

E & G

Quaternary Science Journal



Special issue:
**Recent progress in Quaternary
dating methods**

Guest editors: Frank Preusser, Irka Hajdas and Susan Ivy-Ochs



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Front cover picture: (top left) Erratic boulder from Kaçkar Mountains, Turkey, sampled for dating by cosmogenic nuclides (photo: Naki Akçar); (top right) AMS facilities at ETH Zürich (photo: Susan Ivy-Ochs); (bottom left) sampling of loess for palaeomagnetic measurements (photo: Ulrich Hambach); (bottom right) fluvial deposits at Solenberg, Switzerland, sampled for luminescence dating (photo: Frank Preusser).

Titelbild: (oben links) Dieser erratischer Block aus dem Kaçkar Gebirge, Türkei, wurde für die Datierung mittels kosmogener Nukliden beprobt (Foto: Naki Akçar); (oben rechts) AMS Anlage an der ETH Zürich (Foto: Susan Ivy-Ochs); (unten links) Beprobung eines Lösses für paläomagnetische Messungen (Foto: Ulrich Hambach); (unten rechts) Fluviale Ablagerungen bei Solenberg, Schweiz, die für die Datierung mittels Lumineszenz beprobt worden sind (Foto: Frank Preusser).

Forthcoming Volume of Eiszeitalter & Gegenwart

E&G
Quaternary Science Journal

Special issue 57/3-4 (printed in December 2008)

The Heidelberg Basin Drilling Project

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Preface

This volume of *Quaternary Science Journal (Eiszeitalter und Gegenwart)* comprises a number of articles reviewing the present status of physical dating methods in Quaternary research, with a special focus on continental deposits and archaeological materials.

During the past two decades there has been an increased need for establishing independent timeframes in the context of, in particular, archaeological and palaeoclimate research. Over the same time period, enormous methodological improvements have been achieved with regard to Quaternary dating methods. These have had a similar pace as the developments in computer and information technology.

The ten articles in the present volume aim to provide the non-specialist with an overview on the physical basis of different dating approaches, which kind of procedures are applied from sampling to age calculation, which problems may interfere with proper age calculation and how these problems can be identified and corrected for. The articles should fill the gap between text books, usually only scratching the surface of particular methods, and pure methodological reviews, mostly inaccessible for the non-specialist who lacks the necessary background. We are aware that not all methods for dating Quaternary materials are included but the present volume should cover the most common approaches used in Quaternary research.

FRANK PREUSSER (BERN), IRKA HAJDAS (ZÜRICH) AND SUSAN IVY-OCHS (ZÜRICH)

Vorwort

Diese Ausgabe von *Quaternary Science Journal (Eiszeitalter und Gegenwart)* umfasst eine Anzahl an Artikeln, die den gegenwärtigen Stand physikalischer Datierungsmethoden in der Quartärforschung zusammenfassen, wobei ein spezieller Fokus auf kontinentale Ablagerungen und archäologische Materialien gerichtet ist.

Während der letzten beiden Jahrzehnte hat die Notwendigkeit unabhängige Zeitskalen zu etablieren, vor allem im Zusammenhang mit archäologischer und paläoklimatologischer Forschung, stark zugenommen. Im selben Zeitraum wurden enorme methodische Verbesserungen im Bereich quartärer Datierungsmethoden erzielt, die sich zusammen und mit derselben Rasananz wie die Computer- die Informationstechnologie entwickelt haben.

Die zehn Artikel der vorliegenden Ausgabe verschaffen dem Nichtspezialisten einen Überblick über die physikalischen Grundlagen der verschiedenen Datierungsansätze, welche Prozeduren von der Probenahme bis zur Altersberechnung angewendet werden, welche Probleme eine korrekte Altersberechnung erschweren können und wie diese Probleme erkannt und behoben werden können. Die Artikel sollen helfen die Lücke zu schließen, zwischen Lehrbüchern, die üblicherweise bestimmte Methoden nur oberflächlich behandeln, und reinen methodischen Übersichtsarbeiten, die für den Nichtspezialisten wegen des fehlenden Hintergrundwissens meist kaum zugänglich sind. Es ist uns bewusst, dass nicht alle Methoden zur Datierung quartärer Materialien im vorliegenden Band enthalten sind, die wichtigsten Ansätze, die in der Quartärforschung benutzt werden sollten aber berücksichtigt sein.

FRANK PREUSSER (BERN), IRKA HAJDAS (ZÜRICH) UND SUSAN IVY-OCHS (ZÜRICH)

Radiocarbon dating and its applications in Quaternary studies

IRKA HAJDAS^{*)}

Abstract: This paper gives an overview of the origin of ^{14}C , the global carbon cycle, anthropogenic impacts on the atmospheric ^{14}C content and the background of the radiocarbon dating method. For radiocarbon dating, important aspects are sample preparation and measurement of the ^{14}C content. Recent advances in sample preparation allow better understanding of long-standing problems (e.g., contamination of bones), which helps to improve chronologies. In this review, various preparation techniques applied to typical sample types are described. Calibration of radiocarbon ages is the final step in establishing chronologies. The present tree ring chronology-based calibration curve is being constantly pushed back in time beyond the Holocene and the Late Glacial. A reliable calibration curve covering the last 50,000–55,000 yr is of great importance for both archaeology as well as geosciences. In recent years, numerous studies have focused on the extension of the radiocarbon calibration curve (INTCAL working group) and on the reconstruction of palaeo-reservoir ages for marine records.

[Die Radiokohlenstoffmethode und ihre Anwendung in der Quartärforschung]

Kurzfassung: Dieser Beitrag gibt einen Überblick über die Herkunft von Radiokohlenstoff, den globalen Kohlenstoffkreislauf, anthropogene Einflüsse auf das atmosphärische ^{14}C und die Grundlagen der Radiokohlenstoffmethode. Probenaufbereitung und das Messen der ^{14}C Konzentration sind wichtige Aspekte im Zusammenhang mit der Radiokohlenstoffdatierung. Gegenwärtige Fortschritte in der Probenaufbereitung erlauben ein besseres Verstehen lang bekannter Probleme (z.B. die Kontamination von Knochen) und haben zu verbesserten Chronologien geführt. In diesem Überblick werden verschiedene Aufbereitungstechniken für typische Probengattungen beschrieben. Der letzte Schritt beim Erstellen einer Chronologie ist die Kalibrierung der Radiokohlenstoffalter. Die gegenwärtige auf Baumringzeitreihen basierende Kalibrationskurve wird stetig über das Holozän und Spätglazial hinaus erweitert. Eine zuverlässige Kalibrationskurve für die letzten 50.000–55.000 Jahre ist von herausragender Bedeutung sowohl für die Archäologie als auch die Geowissenschaften. In den letzten Jahren haben zahlreiche Studien an der Erweiterung der Radiokohlenstoff-Kalibrationskurve (INTCAL working group) und an der Rekonstruktion des Paläo-Reservoir effekts in marinen Archiven gearbeitet.

Keywords: Radiocarbon dating, sample preparation, calibration, Quaternary

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1 Introduction

The cosmogenic (produced by cosmic rays) isotope of carbon ^{14}C , also called radiocarbon, is the heaviest of the three isotopes of this element occurring in nature. The two stable isotopes ^{12}C and ^{13}C (representing 99.9% and 0.1%, respectively, of all natural carbon) originate from the primordial composition of the planet. Although ^{14}C takes up only a minute fraction of the carbon content (10^{-12}), its presence in carbon-bearing materials and the half-life ($T_{1/2}$) of 5730 ± 40 yr (GODWIN 1962) form the basis for important geochronological and environmental applications.

Cosmic rays hitting the Earth's atmosphere produce cascades of secondary particles. Thermal neutrons, which are products of cosmic ray interaction, react with nitrogen of the atmosphere (Fig. 1). On average, 2 atoms cm^{-2} of ^{14}C are produced in the atmosphere every second. Oxidised to CO and CO_2 molecules, which takes weeks and weeks to months, respectively (ROM et al. 2000), ^{14}C is then quickly mixed within the atmosphere. Monitoring of the atmospheric ^{14}C levels of the nuclear tests in the 1960s has shown that inter-hemispheric mixing occurs in one to two years. For the purpose of radiocarbon dating, the difference between various regions is marginal. However, the difference between the hemispheres (ca. 5 ‰) might be reconsidered through regional calibration (McCORMAC et al. 2004).

Age measurements are possible because ^{14}C becomes a part of all organic and inorganic carbon compounds and a steady state between the uptake (photosynthesis or food) and the decay of ^{14}C exists as long as the organism is alive (LIBBY et al. 1949). After death, the only remaining process is decay (beta decay in which ^{14}C decays to nitrogen). Measurement of the beta-decay rate (conventional method) or counting the remaining ^{14}C atoms (AMS method) gives a measure of the time that elapsed since the steady state was broken. The half-life of 5730 ± 40 yr (GODWIN 1962) allows the application of this method for the last 50,000-55,000 yr.

The main processes of the carbon cycle also control the ^{14}C exchange of the atmosphere with other main reservoirs: the ocean, the biosphere and the sediments (Fig. 2). The very fast exchange rate between the atmosphere and the biosphere assures equal ^{14}C concentration in contemporary living organisms, which is important for the use of radiocarbon as a dating tool. The ocean, which is the largest carbon reservoir, has a long residence time of ^{14}C and in consequence, the surface water and the deep-water

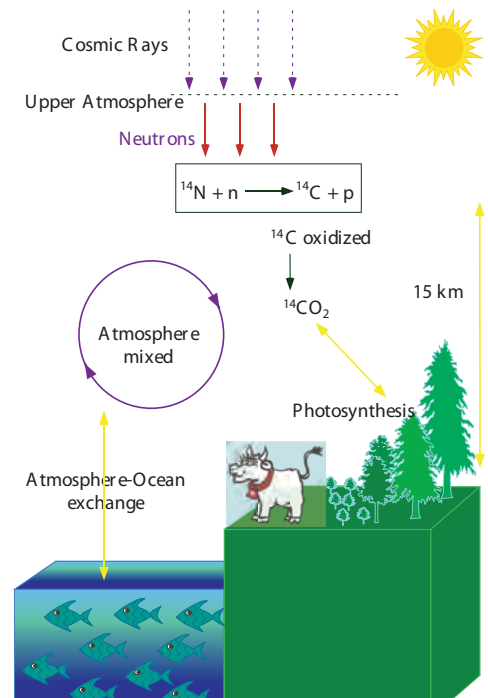


Fig. 1: Production and distribution of cosmogenic ^{14}C . Produced mainly in the upper atmosphere due to interaction of thermal neutrons with nitrogen, ^{14}C is relatively quickly oxidised and mixed in the atmosphere. Through photosynthesis, it enters the biosphere and through gas exchange, the oceans.

Abb. 1: Produktion und Verbreitung von kosmogonem ^{14}C . Das hauptsächlich in der oberen Atmosphäre durch Interaktion von thermischen Neutronen mit Stickstoff produzierte ^{14}C wird relativ schnell oxidiert und in der Atmosphäre vermischt. Durch Photosynthese gelangt es in die Biosphäre und durch Gasaustausch in die Ozeane.

masses have ages relative to the atmosphere, giving rise to the so-called reservoir age.

