viernheim site). All samples give a statistically equal IR-RF age (409 ± 28 ka, 428 ± 30 ka and 384 ± 24 ka, respectively) and demonstrate that Upper Elsterian (OIS 12-11) deposits are preserved at both drilling sites. The IR-RF age of sample VH-RF III (453 ± 17 ka) was not integrated for discussion as it is very likely overestimating the true age maybe due to incomplete bleaching.
- A few metres below LH-RF II, the sample LH-RF III yields a Lower Elsterian age of 566 ± 32 ka (OIS 15).
- Finally the sample VH-RF V yields a Cromerian age (OIS 17-16, 643 ± 28 ka, ~ 100 m core depth).

These results clearly demonstrate discontinuities within the sedimentation. The main stratigraphical hiatus obviously concerns the Eemian and Upper Saalian, before a recent aggradational period takes place during the Middle Weichselian (**Fig. 3**, Lauer *et al.*, 2010).

The age difference between samples LH-RF II (428 ± 30) and LH-RF III (573 ± 37) also suggests some chronological gap. This gives evidence for a period of sediment erosion during the Lower Elsterian period (most likely OIS 14-13).

Due to the Cromerian age of sample VH-RF V another hiatus may be found with the oldest sediments dated at the Viernheim drilling site. However this hiatus may only be due to the lack of dating as 30 m of sediments separates the samples VH-RF IV and VH-RF V.

9. DISCUSSION AND CONCLUSION

A chronology for Middle Pleistocene fluvial deposits from the northern Upper Rhine Graben (Heidelberg Basin) was established using IR-RF dating. It has to be mentioned that only ten aliquots were measured for each sample and the obtained D_e distributions showed a clear scattering but the values were concentrated in the lower range. Therefore the application of a simple statistical method was mandatory to exclude the higher outliers and to obtain age estimates without clearly overestimating the true age. Based on this procedure it was possible to establish a chronology for Middle Pleistocene fluvial sediment units up to ca 650 ka.

Chronology of the Upper Interlayer

The IR-RF samples which were taken at the Viernheim drilling site to frame the sedimentation age of the Upper Interlayer (VH-RF I and VH-RF II) are in good agreement to each other, yielding an age of ~ 300 ka (OIS 9-8). Sample VH-RF I, taken directly on top of the Upper Interlayer is at 288 ± 19 ka and the IR-RF age from sample VH-RF II, taken directly from this layer, is at 302 ± 19 ka. A Holsteinian age for this layer should be very unlikely, because until now in the Upper Interlayer, no pollen of *Pterocarya* could be found and *Buxus* is only rare (personal communication, Maria Knipping). Based on the IR-RF ages we now can assume a deposition during an early Saalian stage for this layer. This means that there is a discrepancy between the interpretation of the available biostratigraphical information based on pollen and molluscs (Rähle, 2005; Knipping, 2008; Weidenfeller and Knipping, 2008; Wedel, 2008) and the IR-RF age estimates. Following pollen contents a Cromerian age (older than OIS 15 if following **Fig. 7**) of the Upper Interlayer could be assumed as it contains pollen markers from different interglacials (Knipping, 2008) and also the found mollusc fauna (Rähle, 2005; Wedel, 2008) would agree with this interpretation.

On the other hand, a clear age underestimation of the IR-RF ages is very unlikely as the radioluminescence signal is far away from saturation for those samples and it is known that the signal is characterized by a high stability what is proven by physical experiments (Erfurt *et al.*, 2000; Trautmann, 2000; Erfurt and Krbetschek, 2003b). If the Upper Interlayer would be Cromerian, the IR-RF ages would underestimate the true age by almost 300 ka. This can be excluded due to the described physical characteristics of the IR-RF signal. Furthermore both ages (samples VH-RF I and VH-RF II) point to the same deposition age (OIS 9-8) and if these ages would underestimate the true age that much, also all other here presented IR-RF ages should be affected by the same problem with even increasing tendency for the older samples. This would for example mean that sample LH-RF III (49.5 m core depth), which yields an IR-RF age of 566 ± 32 ka (OIS 15), would date to below the Brunhes-Matuyama boundary (780 ka). But as mentioned in chapter 2, the upper 70 m of sediments show a normal polarity for the Ludwigshafen core P34 (Rolf *et al.*, 2008) strongly supporting the assumption that such an age un-