Due to various mechanisms such as changes in production rate and/or in the exchange rate between the carbon reservoirs, the atmospheric ^{14}C content fluctuates over time. These variations affect radiocarbon dating and often limit precision of age estimates.

2 Production rate and global carbon cycle.

In general, the cosmic rays flux remains constant (VOGT et al. 1990) and observed fluctuations in production rate of atmospheric ^{14}C are controlled by geomagnetic field strength as well as solar activity. In effect, a correlation exists between the number of solar spots (active sun) and the production rate of cosmogenic isotopes. The influence of solar and geomagnetic shielding on the production rate of cosmogenic isotopes can be simulated (MASARIK & BEER 1999). Based on these simulations, the globally averaged production rate calculated for ^{14}C is $2.02 \text{ atoms cm}^{-2} \text{ s}^{-1}$.

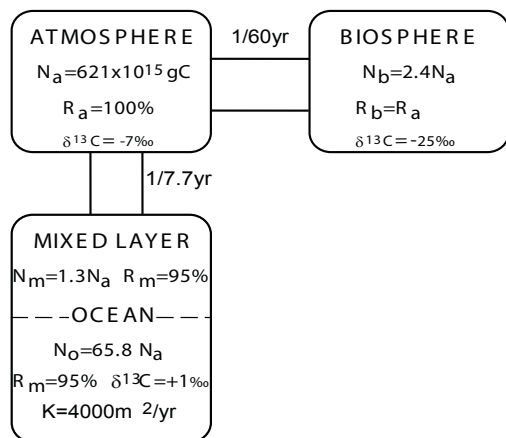


Fig. 2: The global carbon cycle (parameters used by SIEGENTHALER et al. 1980). N_i = amount of carbon in reservoir i ; R_i = $^{14}\text{C}/^{12}\text{C}$ ratio; ^{13}C = isotopic fractionation

Abb. 2: Der globale Kohlenstoffkreislauf (mit den Kenngrößen verwendet von SIEGENTHALER et al. 1980). N_i = Menge Kohlenstoff im Reservoir i ; R_i = $^{14}\text{C}/^{12}\text{C}$ Verhältnis; ^{13}C = isotopische Fraktionierung.

Various natural archives of cosmogenic isotopes have documented variability of production rate with time. For example, higher production rates of the cosmogenic isotopes ^{10}Be and ^{14}C are observed in ice cores and tree rings from the most pronounced periods of low solar activity: the Maunder, Wolf and Spörer minima, when the shielding was weaker (BEER et al. 1988). Variations in the strength of the geomagnetic dipole are also reconstructed using records of relative palaeointensity of the deep-sea sediments (CHANNELL et al. 2000; LAJ et al. 2000). A comparison of production rates derived from geomagnetic strength with the ice core record of ^{10}Be shows a striking correlation (LAJ et al. 2002). Relatively short changes (events) have been observed during the last 50 ka. Higher production rates of the cosmogenic isotopes ^{10}Be , ^{36}Cl and ^{14}C characterised periods/intervals of the Laschamp (41 ka) and Mono Lake (32 ka) geomagnetic excursions when the intensity of the geomagnetic field was low (Fig. 3) (HUGHEN et al. 2004a; LAJ et al. 2002; VOELKER et al. 2000; WAGNER et al. 2000).

Distribution of ^{14}C within and between reservoirs of carbon affects atmospheric ^{14}C content. The main mechanism of these changes is climatic variability because the ocean, which is the largest C reservoir (Fig. 2), is also an important component of the Earth's climate system. For example, changes of deep ocean ventilation during the glaciations had an impact on the atmosphere-ocean exchange of ^{14}C . Models have been developed to reconstruct the effect of such changes in the ocean ventilation on the ^{14}C inventory of both reservoirs. The simple box model of the ocean-atmosphere showed that reduced ventilation rates result in an increase, whereas the vigorous ocean ventilation results in a decrease of atmospheric ^{14}C content (SIEGENTHALER et al. 1980).

3 Anthropogenic impact on atmospheric ^{14}C

3.1 Suess effect

The industrial revolution of the late 19th century changed the ^{14}C content of the atmosphere. The

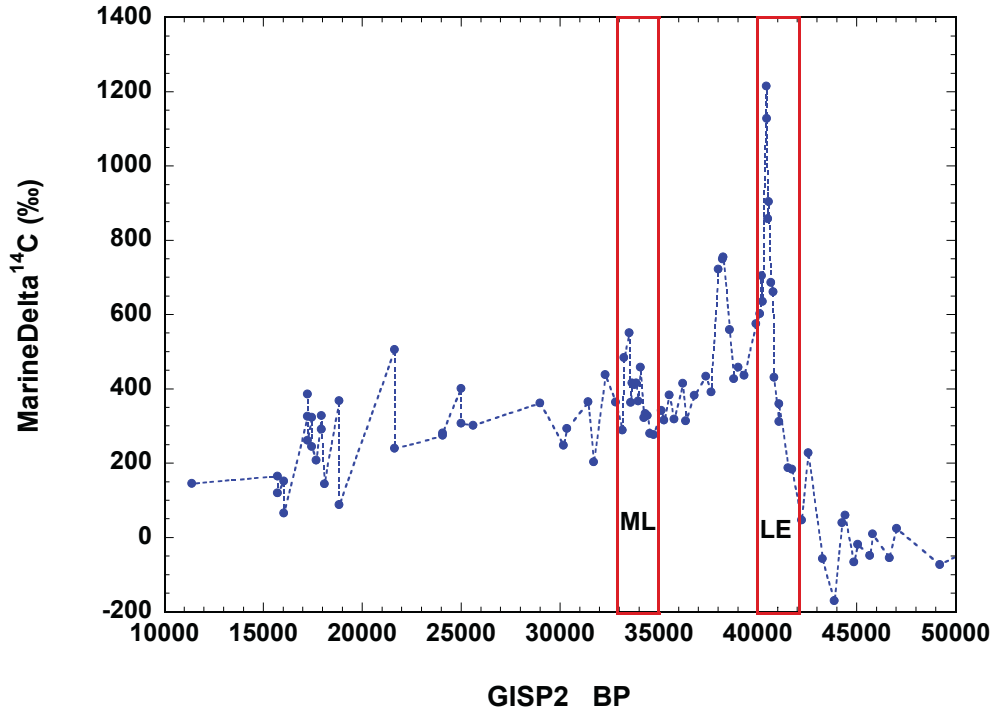


Fig. 3: Variations in ^{14}C concentration in foraminifera shells from Late Pleistocene Icelandic Sea sediments (VOELKER et al. 2000). The increases in marine ^{14}C , the measure of difference from the 'present day, i.e. 1950 AD ^{14}C level (STUIVER & POLACH 1977), observed at 40-42 ka and 33-35 ka BP, coincide with low relative magnetic field intensity (LAJ et al. 2002), i.e. weak geomagnetic shielding of the Laschamp Event (LE) and Mono Lake (ML) event, respectively.

Abb. 3: Variationen in der ^{14}C Konzentration in Foraminiferenschalen aus spätpleistozänen Sedimenten aus dem Isländischen Meer (VOELKER et al. 2000). Der Anstieg des marinen ^{14}C , als Maß des Unterschiedes zu heutigen, d.h. 1950 AD ^{14}C Niveau (STUIVER & POLACH 1977), der bei 40-42 ka und 33-35 ka auftritt, fällt mit einer niedrigen Intensität des magnetischen Feld zusammen (LAJ et al. 2002), d.h. mit einer schwachen geomagnetischen Abschirmung während des Laschamp Exkursions (LE) und des Mono Lake (ML) Exkursions.

increased burning of fossil fuels, which are free of ^{14}C (virtually all of the ^{14}C fixed in organic matter from millions of years ago has already decayed), added a significant part of ^{14}C free carbon dioxide to the atmosphere. In effect, the atmospheric $^{14}\text{C}/^{12}\text{C}$ ratio was lowered and thus, radiocarbon ages measured in post-industrial times will overestimate the real age of the sample. The effect was named after Hans Suess who first described it in the 1950s (Suess 1955). The observed effect of fossil fuel burning is most pronounced in the air from urban areas (LEVIN et al. 2008; PAWELCZYK & PAZDUR 2004).

3.2 The ^{14}C 'bomb peak'

The nuclear tests in the 1950s caused an increased stream of thermal neutrons into the stratosphere, which produced additional ^{14}C atoms and created an excess ^{14}C activity in the atmosphere (Fig. 4). Bomb produced ^{14}C was identified soon after the tests started and continuous monitoring has been carried out (NYDAL et al. 1984; NYDAL & LOVSETH 1965, 1983). The peak (ca. 100% above the normal levels) reached its maximum in 1963 in the northern hemisphere, where most of the tests took place. In the southern hemisphere, the bomb peak

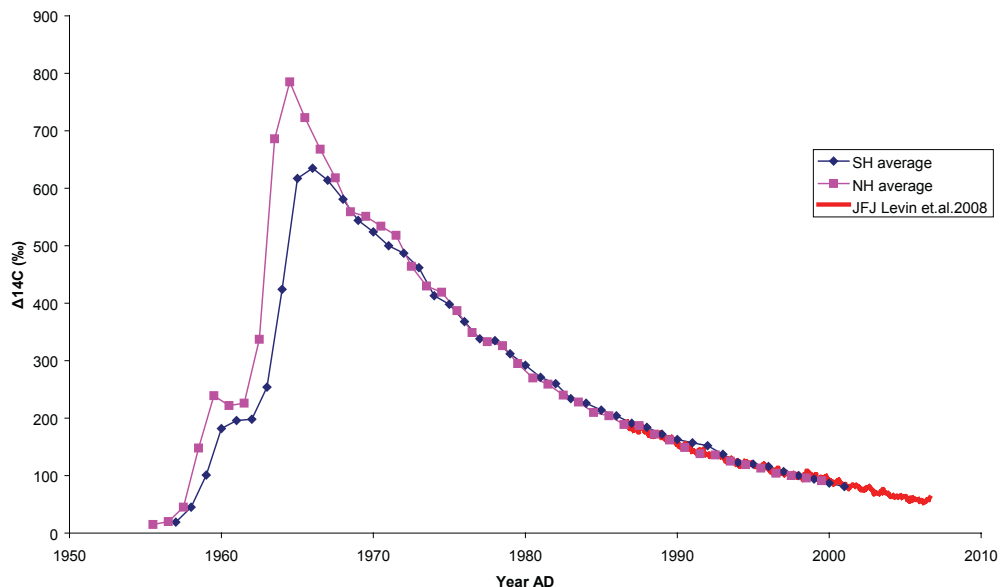


Fig. 4: The ^{14}C ‘bomb peak’. Measurements of post 1950 AD atmospheric ^{14}C concentration for both the Northern (NH) and Southern Hemispheres (SH) compiled by HUA & BARBETTI (2004). The monthly atmospheric ^{14}C data sets from measurements at the High Alpine Research Station Jungfraujoch (JFJ) show the present level of atmospheric ^{14}C (LEVIN et al. 2008).

Abb. 4: Der ^{14}C „Bomben-Peak“. Die nach 1950 AD durchgeführten Messungen der ^{14}C Konzentration für sowohl die Nördliche (NH) als die Südliche Hemisphäre (SH) wurden von HUA & BARBETTI (2004) kompiliert. Der monatliche ^{14}C -Datensatz von Messungen an der Hochalpinen Forschungsstation Jungfraujoch (JFJ) zeigt das gegenwärtige Niveau des atmosphärischen ^{14}C (LEVIN et al. 2008).

reached lower values (ca. 80 % of normal level) and was delayed by ca. two years. After the ban on above-ground nuclear tests in 1963, the atmospheric ^{14}C content began to decrease mainly due to uptake by the ocean and the biosphere (NYDAL et al. 1980). During the last decade, the decline has been caused mostly by dilution of the atmospheric CO_2 with ^{14}C -free fossil fuel CO_2 (ca. 2-3 per mil yr^{-1}) (LEVIN et al. 2008). Continuous monitoring of the atmospheric $^{14}\text{C}/^{12}\text{C}$ ratio during the years following the nuclear tests provided the basis for environmental studies (LEVIN & KROMER 2004; ROZANSKI et al. 1995). Applications range from studies of the ocean circulation, CO_2 uptake, and carbon storage in soils to medical and forensic studies as well as detection of forgeries (LEVIN & HESSHAIMER 2000; SPALDING et al. 2005a,b; TAYLOR et al. 1992).

4 Radiocarbon dating

The method as established by Libby and co-workers in late 1940s assumed a constant atmospheric ^{14}C content. For the reasons discussed above, such an assumption is invalid, as shown by more precise measurements of tree rings performed in early 1950s. Moreover, the half-life $T_{1/2} = 5568$ yrs used by ARNOLD & LIBBY (1949) was later found to be off by 3 % from its real value. However, an agreement has been reached that conventional radiocarbon ages (Libby ages) are calculated using the Libby half-life and then later calibrated (where a calibration curve is available) to obtain calendar ages that correspond to the measured ^{14}C concentration (age).

4.1 Conventional radiocarbon ages (Libby radiocarbon ages)

The ^{14}C concentration measured either by counting (KROMER & MÜNNICH 1992; HOGG et al. 2006 and references therein) or AMS techniques (FINKEL & SUTER 1993; JULL & BURR 2006 and references therein) provides information about the time elapsed since the time of deposition or death.

The activity of ^{14}C can be measured by counting of beta particles emitted by decaying ^{14}C or by measuring the $^{14}\text{C}/^{12}\text{C}$ ratio using accelerator mass spectrometry (AMS). Both methods allow the dating of natural carbon-bearing material. After death or deposition, the equilibrium between uptake from the environment (atmosphere, ocean, lake) and ^{14}C decay is broken. When the, new ^{14}C atoms cannot be incorporated by the organism, the activity begins to decrease with a half-life of 5730 yr. Application of the decay law for radiocarbon dating requires that the activity of the organic matter after the death of the organism changes only due to radioactive decay (i.e. the dated matter was a closed system). Radiocarbon age (conventional or Libby radiocarbon age) T can be calculated thusly:

$$T = 8033 \ln (A/A_0)$$

A = ^{14}C activity at the time of dating

A_0 = initial ^{14}C activity at time t_0
(deposition, death)

$T_{1/2}/\ln 2 = 8033$ yr, where

$T_{1/2} = 5568$ yr half-life used by Libby

In order to calculate ages, the initial activity A_0 at time t_0 must be known. As already discussed, A_0 is not constant over time and therefore an activity of pre-industrial wood grown in 1890 AD was chosen as reference. Although the half-life $T_{1/2}$ of 5568 yr (Libby half-life) is underestimated, it was accepted for use in the calculation of conventional radiocarbon ages (OLSON et al. 1966; STUIVER & POLACH 1977). Conventional radiocarbon age can then be corrected and expressed in radiocarbon ages for the half-life of 5730 years ($t_{5730} = 1.03 t_{\text{Libby}}$).

In addition, small variations in the initial activity of the sample are related to the mass dependent fractionation of carbon isotopes by chemical and physical reactions that occur in nature, e.g. photosynthesis in the case of plants (which is then passed to animal tissue via the food chain) or precipitation processes of carbonates. The degree of fractionation in a dated sample, defined as a depletion or enrichment of the isotope relative to the standard material (CRAIG 1953), is different for various materials (CRAIG 1954; STUIVER & POLACH 1977). Counting techniques use mass spectrometry measurements of gas split of CO_2 in order to obtain the correction value and some of AMS facilities measure the $^{14}\text{C}/^{13}\text{C}$ as well as $^{13}\text{C}/^{12}\text{C}$ ratios, which then can be applied to correct for fractionation.

Because the ^{14}C fractionation effect is approximately twice as much for ^{13}C , the correction applied to the ^{14}C content measured in the dated material is two times ^{13}C . Recently MOOK & VAN DER PLICHT (1999), REIMER et al. (2004c) and VAN DER PLICHT & HOGG (2006) summarised the conventions used for age calculation. The nomenclature used by these authors is summarised below. The ^{14}C concentration or ^{14}C activity of a sample is described as a fraction of reference material:

$$^{14}\text{a} = ^{14}\text{A} (\text{sample}) / ^{14}\text{A} (\text{reference})$$

^{14}A (reference) or standard activity corresponds to 95% of the activity of a specific batch of the Oxalic Acid standard HOx1 in the year 1950 AD. The secondary standard HOx2 used presently by most laboratories has a defined activity ratio of 1.2933 in relation to the original HOx1 (MANN 1983).

$$^{14}\text{A}_{\text{RN}}^0 = 0.95 \ ^{14}\text{A}_{\text{OX1N}}^0 = 0.7459 \ ^{14}\text{A}_{\text{OX2N}}^0 = 13.56 \pm 0.07 \text{ dpm gC}^{-1}$$

dpm gC^{-1} = disintegrations
per minute and per gram of carbon

$^{14}\text{A}_{\text{RN}}$ is the specific activity of the standard corrected for isotopic fractionation to

$^{13}\text{C} = -25\text{‰}$ for HOx2 and $^{13}\text{C} = -19.0\text{‰}$ for HOx1

The activity ratio of the measured sample or concentration is defined as:

$$^{14}\text{a} = ^{14}\text{A}/^{14}\text{A}_{\text{RN}}$$

This value is independent of when the measurement was carried out because it is relative to the standard of 1950 AD. The relative activity is expressed in per mil and defined as

$$^{14}\text{‰} = ^{14}\text{a} - 1$$

The measured isotopic ratio has to be corrected for mass fractionation, which occurs during chemical and physical processes in nature (see above). The ^{14}C activity and the relative content are normalised to $^{13}\text{C} = -25\text{‰}$ (wood):

$$^{14}\text{a}_{\text{N}} = ^{14}\text{a} \left[\frac{1 + ^{13}\delta_{\text{N}}}{1 + ^{13}\delta} \right]^2 = ^{14}\text{a} \left[\frac{0.975}{1 + ^{13}\delta} \right]^2$$

Another symbol for the activity ratio has been proposed by REIMER et al. (2004c) and is frequently used in publications:

$$\text{F}^{14}\text{C} = ^{14}\text{a}_{\text{N}}$$

This expression stands for the activity ratio of the measured sample to the standard corrected for the fractionation (as defined above) and for the background activity (blank values for AMS samples). Similar to the convention discussed above and published by STUIVER & POLACH (1977), the radiocarbon age T is calculated using the measured and normalised values of $^{14}\text{a}_{\text{N}}$ or F^{14}C :

$$T = -8033 \ln(\text{F}^{14}\text{C}) \text{ or } T = -8033 \ln(^{14}\text{a}_{\text{N}})$$

Conventional radiocarbon ages are reported as Before Present (BP), where 0 BP = year 1950 AD.

VAN DER PLICHT & HOGG (2006) define the frequently used value of ^{14}C (STUIVER & POLACH 1977), which illustrates the variations of ^{14}C in the atmosphere. The original ^{14}C content

of the measured sample can be reconstructed for samples of known calendar age (tree rings, varves, U/Th dated carbonates). The measured ^{14}C content of the sample in year 1950 AD is corrected for the decay of ^{14}C (with the correct decay constant) during the time that has elapsed since the sample was formed, t_i :

$$^{14}\delta_{\text{N}}^i = ^{14}\text{a}_{\text{N}}^i - 1$$

$$\begin{aligned} ^{14}\text{a}_{\text{N}}^i &= ^{14}\text{a}_{\text{N}} \exp\{-(t_i - 1950)/8267\} \\ &= \text{F}^{14}\text{C} \exp\{\text{calBP}/8267\} \end{aligned}$$

where $T_{1/2}/\ln 2 = 8267$ yr for $T_{1/2} = 5730$ yrs and calBP is the calendar age (cal BP) of the sample.

4.2 Precision and accuracy of measured radiocarbon ages

Precision characterises the degree of agreement among a series of individual measurements, i.e. the uncertainty of the measurement and is often quoted as the one-sigma error. This includes statistical error of counting atoms (AMS) or beta particles as well as uncertainty of measuring standards and blank values included in the calculation of radiocarbon ages. The counting error can be reduced by improved counting statistics. This can be achieved by increasing counting time. In the AMS technique, this is usually limited by the sample size as well as performance and stability of the AMS device.

Accuracy describes the difference between the calculated radiocarbon and the true age of a sample. This is independent of the measurement precision, i.e. radiocarbon ages can be very precise but their accuracy might be low or vice versa (SCOTT et al. 2007a). Radiocarbon laboratories check their accuracy using measurements of known age samples (for example a set of C samples available from IAEA). Moreover, regular proficiency checks known as inter-lab comparison are performed (SCOTT 2003a,b,c; SCOTT et al. 2004). Recently these comparison measurements were extended to various types of samples (NAYSMITH et al. 2007; SCOTT et al. 2007b).

4.3 Calibration of radiocarbon ages

Due to the variability of the atmospheric ^{14}C content and the used convention discussed above, radiocarbon ages are only 'defined' values and require calibration in order to obtain calendar ages. Calibration of radiocarbon ages compensates for both the error introduced by conventional (Libby) half-life $T_{1/2}$ and for the temporal variability of the atmospheric ^{14}C content.

Calibration is a mathematical procedure that places the measured radiocarbon ages (taken with their error) on the experimentally drawn curve. In the probabilistic calibration procedure, calendar ages, corresponding to the radiocarbon age obtained for the measured material,

are given with the probability distribution (68% and 95% confidence intervals for 1 sigma and 2 sigma error, respectively) (STUIVER & REIMER 1986, 1989). The calibrated radiocarbon ages are then reported as 'cal AD/BC' or 'cal BP' (calendar years BP). Because of the wiggles on the calibration curve, the transfer to calendar time scale may have a complicated probability distribution (Fig. 5).