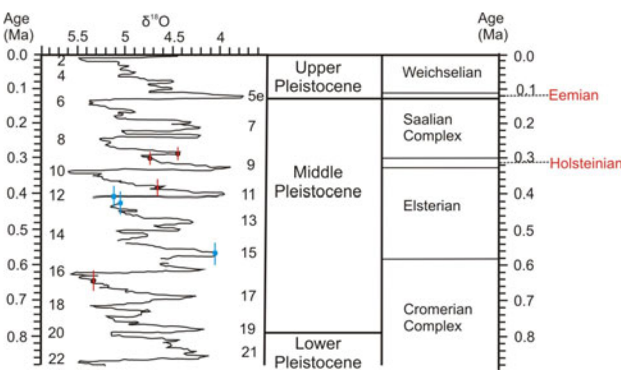

Fig. 7. *Marine Isotope record with plotted IR-RF ages from samples VH-RF I, II, IV & V (red dots) and samples LH-RF I-III (blue squares). The IR-RF age from sample VH-RF III was not inserted because it is most likely overestimating the true age. The Isotope record graph was taken out of Litt (2007) 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).*
It is important to underline that the discussions on the chronology of the dated fluvial units follow this chronostratigraphical tables but there are different interpretations in literature about the chronological framework of the Cromerian.
Following the here used tables, the Cromerian ends at around 580 ka but Bittmann & Müller (1996) correlate the youngest warmer stage of the Cromerian with the so called Kärlich-Interglacial (see also Litt et al., 2007) which was dated using Ar/Ar method to ~ 400 ka (Van den Boogard et al., 1989).

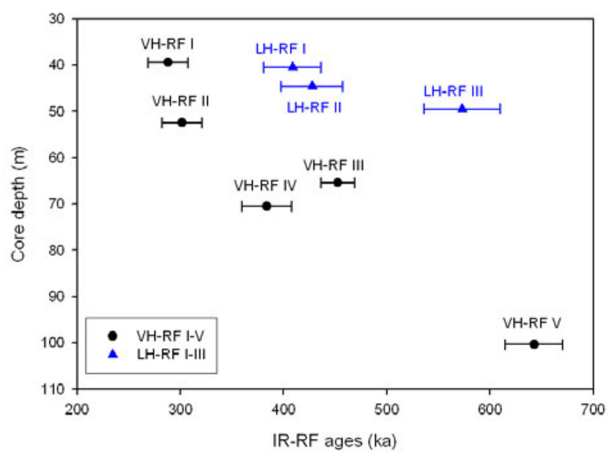

Fig. 8. *IR-RF ages plotted against the core depth.*

derestimation of the IR-RF ages is not the case and that the here presented IR-RF data can be regarded as valid. Therefore the question about the chronology of the Upper Interlayer needs further discussions in the future, but following the IR-RF ages a Cromerian deposition age is now very unlikely.

Control mechanisms for fluvial dynamics

The new chronology can be used for a better understanding of the internal and external forcing on the Rhine fluvial dynamics. When discussing the changes in fluvial dynamics of a huge river system like the Rhine, control mechanisms like sea-level change, climate variations and tectonic activity should be regarded as major forces (Miall, 1996; Roe, 1999; Blum and Törnqvist, 2000; Peters and Van Balen, 2007; Ziegler and Fraefel, 2009). In the case of the Heidelberg Basin, Middle Pleistocene changes in sea level of the North Sea should be regarded as little relevant as the Heidelberg Basin is located relatively upstream. Its fluvial dynamics should hence be driven by tectonic and climate forcing.

The climatic factor can not be neglected as it controls the intensity of fluvial discharge, influences weathering rates and has therefore strong influence on the amount of sediment load. Nevertheless, following the IR-RF ages, the influence of climate cycles is difficult to establish due to the error of the age estimates. Most of the samples may be allocated to cold, warm, or transitional periods. This makes it difficult to discuss this issue but the role of climate was assessed by Lauer *et al.* (2010) who demonstrated for the younger deposits that fluvial accumulation occurred during the Weichselian periods of warming.