Nowadays, calibration can be done using calibration software available via the web site of the *Radiocarbon* journal (<http://www.radiocarbon.org>). The calibration data used by most of the programs are based on the calibration data sets prepared and published by the International Calibration Group (INTCAL). The most recent set of data IN-

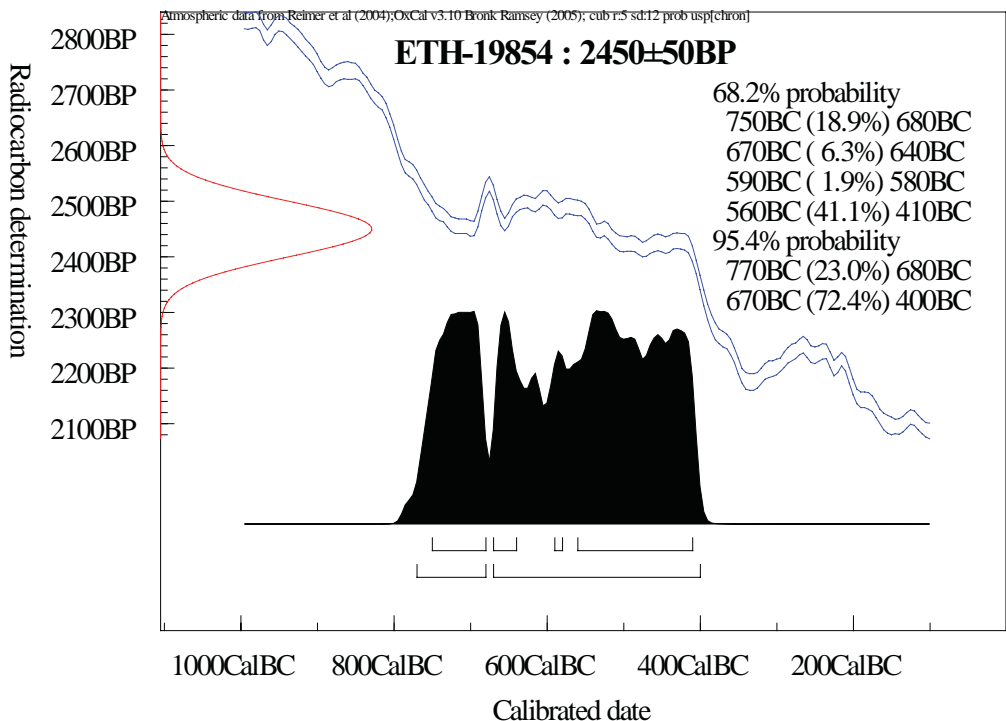


Fig. 5: Example of calibration of radiocarbon age (one of the samples from Ulandryk series, HAJDAS et al. 2004b), which is in the range of an age plateau (see text). The complex nature of the calibration curve (wiggles) results in multiple calendar age intervals.

Abb. 5: Beispiel für die Kalibrierung eines Radiokohlenstoffalters (eine der Proben aus der Ulandryk Serie, HAJDAS et al. 2004b), welches sich im Bereich eines Altersplateaus befindet (s. Text). Die komplexe Natur der Kalibrierungskurve („wiggles“) resultiert in einer Anzahl von möglichen Kalenderjahr-Intervallen.

TCAL04 was published in 2004 (HUGHEN et al. 2004b; REIMER et al. 2004a). The current state of the calibration curve and efforts to extend it to the limits of the method will be discussed later.

5 Sample types and preparation techniques

During the six decades of radiocarbon dating, the method has evolved towards more sophisticated applications that, in many cases, require very small sample sizes. The ultimate goal is to obtain a material for radiocarbon dating that is free of foreign carbon i.e., contaminants. Because carbon is such a common element in the environment, removing either modern (high amount of ^{14}C) or ancient (free of ^{14}C) contaminations has always been a challenging task. An overview of sample preparation presented here is relevant to both conventional and AMS sample preparation laboratories. The focus is however set on samples prepared for the AMS technique because many applications nowadays involve only minute sample amounts. While previous sample treatments were designed to remove contamination assuming that the material has been part of a closed system, present preparation involves evaluation of possible sources of contamination as well as extraction of the fractions that will give the most accurate age estimates. This requires understanding of the environment in which the dated material had been formed and preserved until sampling.

5.1 Sample types

The common types of samples include charcoal, wood, bones, animal tissue, textiles, paper, macroscopic remains of the plants (leaves, fruits, flowers), carbonates (corals, sediments, stalagmites and stalactites), water, air and organic matter from various sediments, soil, palaeosol and peat deposits. Depending on the sample type, an appropriate treatment is chosen to select the most relevant fraction of carbon, which will yield the accurate age of the studied object.

5.2 Sources of contamination

Modern dust that includes particles of, for example, hair, tissue, paper or pollen grains, is the most common source of contamination with modern carbon. In addition, bacterial growth might contaminate sediments and other organic matter during storage. Therefore, freezing or drying of the material to be dated is recommended.

Carbonates and humic acids infiltrate natural environments over the years during which the sample material has been buried in geological or archaeological settings. Diagenesis is the source of contamination that is introduced into the molecular structure of the material to be dated and such effects may often remain unnoticed. The process of exchange of carbon might lead to contamination with either young or old carbon. The most common problem in dating archaeological samples is degradation of bones. While buried in soil, they may incorporate humic acids from the environment. Another example found in geological applications is dissolution and re-crystallization of carbonates. Different types of carbonates may be affected to different degrees.

Conservative substances used to preserve artefacts or bones will introduce either modern or old carbon. Substances which had been used in the past cannot always be tracked down and therefore a range of solvents is used that should remove most commonly used conservative substances. A Soxhlet extraction of contaminants is routinely applied in many laboratories to pre-treat wood, bone and textile samples. Some geological samples also require such treatment if they were buried in bituminous sediments.

5.3 Treatment methods

As the sources and types of contamination vary, so do the methods of pre-treatment developed in order to provide the most accurate radiocarbon ages. The first step in applying these methods is an assessment made on the degree of contamination. Recent samples such as well-preserved wood and charcoal, as well

as textiles and in some cases bones, are usually viewed as intact materials where contaminants remain outside molecular structure of organic matter (M methods). In such cases macroscopic methods, which have been used for many decades by radiocarbon laboratories, are applied (VAN KLINKEN & HEDGES 1998). A small but ever increasing portion of samples are pre-treated at the molecular level where specific compounds are isolated, allowing more accurate dating of specific material such as degenerated bones, organic compounds in soil or deep-sea sediments (S-methods). With the exception of dating alpha-cellulose, isolation of specific compounds from a sample for dating would require an AMS facility to handle samples as small as mg and μg of carbon. The development of gas ion sources will allow an omission of the graphitisation process, which is limited by sample size, and thus direct measurement on CO_2 samples as small as a couple of micrograms.

5.3.1 Mechanical cleaning

Sample material will undergo visual examination after its arrival in the laboratory. All visible contaminants are removed under a binocular and the surface of the sample is cleaned if necessary. A short cleaning in an ultrasonic bath is applied to charcoal, textiles, wood and shells of molluscs and foraminifera to remove dust particles, etc. Some laboratories apply vacuuming of textiles surface because an ultrasonic bath might destroy and create a mash of the material. In the case of solid surfaces such as bones or corals, sand blasting is used to clean the surface (PATERNE et al. 2004). Soft materials such as sediments have their surfaces scraped. In the case of small plant fragments washed out of sediments, remains of aquatic plants must be removed with tweezers (HAJDAS 1993; HAJDAS et al. 1993). Rootlets that might have grown in the layer are removed from peat or soil samples by sieving.

5.3.2 The acid-alkali-acid (AAA) method

The AAA pre-treatment is known as the standard chemical treatment of organic matter for

radiocarbon dating, which is often applied to remove contamination by carbonates and humic acids (DE VRIES & BARENSEN 1954). The first wash in acid solution (Fig. 6) removes carbonate contamination attached to the surface. The duration of this step applied by different laboratories varies depending on the treated material. After washing with distilled water, a treatment with a weak base (for example 0.1 M NaOH) is applied to dissolve humic acids. Following rinsing to neutral pH, the final wash is a repeated with a hot acid bath to remove carbonates that precipitated from modern atmospheric CO_2 , which is dissolved in an alkali solution. This step is shorter than the other two and is followed with a final rinsing to neutral pH. The AAA (or ABA, acid-base-acid) method is relatively straightforward, hence commonly used in most laboratories. However, several studies (HATTÉ et al. 2001, HEAD & ZHOU 2000) have shown that the alkali step of AAA might be responsible for contamination of some material with modern C from atmospheric CO_2 dissolved during this step and incorporated into the sample structure. Such contamination cannot be entirely removed in the last acid wash if the commonly used weak HCl solution is applied. Materials that require such modification of classical AAA treatment include wood, peat and palaeosols, i.e. common objects for radiocarbon dating. The effect of contamination becomes significant when old material is prepared for radiocarbon dating. HATTÉ et al. (2001) proposed alternative treatments, which can be applied to samples that are susceptible to contamination of their inner structure. These are designed to remove modern carbon in the last step of treatment (modification of the last step of the AAA (Fig. 6)). For example, modern carbonates, which replaced functional groups in the samples of old wood during acid and base steps, can be detached from the structure by stronger agents such as sulphuric acid. In contrast to hydrochloric acid, H_2SO_4 has a stronger ionic affinity for carbonates.

5.3.3 ABOX Acid-Base-Oxidation

Another modification of the AAA (ABA)

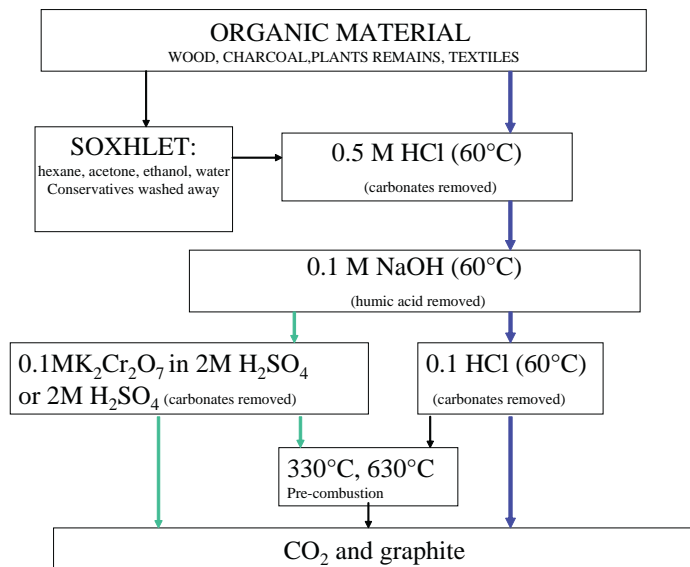


Fig. 6: Overview of pre-treatment methods routinely applied for cleaning organic material. The Soxhlet treatment is applied to material with suspected contamination caused by conservative substances or natural bituminous carbon (see text). The standard AAA treatment (blue path) might be enhanced by pre-combustion steps. ABOX (green path) is usually applied to treatment of material older than 20 ka.

Abb. 6: Überblick über Aufbereitungsmethoden, die routinemäßig für die Reinigung von organischem Material verwendet werden. Die Soxhlet Behandlung wird angewendet an Material, welches vermutlich durch konservierende Substanzen oder natürliches Bitumen verunreinigt ist (s. Text). Die Standard AAA Behandlung (blauer Pfad) kann durch Vorverbrennungsschritte verstärkt werden. ABOX (grüner Pfad), wird üblicherweise für Material verwendet, welches älter als 20 ka ist.

method involves a final oxidising step in the pre-treatment. BIRD et al. (1999) applied two methods of oxidation using a highly oxidising agent of acid dichromate solution (0.1M $K_2Cr_2O_7$ in a 2M solution of H_2SO_4) (Fig. 6), so-called 'wet oxidation', and pre-combustion of the sample in oxygen flow at 330°C and 630°C, i.e. temperatures lower than the final combustion at 850°C. BIRD et al. (1999) used these two methods and showed that the oldest ages of very old charcoal, i.e. the most effective removal of contamination can be obtained when 'wet oxidised' samples are also pre-combusted.

HATTÉ et al. (2001) proposed a replacement of the last hydrochloric wash of peat and paleosols (sediments rich in Fe) by a wash with an oxidising agent such as $K_2Cr_2O_7$. In this study, it was shown that the oxidation process breaks

insoluble ferrous compounds that were formed during the alkali step, which could possibly incorporate modern CO_2 .

5.3.4 Soxhlet treatment

Samples originating from museums and private collections often have their own history of conservation. In most cases, records of restoration are sparse or non-existent. Materials used as conservative substances contain carbon, which is either of modern or fossil origin and can contaminate samples. Naturally occurring samples can also be contaminated by fossil carbon. The effect has been observed in archaeological samples from Terqa, Syria where asphalt was present in the deposits. Some charcoal samples from this region were dated to be as old as 28,700 BP whereas the archaeological context

was dated to 3000 BP or younger (VENKATESAN et al. 1982). Therefore it is advisable to perform Soxhlet treatment of organic matter recovered from soils, sediments and any tar-containing environments.

For Soxhlet treatment, a standard procedure has been developed (BRUHN et al. 2001, HAJDAS et al. 2004c) and the most common conservative substances are removed in a sequence of solvents bathing (hexane, acetone and methanol). The sample is placed in the Soxhlet apparatus as shown in Fig. 7 and the liquid is brought to the boiling point. The condensate circulates in the Soxhlet for 30 min so that the sample is immersed in the clean hot solvent almost all the time. Contamination is removed with each spilling of the solvent through the siphon back to the heated bottle.

5.3.5 Leaching surface of carbonate samples

Surface of carbonate samples such as tufa, corals and shells may be contaminated due to diagenic processes. In order to remove such contamination, samples are cleaned mechanically (using an ultrasonic bath) and subjected to various leaching procedures, which involve acid and/or H_2O_2 solution. The degree of leaching varies, depending on the material dated. BURR et al. (1992) showed that 80 % leaching of coral surfaces yields satisfactory ages. Ages of ostracode shells from sediments of Mono Lake became older as the shells were successively dissolved (Fig. 8) (HAJDAS et al. 2004d).

5.3.6 Bone organic fraction

Bone contains mainly mineralised hydroxylapatite and carbonate-apatite as well as partly fluorapatite and chlorapatite, which are bound to a matrix of collagen. Collagen makes up to 20 % of dried, defatted fresh bone. A smaller fraction of the organic matrix is formed by non-collagen proteins such as osteocalcin or ferritin.

Most laboratories employ the method developed by LONGIN (1971) (Fig. 9). Bones are

first crushed or ground in a mill to speed up the dissolution in acid. This step removes contaminants but may remove collagen as well. Therefore, time and acid strength should be controlled during the process.

Gelatine is obtained by dissolution of collagen in a weak acid (LONGIN 1971). The demineralised sample is placed in 0.01N HCl solution and left at 58°C for 18-24 hr. The solution is then filtered and freeze-dried (Fig. 9) prior to combustion. In order to remove potential contamination of the bone, additional separation techniques have been developed. A modification of the LONGIN (1971) method, including

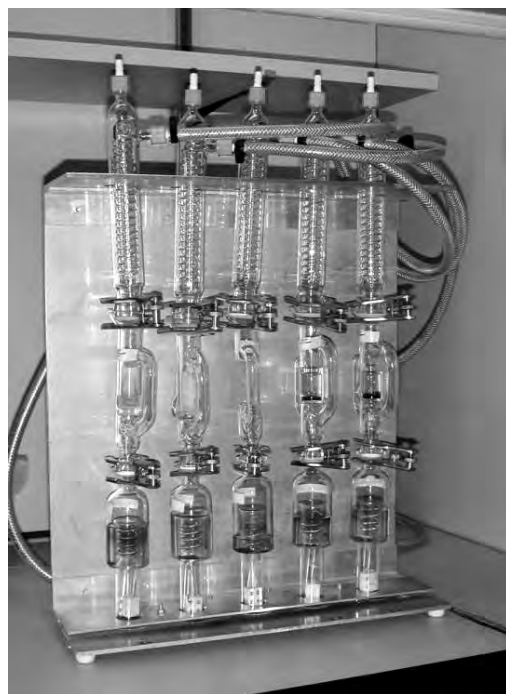


Fig. 7: Soxhlet preparation system at the ETH lab. The sample holders (middle part) are placed above a boiling solvent (lower part). The vapours of the solvent are cooled (upper part) and precipitated into the sample holder.

Abb. 7: Soxhlet Aufbereitungssystem im Labor der ETH. Die Probenhalter (mittlerer Teil) werden oberhalb eines kochenden Lösungsmittels platziert (unterer Teil). Der Dampf des Lösungsmittels wird gekühlt (oberer Teil) und schlägt sich im Probenhalter nieder.

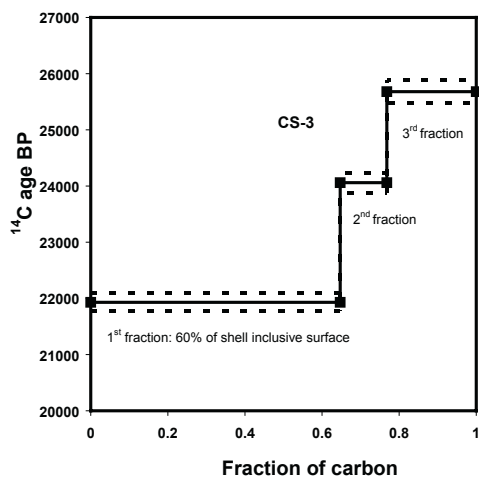


Fig. 8: Effect of consecutive leaching on radiocarbon age of ostracode shells from Carlson Sink, Mono Lake, USA (HAJDAS et al. 2004d).

Abb. 8: Effekt des fortlaufenden Ablaugens auf Radiokohlenstoffalter von Ostrakodenschalen aus Carlson Sink, Mono Lake, USA (HAJDAS et al. 2004d).

a short treatment of 'collagen' with a base solution prior to gelatinisation, has been proposed by ARSLANOV & SVEZHENTSEV (1993). This procedure was adopted by HAJDAS ET AL. (2007) and PIOTROWSKA & GOSLAR (2002) and proved very effective in removing contamination. Separation of molecular masses by ultra-filtration was proposed by BROWN et al. (1988) and improved providing very promising results (HIGHAM et al. 2006a, 2006b). The procedure of ultra-filtration involves separation of molecular masses because those of peptides are heavier than the possible contaminant of humic and fulvic acids. The liquid is placed in ultra-filtration tubes with a 30 kDa filter (Viva Spin or Millipore) and centrifuged at 4000 rpm for 15 to 30 min (depending on the volume of the tube). In effect, molecules heavier than 30 kDa will remain above whereas the smaller (contamination fraction) will pass through the filter and be discarded. The heavier fraction obtained is freeze-dried and then subjected to combustion.

6 Site and sample specific problems of radiocarbon dating

In order to obtain the most accurate ages and reliable chronologies for the studied objects or sites (sedimentary records), appropriate material must be sampled for radiocarbon dating. For lake sediments, terrestrial macrofossils should be sampled to avoid potential 'hard water' effects caused by old carbonates built into organic matter of aquatic plants (HAJDAS et al. 1993, 1995, 1998). The presence of old carbon dissolved from bedrock is different from site to site and the 'hard water' effect may range from hundreds of years to nil. More locally, radiocarbon ages of organic matter can also be influenced due to volcanic (^{14}C free) CO_2 added to the atmosphere or dissolved in water. For example, remains of aquatic plants found in sediments of Lake Monticchio (Italy) were more than 10,000 ^{14}C yr older compared to terrestrial macrofossils in the same sediment layer (HAJDAS et al. 1998). Another source of old carbon has been observed in polar regions where slow decomposition of organic matter and longevity of some species of mosses might result in older radiocarbon ages (HUMMUM et al. 2005). Similarly, too old ages are observed when re-deposited or re-used material is dated. The best example is the 'old wood' effect known in dating wooden artefacts.

For marine records, hand picked foraminifer shells of plankton living in the upper layer of the ocean are usually used for dating. The radiocarbon age of water in this layer varies from 400 to 1200 ^{14}C yr. Correction for the marine reservoir effect is based on measurements of marine radiocarbon ages of material with known age (REIMER & REIMER 2001). Other problems in dating marine records are related to bioturbation and poor preservation of shells as discussed by BROECKER et al. (1999, 2006).

7 Graphite preparation for AMS ^{14}C measurements

For AMS measurements of $^{14}\text{C}/^{12}\text{C}$ ratios, the autochthonous carbon present in the sample

material has to be transformed into pure graphite. The first step in this process is obtaining CO_2 by either combustion (organic matter) or acidification (shells and inorganic sediments). For combustion, dried organic matter is weighed and placed in a quartz glass tube, which contains wire-formed CuO for oxidation of the material that is sealed after the tube is evacuated. Silver wire or powder is added together with the sample to bind SO_2 and other halogen gases that could poison the graphitisation reaction. The sealed tubes are placed in an oven at 950°C for 2 hr. Carbonate samples are placed in a special chamber, which is evacuated, then mixed with concentrated phosphoric acid and dissolved. The resulting CO_2 is then purified and frozen using liquid nitrogen (HAJDAS et al. 2004d). The experimental details are given in HAJDAS et al. (2004c). Most of the AMS laboratories use the graphitisation method described by VOGEL et al. (1984),

where the reaction of $\text{CO}_2 + 2 \text{H}_2$ (heated to 580 to 600°C for ca. 2 to 4 hr) over catalyst (cobalt or iron powder) results in reduction to C and H_2O . The graphite that forms on the surface of the catalyst is then pressed onto a target designed for AMS measurement.

Measurements of the $^{14}\text{C}/^{12}\text{C}$ and $^{13}\text{C}/^{12}\text{C}$ ratios of the samples to be dated are determined relative to the respective NBS oxalic acid I or oxalic acid II standard values. Chemistry blanks prepared from natural graphite, coal or marble (for example the C-1 standard of IAEA, Vienna) is usually analysed in order to determine contamination introduced during sample preparation. All samples (unknowns, standards and blanks) of one series are measured several times (typically three or four). The total measuring time per sample is around 30 to 40 min. For details of measurements performed at the AMS facility at the ETH Zurich see BONANI et al. (1987) and SYNAL et al. (1997, 2007).