However, it is very likely that for the Middle Pleistocene differences in tectonics play the key role for the preservation of the Rhine fluvial archives. The importance of fault tectonics and variations in subsidence was already demonstrated by Weidenfeller and Kärcher (2008) for the formation of the "Frankenthaler Terrasse" (north-western URG). For the now studied archives near Viernheim and at Ludwigshafen the intensity of subsidence of the Heidelberg Basin should be regarded as the main force controlling the aggradation and preservation of fluvial deposits. Therefore the IR-RF ages can help us to better reconstruct the tectonic history of the Heidelberg Basin. This can be done by having a look on the various mean aggradation rates derived from the chronological data, but when discussing sedimentation rates we have to keep in mind that high resolution dating was not possible and only tendencies for sediment supply in space and time can be deduced. Nevertheless, for the Viernheim core we now can assume an aggradation rate of ~ 10 to 14 m/100 ka in between 100 and 70 m core depth (time period: 643 ± 28 ka – 384 ± 24 ka // OIS 17/16 – OIS 12/11). In between 70 m and 40 m core depth (time period: 384 ± 24 ka – 288 ± 19 ka // OIS 12/11 – OIS 9/8) we can assume a much higher about aggradation rate of 22 to 40 m/100 ka. It can be assumed that during phases of increased subsidence, aggradation space was created for incoming sediments and relatively complete successions of fluvial archives could be preserved. During phases of only minor or without subsidence, the probability of sediment preservation decreased and climatic controlled erosion consequently caused hiatuses being present in between the fluvial units.

The IR-RF age estimates clearly demonstrate the complexity of the fluvial archives preserved at the Heidelberg Basin which might be explained by small scaled differences in tectonics. It is shown that the successions of Middle Pleistocene fluvial and limnic fluvial sediments are more completely preserved at the Viernheim site than at the Ludwigshafen site (**Fig. 8**). At Viernheim, the deposits at ~ 52.5 m core depth yield an IR-RF age of 302 ± 19 ka whereas the deposits taken from ~ 40.5 m core depth at the Ludwigshafen site yielded an IR-RF age of already 409 ± 28 ka.

It is however essential to keep in mind that the fluvial sequence recorded in the URG results from numerous aggradational and erosional periods. It is hence very likely that the Eemian (OIS 5e) and Upper Saalian (OIS 7-6) sediments had been deposited and were eroded afterwards. In particular the fine-grained Eemian sediments should have been eroded during Weichselian, most likely due to a tectonic impulse but also an increase in fluvial dynamics caused by climate change should have contributed to the erosion of those sediments. One explanation for the absence of Upper Saalian deposits might also be that there was only limited aggradation during this time. During the penultimate glaciation (Saalian or Riss), the glacier extend reached its maximum in the Alpine foreland and also mountains like the Black Forest were covered with widespread ice sheets (Fiebig *et al.*, 2004). This means that huge amounts of water were bound what might have significantly reduced the fluvial discharge and sediment supply towards the Heidelberg Basin at least during cold seasons. But during melting period, the sediment supply was surely reactivated and hence this cannot be the only reason for the absence of Upper Saalian deposits at the drilling sites.

One further reason for the lack of Upper Saalian fluvial deposits at the drilling sites might be explained by the migration of the Rhine in the wide floodplain of the Rhine Graben. Upper Saalian deposits are not preserved at the drilling sites but could possibly be found at other locations within the northern Rhine Graben due to the palaeo-flow regime of the Rhine. The latter was surely significantly influenced by tectonic movements and it was demonstrated by Hagedorn and Boenigk (2008) for the western Graben area that the Rhine was temporarily absent during Quaternary, most likely during times of increased subsidence of the eastern Graben site (Heidelberg Basin). In return it can be assumed that during times of reduced subsidence of the Heidelberg Basin, the Rhine temporary shifted towards the western Rhine Graben.

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REFERENCES

Aitken MJ, 1985. *Thermoluminescence dating*. London, UK Academic Press: 359pp.

Aitken MJ, 1998. *Introduction to Optical Dating*. Oxford, Oxford University Press: 280pp.

Arnold LJ, Bailey RM and Tucker GE, 2007. Statistical treatment of fluvial dose distributions from southern Colorado arroyo deposits. *Quaternary Geochronology* 2(1-4): 162-167, DOI 10.1016/j.quageo.2006.05.003.