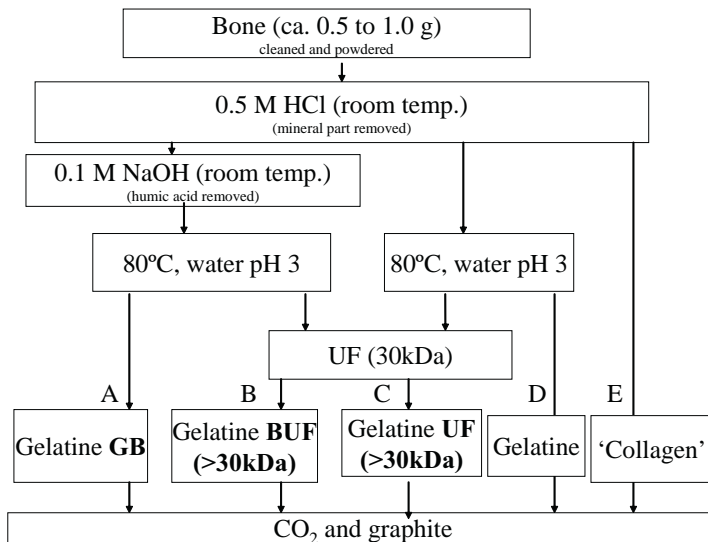


Fig. 9: Overview of bone treatment (organic fraction): path A: modified Longin method (ARSLANOV & SVEZHENTSEV 1993), path B: modified Longin method combined with Ultra Filtration, path C: Longin method + Ultra-Filtration (BROWN et al. 1988), path D: Longin method (LONGIN 1971), path E: 'collagen'.

Abb. 9: Überblick über der Aufbereitung von Knochen (organische Fraktion): Pfad A: modifiziertes Longin-Verfahren (ARSLANOV & SVEZHENTSEV 1993), Pfad B: modifiziertes Longin-Verfahren kombiniert mit Ultra-Filtration, Pfad C: Longin-Verfahren+Ultra-Filtration (BROWN et al. 1988), Pfad D: Longin-Verfahren (LONGIN 1971), Pfad E: 'Kollagen'.

8 Calibration issues

8.1 ^{14}C age ‘plateaux’, rapid ‘jumps’ and ‘wiggles’

Precise radiocarbon dating of certain periods of interest may be complicated by extreme variability of atmospheric ^{14}C content present at times in the radiocarbon calibration curve. These complications arise from variations in atmospheric ^{14}C content caused by the changes in the production rate and changes in the carbon cycle and are known as ‘wiggles’ in the calibration curve. Radiocarbon age ‘plateaux’, for example, caused by a decrease in the atmospheric ^{14}C concentration, appears as a slowing down of the ^{14}C clock such as one that occurred at 2500-2400 BP, i.e. between 700 BC and 400 BC (‘Hallstatt plateau’) (Fig. 10). In effect, similar radiocarbon ages may appear to correspond to a range of up to 500 calendar years. The opposite is observed when atmospheric ^{14}C levels increase so that the ^{14}C clock appears to speed up. Therefore, due

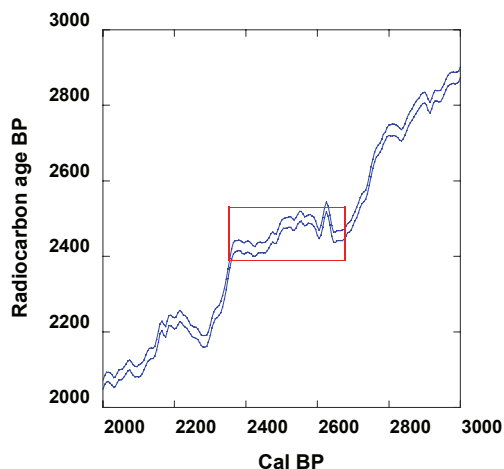


Fig. 10: Radiocarbon age plateau at 2400-2500 BP (700 BC and 400 BC) known as a ‘Hallstatt plateau’ marked on the calibration curve of INTCAL04 (REIMER et al. 2004a).

Abb. 10: Radiokohlenstoff Altersplateau zwischen 2400 und 2500 BP (700 BC bis 400 BC), bekannt als “Hallstatt Plateau”, markiert auf der Kalibrierungskurve INTCAL04 (REIMER et al. 2004a).

to the ‘wiggles’, a single radiocarbon age may correspond to more than one calendar age range or the ^{14}C clock may even appear to be reversed with respect to actual time.

The set of radiocarbon ages obtained on a trunk of a larch tree found in Scythian tombs/kurgans of Altai Mountains illustrates the effect the age plateaux can have on the precision of calendar ages and shows how the wiggle match can help to resolve it. Eleven out of sixteen calibrated ages fall on the 2400-2500 BP age plateau. In effect, the calibrated ages of all these samples range from 700 BC to 400 BC (Fig. 11). However, a χ^2 fit of the age sequence to the calibration curve resulted in a precise dating of the last ring to be $2267^{+13/-21}$ cal BP ($317^{+13/-21}$ BC) (Fig. 12), which was presumably the year when the kurgan was built (HAJDAS et al. 2004b).

Another method to improve calendar chronologies, despite the ^{14}C plateaux, is the use of models such as wiggle-matching or calibration models, where additional information about the samples (sequence order) might be applied to reduce the intervals of calibrated ages (RAMSEY et al. 2001). This Bayesian approach to calibration of radiocarbon ages (BUCK et al. 1996) is now included in calibration programs such as BCal (<http://bcal.sheffield.ac.uk>) (BUCK et al. 1999), OxCal (RAMSEY 2008) or Bpeat (BLAAUW & CHRISTEN 2005). ‘Wiggle-matching’ with the Bpeat program has been applied to develop age-depth models of Holocene peat sections from the Netherlands (BLAAUW et al. 2007). The high-resolution radiocarbon dating combined with Bayesian modelling was used to establish chronology of climatic events of Kaipo Bog (New Zealand) (HAJDAS et al. 2006). Similarly, the late Glacial sedimentary record of Hauterive/Rouges-Terres, Lake Neuchâtel (CH) (HAJDAS et al. 2004a) were radiocarbon dated and the age model was built using Bayesian model of OxCal v3 program (RAMSEY 2001).

8.2 Extension of the calibration curve

The history of variations in atmospheric ^{14}C production during the last 12,400 years is based on tree ring measurements and dendrochronol-

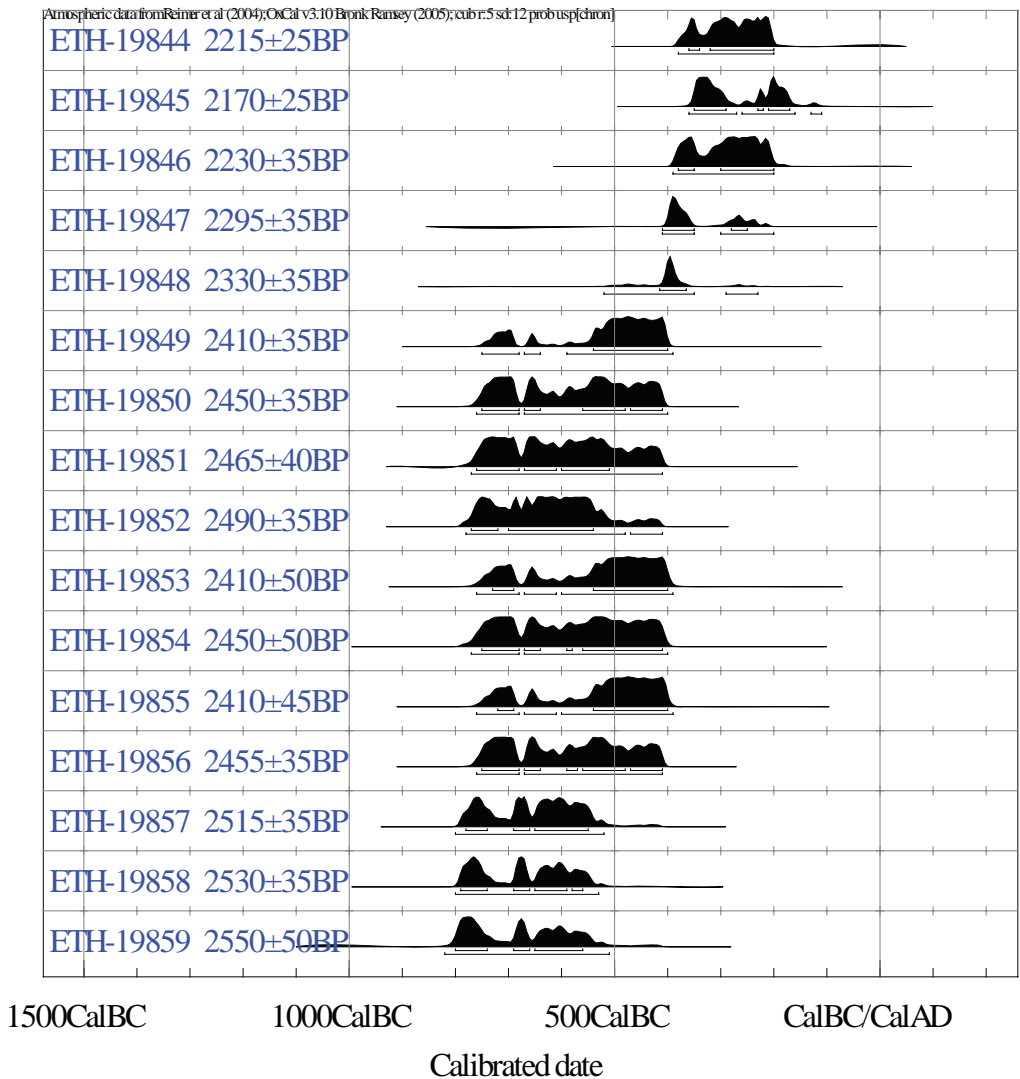


Fig. 11: Results of calibration of radiocarbon ages obtained for a tree ring sequence of samples from a larch tree recovered from a kurgan (burial mound) in Ulandryk (Altai Mountains) (HAJDAS et al. 2004b). Note the wide ranges of calibrated age intervals obtained for radiocarbon ages hitting the plateau (see text). An exception is the sample ETH-19848, 2330 ± 35 BP, as this age corresponds to a steep slope on the calibration curve

Abb. 11: Ergebnisse der Kalibrierung von Radiokohlenstoffaltern für Proben aus einer Baumringsequenz aus einer Lärche, die aus einem Kurgan (Grabhügel) aus Ulandryk (Altai Gebirge) geborgen wurde (HAJDAS et al. 2004b). Bemerkenswert ist der breite Bereich an kalibrierten Altersintervallen für Radiokohlenstoffaltern, die auf Plateaus liegen (siehe Text). Eine Ausnahme ist Probe ETH-19848, 2330 ± 35 BP, weil diese Probe mit einem steilen Abfall in der Kalibrationskurve zusammenfällt.