Bartz J, 1974. Die Mächtigkeit des Quartärs im Oberrheingraben. In: Illies JH and Fuchs K, eds., *Approaches to Taphrogenesis* vol. 8. Inter-Union-Commission on Geodynamics Scientific Report, Stuttgart, Schweitzerbart: 78-87.

Bittmann F and Müller H, 1996. The Kärlich Interglacial site and its correlation with the Bilshausen sequence. In: Turner C, ed., *The early middle Pleistocene in Europe*. Balkema: 187-193.

Blum MD and Tornqvist TE, 2000. Fluvial responses to climate and sea-level change: a review and look forward. *Sedimentology* 47(Suppl. 1): 2-48, DOI 10.1046/j.1365-3091.2000.00008.x.

Cloetingh S, Ziegler PA, Beekman F, Andriessen PAM, Matenco L, Bada G, Garcia-Castellanos D, Hardebol N, De´ zes P and Sokoutis D, 2005. Lithospheric memory, state of stress and rheology: neotectonic controls on Europe's intraplate continental topography. *Quaternary Science Reviews* 24(3-4): 241-304, DOI 10.1016/j.quascirev.2004.06.015.

Ellwanger D, Gabriel G, Hoselmann C, Lämmermann-Barthel J and Weidenfeller M, 2005. The Heidelberg drilling project (Upper Rhine Graben, Germany). *Quaternaire* 16: 191–199.

Ellwanger D, Gabriel G, Simon T, Wielandt-Schuster U, Greiling RO, Hagedorn EM, Hahne J and Heinz J, 2008. Long sequence of Quaternary Rocks in the Heidelberg Basin Depocentre. *Quaternary Science Journal (Eiszeitalter und Gegenwart)* 57: 316-337.

Erfurt G, Krbetschek MR, Trautmann T, W. Stolz W, 2000. Radioluminescence (RL) behaviour of Al_2O_3:C-potential for dosimetric applications. *Radiation Measurements* 32(5-6): 735-739, DOI 10.1016/S1350-4487(00)00052-4.

Erfurt G, 2003. Infrared luminescence of Pb^+ centres in potassium-rich feldspar. *Physica status solidi (a)* 200(2): 429-438.

Erfurt G and Krbetschek MR, 2003a. IRSAR - A single-aliquot regenerative-dose dating pro-tocol applied to the infrared radiofluorescence (IR-RF) of coarse-grain K feldspar. *Ancient TL* 21: 21-28.

Erfurt G and Krbetschek MR, 2003b. Studies on the physics of the infrared radioluminescence of potassic feldspar and on the methodology of its application to sediment dating. *Radiation Measurements* 37(4-5): 505-510, DOI 10.1016/S1350-4487(03)00058-1.

Erfurt G, Krbetschek MR, Bortolot VJ and Preusser F, 2003. A fully automated multi-spectral radioluminescence reading system for geochronometry and dosimetry. *Nuclear Instruments and Methods in Physics Research Section B* 207(4): 487-499, DOI 10.1016/S0168-583X(03)01121-2.

Fiebig M, Buiter SJH and Ellwanger D, 2004. Pleistocene glaciations of South Germany. In: Rose J (ed.), *Developments in Quaternary Science 2*, Amsterdam, Elsevier: 147-154.

Gabriel G, Ellwanger D, Hoselmann C and Weidenfeller M, 2008. The Heidelberg Basin Drilling Project. *Quaternary Science Journal (Eiszeitalter und Gegenwart)* 57: 253-260.

Galbraith RF, Roberts RG, Laslett GM, Yoshida H and Olley JM, 1999. Optical dating of single and multiple grains of quartz from Jinmium Rock Shelter, northern Australia: Part 1, experimental design and statistical models. *Archaeometry* 41(2): 339-364, DOI 10.1111/j.1475-4754.1999.tb00987.x.

Hagedorn EM and Boenigk W, 2008. The Pliocene and Quaternary sedimentary and fluvial history in the Upper Rhine Graben based on heavy mineral analyses. *Netherlands Journal of Geosciences - Geologie en Mijnbouw* 87(1): 21-32.