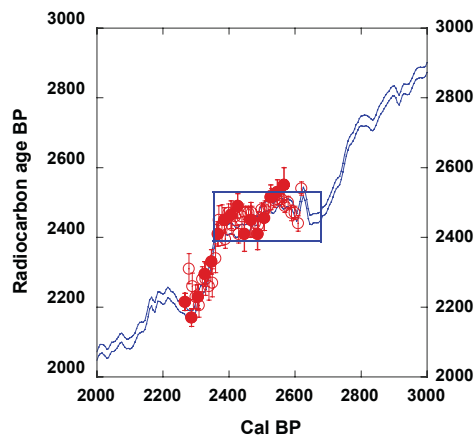


Fig. 12: Results from the Ulandryk kurgan (Fig. 11) fitted to the calibration curve (see text): filled circles (HAJDAS et al. 2004b) and open circles (KUZMIN et al. 2004). The last ring is dated to 2267^{+13}_{-21} cal BP (317^{+13}_{-21} BC).

Abb. 12: Ergebnisse für den Ulandryk Kurgan (Abb. 11) projiziert auf die Kalibrierungskurve (siehe Text): ausgefüllte Kreise (HAJDAS et al. 2004b) und offene Kreise (KUZMIN et al. 2004). Der letzte Ring wurde auf 2267^{+13}_{-21} cal BP (317^{+13}_{-21} BC) datiert.

ogy. However, the older part of the calibration has to be based on records other than the trees. The radiocarbon ages of foraminifera shells from sediments of Cariaco Basin and measurements of ^{14}C on U/Th dated corals, all corrected for corresponding local reservoir age, have been used to construct the late glacial and glacial part of the calibration curve (12,400-26,000 cal BP) (REIMER et al. 2004b) (Fig. 13). In addition to the atmospheric calibration curve, a global mix layer marine reservoir age has been modelled and used for construction of a marine calibration (HUGHEN et al. 2004b). Extension of the calibration curve to the limits of the radiocarbon method is of great interest to the wide range of palaeoclimate studies and archaeological research. Corals, marine and lake sediments and stalagmites may provide continuous records of ^{14}C changes (Fig. 14). In addition, Marine Isotope Stage 3 Kauri wood found in bogs of New Zealand is being studied for this purpose (HOGG et al. 2007, TURNEY et

al. 2007). In the future, an updated data set of INTCAL04 will allow conversion of radiocarbon ages older than 21000 BP to the estimated (not yet calibrated) calendar ages (RAMSEY et al. 2006).

Another problem of the calibration curve that must be considered is the reconstruction of the temporal as well as spatial variations of the marine reservoir effect. Variations documented for specific locations and periods (for summary see REIMER & REIMER 2001) may affect radiocarbon-based chronologies of the marine records. Efforts are being made to reconstruct such variations using high-resolution chronologies of marine records (BONDEVIK et al. 1999, 2001, 2006; MANGERUD et al. 2006) or to model it (FRANKE et al. 2008).

9 Summary and outlook

As long as carbon-bearing material is available, radiocarbon dating methods allow measurement of the isotopic composition of the material, which is only limited to the materials formed during the last 50-55 ka. The measured $^{14}\text{C}/^{12}\text{C}$ ratio relative to the reference material allows calculation of the radiocarbon age. However, due to the complicated history of ^{14}C , this age requires translation to the calendar age.

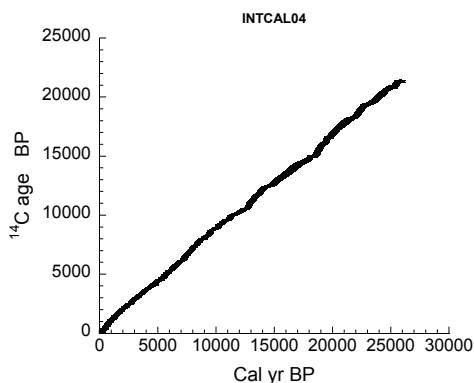


Fig. 13: Radiocarbon calibration curve of INTCAL04 (REIMER et al. 2004a).

Abb. 13: Radiokohlenstoff-Kalibrierungskurve INT-CAL04 (REIMER et al. 2004a).

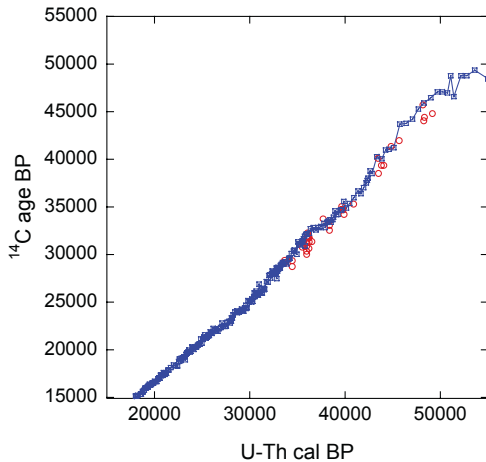


Fig. 14: Reconstruction of past variation in ^{14}C content based on measurements of foraminifera (squares) from Cariaco Basin; with the calendar time scale obtained by climatic correlation to U/Th dated stalagmites from the Hulu Cave (HUGHEN et al. 2006; WANG et al. 2001). Included are also corals ages (circles) dated by both radiocarbon and U/Th (FAIRBANKS et al. 2005).

Abb. 14: Rekonstruktion vergangener Variationen im ^{14}C Gehalt basierend auf Messungen von Foraminiferen (Vierecke) aus dem Cariaco Becken, die durch Klimatische Proxy mit der Kalender-Zeitreihe U/Th-datierter Stalagmiten aus der Hulu Cave korreliert wurden (HUGHEN et al. 2006; WANG et al. 2001) und Korallen (Kreise). Zudem sind Korallenalter enthalten, die sowohl durch Radiokohlenstoff als auch U/Th datiert wurden (FAIRBANKS et al. 2005).

Sampling and sample preparation are important steps in obtaining reliable radiocarbon ages. Measurements and error analysis, corrections for fractionation and reservoir effect are the next steps that are followed by the calibration of the radiocarbon age. From the very beginning of the method, all these components have been under constant development. Improved preparation techniques allow the selection of material free of contamination that is most suitable for dating. Twenty-five years ago, the development of the AMS revolutionised radiocarbon dating allowing for analyses on samples containing only a few mg of carbon

(as compared to grams needed for conventional radiocarbon dating method). Calibration issues were also recognised and great efforts of many international groups resulted in the calibration curves that are presently in use. This work is being continued and remains a great challenge to the radiocarbon dating community.

During the last ten years, novel solutions in the AMS technique resulted in lowering energies needed for successful separation of C isotopes. Radiocarbon-dating dedicated small AMS setups are becoming a reality, allowing for higher throughput and shorter turnaround time of samples (SUTER et al. 2007; SYNAL et al. 2007). Moreover, sophisticated preparative methods allow for separation of carbon at the molecular level (e.g., EGLINTON et al. 1996, INGALLIS et al. 2004, VON REDEN et al. 1998), providing opportunity for tracing contamination. The AMS systems that operate with a gas ion source allow measurements to be performed on CO_2 (RUFF et al. 2007). Samples containing few micrograms of C are combusted and their isotopic composition is measured without the graphitisation step involved. This technical development marks another revolution in radiocarbon dating that will allow radiocarbon dating at the molecular level. Thanks to this development, the great value of ^{14}C as an environmental tracer is becoming very appealing in addition to its usefulness as a dating tool.

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Magnetic dating of Quaternary sediments, volcanites and archaeological materials: an overview

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Abstract: Magnetic dating includes all approaches dealing with the temporal variation of the Earth's magnetic field (EMF) as well as with the application of climate dependent variations of rock magnetic properties of sedimentary sequences and their correlation to independently dated palaeoclimatic archives. Palaeomagnetism has an outstanding impact on geosciences in general and especially on Quaternary chronology and palaeoclimate research. Palaeomagnetic dating employs the temporal variation of the direction as well as the intensity of the EMF on time scales from 10^2 to 10^7 years. The well-known temporal pattern of reversals of the EMF on time scales from 10^4 to 10^7 years and the shorter secular variation (amplitude 10–30°, time scale 1 to 10^3 years) provide an excellent tool for stratigraphic subdivisions. Records of the intensity variations of the EMF as well as the indirect dating by means of correlating rock magnetic property variations from sedimentary archives to dated palaeoclimatic records also serve as dating tools. Field methods as well as laboratory methods and techniques in data analysis will not be discussed in this paper. It is our aim to give a short and subjective overview on palaeomagnetism and magnetic susceptibility stratigraphy as dating tools in Quaternary science.

[Magnetische Datierung quartärer Sedimente, Vulkanite und archäologischer Materialien: Ein Überblick]

Kurzfassung: Unter magnetischer Datierung versteht man sowohl die Verfahren, die die zeitlichen Variationen des Erdmagnetfeldes (EMF) zur Altersbestimmung benutzen, als auch die Verwendung der klimaabhängigen Änderungen der gesteinsmagnetischen Parameter und ihre Korrelation mit unabhängig datierten paläoklimatischen Archiven. Die Paläomagnetik hat einen herausragenden Einfluss auf die Geowissenschaften genommen und im Besonderen zur Etablierung der Chronologie des Quartärs und der Paläoklimaforschung beigetragen. Die paläomagnetische Datierung benutzt die zeitlichen Variationen der Richtung wie der Intensität des EMFs auf Zeitskalen von 10^2 bis 10^7 Jahren. Das wohlbekanntes zeitliche Muster von Polaritätswechseln des EMFs auf Zeitskalen von 10^4 bis 10^7 Jahren wie auch die Säkularvariation (Amplitude 10–30°, Zeitskala 1 bis 10^3 Jahre) stellen ein hervorragendes Werkzeug für die Unterteilung von sedimentären oder vulkanischen Gesteinsabfolgen dar. Aufzeichnungen der Änderungen der Intensität des EMFs sowie die indirekte Datierungen über den Vergleich der Variation gesteinsmagnetischer Parameter in Sedimentarchiven mit anderen datierten Paläoklimaserien können ebenfalls zur Datierung herangezogen werden. Weder die Methoden im Gelände und im Labor, noch die Techniken der Datenanalyse werden in dieser Publikation angesprochen. Unser Ziel ist vielmehr, einen kurzen und subjektiven Überblick über die Paläomagnetik bzw. magnetische Suszeptibilitätsstratigraphie als Datierungswerkzeug in der Quartärforschung zu geben.

Keywords: magnetic dating, Quaternary, palaeomagnetism, secular variation, magnetic susceptibility, stratigraphy

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1 Introduction

Palaeomagnetism, as a generic term for several sub-disciplines in geomagnetism (e.g., magnetostratigraphy, apparent polar wander, archaeomagnetism), is an essential part of geosciences. Its outstanding impact on geosciences in general and on the development of the model of plate tectonics in particular is widely appreciated. Rock magnetism (environmental magnetism) became famous in Quaternary research after its successful application as magnetic susceptibility stratigraphy for indirect dating of Asian loess deposits and their correlation to the global ice volume record.

The success and achievements of palaeo- and rock magnetism in earth sciences are amongst others, proven by the plenitude of monographs on these topics. For a deeper insight into the methods of palaeomagnetism, we refer to following textbooks without any claim for completeness (in chronological order): COLLINSON et al. (1967), McELHINNY (1973), STACEY & BANERJEE (1974), COLLINSON (1983), TARLING (1983), THOMPSON & OLDFIELD (1986), SOFFEL (1991), BUTLER (1992), VAN DER VOO (1993), OPDYKE & CHANNELL (1996), DUNLOP & ÖZDEMİR (1997), TAUXE (1998), MAHER & THOMPSON (1999), McELHINNY & McFADDEN (2000), EVANS & HELLER (2003), LANZA & MELONI (2006).