Haimberger R, Hoppe A and Schäfer A, 2005. High-resolution seismic survey on the Rhine River in the northern Upper Rhine Graben. *International Journal of Earth Sciences* 94(4): 657-668, DOI 10.1007/s00531-005-0514-z.

Hoselmann C, 2008. The Pliocene and Pleistocene fluvial evolution in the northern Upper Rhine Graben based on results of the research borehole at Viernheim (Hessen, Germany). *Quaternary Science Journal (Eiszeitalter und Gegenwart)* 57: 286-315.

Huntley DJ and Baril MR, 1997. The K content of the K-feldspars being measured in optical dating or thermoluminescence dating. *Ancient TL* 15(1): 11-13.

Huntley DJ and Lamothe M, 2001. Ubiquity of anomalous fading in K-feldspars and the measurement and correction for it in optical dating, *Canadian Journal of Earth Sciences* 38(7): 1093-1106, DOI 10.1139/cjes-38-7-1093.

Jain M, Murray A and Bøtter-Jensen L, 2004. Optically stimulated luminescence dating: how significant is incomplete bleaching in fluvial environments. *Quaternaire* 15: 143–157.

Kars RH, Wallinga J and Cohen KM, 2008. A new approach towards anomalous fading correction for feldspar IRSL dating – tests on samples in field saturation. *Radiation Measurements* 43(2-6): 786-790, DOI 10.1016/j.radmeas.2008.01.021.

Kemna HA, 2005. Pliocene and Lower Pleistocene Stratigraphy in the Lower Rhine Embayment, Germany. *Kölner Forum für Geologie und Paläontologie* 14: 1-121.

Knipping M, 2008. Early and Middle Pleistocene pollen assemblages of deep core drillings in the northern Upper Rhine Graben, Germany. *Netherlands Journal of Geosciences - Geologie en Mijnbouw* 87(1): 51-66.

Krbetschek MR, Trautmann T, Dietrich A and Stolz W, 2000. Radioluminescence dating of sediments: Methodological aspects. *Radiation Measurements* 32(5-6): 493-498, DOI 10.1016/S1350-4487(00)00122-0.

Krbetschek MR, Degering D und Alexowsky W, 2008. Infrarot-Radiofluoreszenz-Alter (IR-RF) unter-saalezeitlicher Sedimente Mittel- und Ostdeutschlands. *Zeitschrift der Deutschen Gesellschaft für Geowiss*enschaften 159(1): 133-140.

Lauer T, Frechen M, Hoselmann C and Tsukamoto S, 2010. Fluvial aggradation phases in the Upper Rhine Graben - New insights by quartz OSL dating. *Proceedings of Geologists´ Association* 121: 154-161, DOI 10.1016/j.pgeola.2009.10.006.

Lepper K and McKeever SWS, 2002. An objective methodology for dose distribution analysis. Radiation Protection Dosimetry. *Solid State Dosimetry* 101: 349-352.

Litt T, 2007. Introduction: Climate, Vegetation and Mammalian Faunas in Europe during Middle Pleistocene Interglacials (MIS 7,9,11). In: Sirocko F, Claussen M, Sánchez Goni MF and Litt T, eds., *The climate of past interglacials*. Elsevier: 352-358.

Litt T, Behre KE, Meyer KD, Stephan HJ and Wansa S, 2007. Stratigraphische Begriffe für das Quartär des norddeutschen Vereisungsgebietes / Stratigraphical Terms for the Quaternary of the North German Glaciation Area. In: Litt T, ed., *Stratigraphie von Deutschland*, special issue in *Eiszeitalter und Gegenwart* 56(1/2)*:* 7-66.

Miall AD, 1996. The Geology of Fluvial deposits. Sedimentary Facies, Basin Analysis, and Petroleum Geology. Berlin, Springer: 583 pp.

Miallier D, Sanzelle S and Fain J, 1983. The use of flotation technique to separate quartz from feldspar. *Ancient TL* 1: 5-6.

Peters G and Van Balen RT, 2007. Tectonic geomorphology of the northern Upper Rhine Graben, Germany. *Global and Planetary Change* 58(1-4): 310-334, DOI 10.1016/j.gloplacha.2006.11.041.

Preusser F, 2008. Characterisation and evolution of the River Rhine system. *Netherlands Journal of Geosciences – Geologie en Mijnbouw* 87 (1): 7-19.

Rähle W, 2005. Eine mittelpleistozäne Molluskenfauna aus dem Oberen Zwischenhorizont des nördlichen Oberrheingrabens (Bohrung Mannheim-Lindenhof). *Mainzer Geowissenschaftliche Mitteilungen* 33: 9-20.

Rodnight H, 2008. How many equivalent dose values are needed to obtain a reproducible distribution? *Ancient TL* 26: 3–10.

Roe HM, 1999. Late Middle Pleistocene sea-level change in the southern North Sea: the record from eastern Essex, UK. *Quaternary International* 55(1): 115-128, DOI 10.1016/S1040-6182(98)00032-9.

Rolf C, Hambach U and Weidenfeller M, 2008. Rock and palaeomagnetic evidence for the Plio-Pleistocene palaeoclimatic change recorded in Upper Rhine Graben sediments (Core Ludwigshafen-Parkinsel). *Netherlands Journal of Geosciences - Geologie en Mijnbouw* 87(1): 39-48.

Templer RH, 1986. The localised transition model of anomalous fading. *Radiation Protection Dosimetry* 17: 493-497.

Trautmann T, Krbetschek MR, Dietrich A and Stolz W, 1998. Investigations of Feldspar Radioluminescence: Potential for a new Dating Technique. *Radiation Measurements* 29(3-4): 421-425, DOI 10.1016/S1350-4487(98)00012-2.

Trautmann T, Krbetschek MR, Dietrich A and Stolz W, 1999a. Radioluminescence dating: a new tool for Quaternary geology and archaeology. *Naturwissenschaften* 86: 441-444.

Trautmann T, Krbetschek, MR, Dietrich A and Stolz W, 1999b. Feldspar radioluminescence: a new dating method and ist physical background. *Journal of Luminescence* 85(1-3): 45-58, DOI 10.1016/S0022-2313(99)00152-0.

Trautmann T, Krbetschek MR, Dietrich A and Stolz W, 2000. The basic principle of radioluminescence dating and a localized transition model. *Radiation Measurements* 32(5-6): 487-492, DOI 10.1016/S1350-4487(00)00119-0.

Van den Boogard C, Van den Boogard P and Schmincke HU, 1989. Quartärgeologisch-tephrostratigraphische Neuaufnahme und Interpretation des Pleistozänprofils Kärlich. *Eiszeitalter und Gegenwart* 39: 62-86.

Vasil´chenko V, Molod´kov A and Jaek I, 2005. Tunneling processes and anomalous fading in natural feldspars extracted from quaternary deposits. *Journal of Applied Spectroscopy* 72(2): 218-223, DOI 10.1007/s10812-005-0058-9.

Wallinga J, 2002. Optically stimulated luminescence dating of fluvial deposits: a review. *Boreas* 31(4): 303-322, DOI 10.1111/j.1502-3885.2002.tb01076.x.

Wedel J, 2008. Pleistozäne Mollusken aus Forschungsbohrungen des Heidelberger Beckens. *Quaternary Science Journal (Eiszeitalter und Gegenwart)* 57: 382-402.

Weidenfeller and Kärcher, 2008. Tectonic influence on fluvial preservation: aspects of the architecture of Middle and Late Pleistocene sediments in the northern Upper Rhine Graben, Germany. *Netherlands Journal of Geosciences - Geologie en Mijnbouw* 87(1): 33-40.

Weidenfeller M and Knipping M, 2008. Correlation of Pleistocene sediments from boreholes in the Ludwigshafen area, western Heidelberg Basin. *Quaternary Science Journal (Eiszeitalter und Gegenwart)* 57: 270-285.

Wintle AG, 1973. Anomalous fading of thermoluminescence in mineral samples. *Nature* 245(5421): 143-144, DOI 10.1038/245143a0.

Ziegler PA and Fraefel M, 2009. Response of drainage systems to Neogene evolution of the Jura fold-thrust belt and Upper Rhine Graben. *Swiss Journal of Geosciences* 102(1): 57-75, DOI 10.1007/s00015-009-1306-4.