Field methods as well as laboratory methods and techniques in data analysis will not be discussed here. For these topics we refer to textbooks (e.g., COLLINSON 1983; BUTLER 1992).

This paper does not want to appear as a textbook in miniature. The aim of this paper is to give a short overview on palaeomagnetism and magnetic susceptibility stratigraphy as dating tools in Quaternary science and to draw the reader's attention to the possibilities and problems of the methods.

2 The Earth's magnetic field

Our planet is surrounded by a magnetic field. The intensity of the Earth's magnetic field (EMF) on its surface is extremely weak

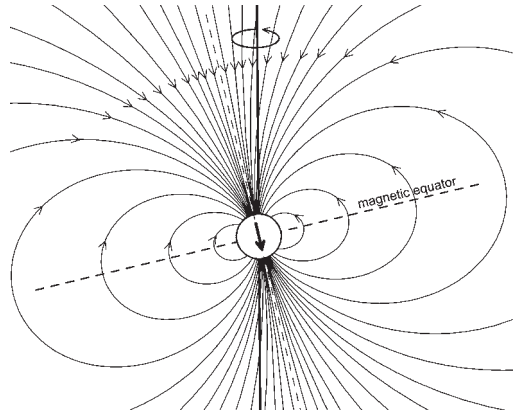


Fig. 1: Sketch of the Earth magnetic field (EMF) lines displaying its dipole character. Note the deviation of the dipole axis (dashed line) from the Earth's rotation axis. Redrawn from SCHNEPP (2007).

Abb. 1: Skizze der Feldlinien des Erdmagnetfeldes (EMF), welche den Dipolcharakter des Feldes anzeigt. Zu beachten ist auch, dass die Dipolachse (gestrichelte Linie) im Vergleich zur Rotationsachse der Erde geneigt ist (nach SCHNEPP 2007).

($30.000 - 60.000 \cdot 10^{-9}$ (nano) Tesla) compared with technically used magnetic fields. It is, for example, several hundred times weaker than the field between the poles of a toy horseshoe magnet (JACOBS 1994). Monitoring the EMF in observatories all over the world during the last 400 years demonstrates its dominant dipole character. This dipole is inclined by about 10° against the rotation axis of the Earth (Fig. 1) today. The main elements (components) of the geomagnetic field vector are declination (D) and inclination (I) measured in degrees, the intensity of the total field (F) described by the horizontal component (H) and the vertical component (Z) pointing downwards into the Earth on the northern hemisphere (Fig. 2a). Declination describes the deviation of a compass needle (magnetic north) from true (geographic) north. Inclination is the angle between the horizontal plane and the total field vector, measured positive into the Earth. At the magnetic poles, the magnetic needle of an inclinometer points vertically; the horizontal intensity is zero, while total intensity reaches the maximum. At the magnetic equator, the

inclination is zero and the horizontal component is about half of the intensity at the pole. Accordingly, inclination as well as F, H and Z depend on the geographic latitude (Fig. 2b).

The elements of the geomagnetic field vary on time scales ranging from days to tens of millions of years owing to complex magneto-hydrodynamic processes that generate the geodynamo in the outer liquid core of the earth. The extent of these temporal changes is dependent upon the location on Earth due to non-dipole sources of the EMF. In terms of directional changes with time, we have to differentiate between three features: the most prominent is called geomagnetic polarity reversal, meaning a 180° flip of declination and a change of sign of the inclination (Fig. 3), i.e. the dipole would point to the geographic North Pole in Figure 1. The duration of such a transition is in the order of 10^3 to 10^4 years. Superimposed to this relatively abrupt and large change during a reversal, is a background variability of angular variation (10-30°) called secular variation (SV). This movement of the geomagnetic elements occurs on time scales from 1 to 10^3 years. The third feature is called geomagnetic excursion. This describes an incomplete reversal or a short time interval of anomalously high secular variation (more than 30°). This phenomenon is not fully understood at present. The strength of the field vector (intensity) varies with time on the same time scales, but the long-term behaviour is not well understood, because the little data that is available is also poor. Secular variation of intensity is in the order of $\pm 50\%$ of the present field strength, while during excursions and reversals a strong decay in intensity down to about 10% of the present intensity is observed (MERRILL et al. 1996). Palaeointensity is much more difficult to measure and its palaeomagnetic determination needs more complicated procedures than determination of palaeodirection. While measurements of the palaeofield direction are quick and applicable to most kinds of rocks, special conditions are needed for a successful determination of the absolute palaeointensity.

It is the main task of palaeomagnetism to identify the ancient geomagnetic direction and in-

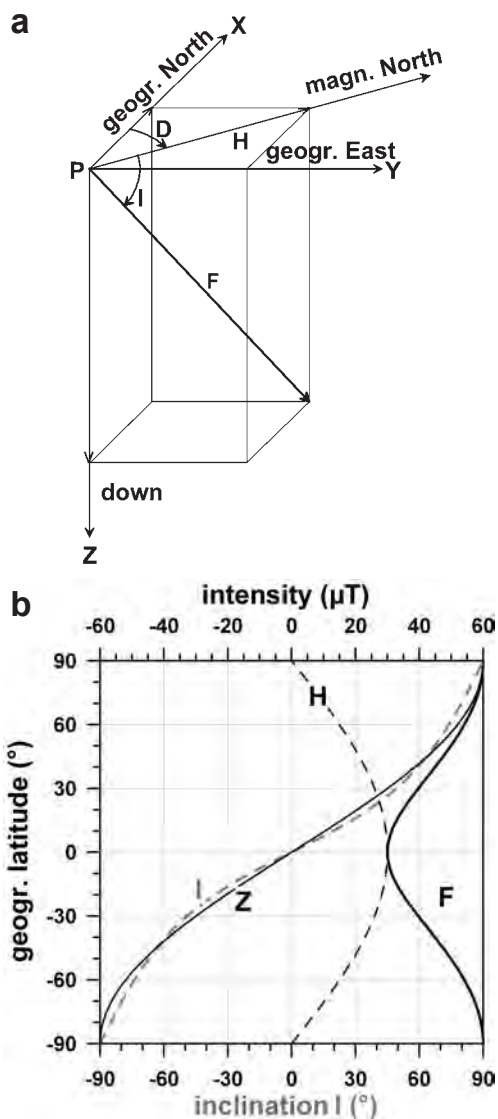


Fig. 2: (a) Earth magnetic elements shown in a Cartesian coordinate system at a point P of the Earth's surface (redrawn from SCHNEPP, 2007), (b) dependence of inclination (I) as well as total (F), horizontal (H) and vertical (Z) intensity (black) on geographic latitude.

Abb. 2: (a) Die erdmagnetischen Elemente sind in einem kartesischen Koordinatensystem für einen Punkt P auf der Erdoberfläche dargestellt (verändert nach SCHNEPP 2007); (b) zeigt den Zusammenhang zwischen Inklination (I) bzw. Total- (F), Horizontal- (H) und Vertikalintensität (Z, jeweils schwarz) und der geographischen Breite.

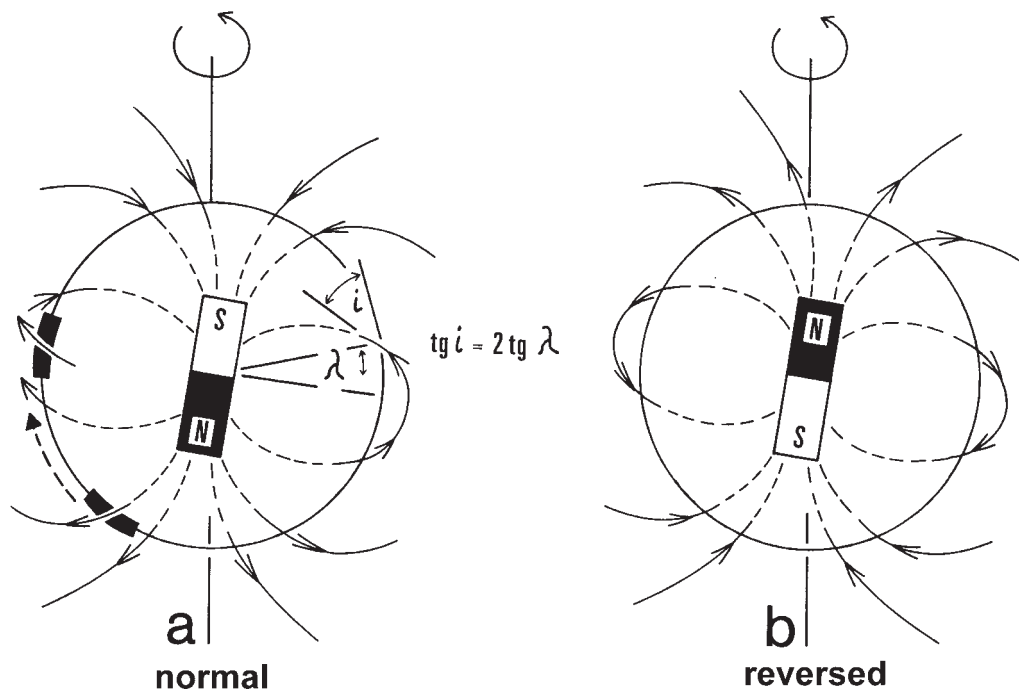


Fig. 3: Sketch of the Earth's magnetic dipole field in normal (a) and reversed (b) state. The bar magnet in the Earth's centre symbolizes the virtual dipole moment which is the main element of the EMF. Note the orientation of magnetic South and North. Also given is the dependence (dipole formula, see Fig. 2 (a)) of inclination (i) on geomagnetic latitude (λ). Redrawn from HAMBACH & KRUMSIEK (2000).

Abb. 3: Skizze des magnetischen Dipolfeldes der Erde mit normaler (a) bzw. inverser (b) Polarität. Der Stabmagnet im Erdmittelpunkt symbolisiert das axiale virtuelle Dipolmoment, den Hauptterm des EMFs. Zu beachten ist die Orientierung von magnetisch Süd und Nord. Weiterhin ist die Zusammenhang (Dipolformel, vergl. auch Abb. 2 (a)) von Inklination (i) und geographischer Breite (λ) angegeben (nach HAMBACH & KRUMSIEK, 2000).

tensity at the moment of formation of the rocks under investigation and to derive a chronology of temporal changes of these elements. This knowledge of the temporal geomagnetic field in the past offers chronometric possibilities on various geologic time scales. For chronological investigations, the use of quite short-lasting subchrons or even magnetic excursions is more effective than the use of chrons. In the following chapters, we will give examples and simple explanations for the use of the temporal change of elements of the geomagnetic field as dating tools in Quaternary research. In doing so, we will focus on examples from continental sedimentary archives and archaeomagnetic studies.

For a deepened insight on palaeomagnetism see, for example, BUTLER (1992) and on the temporal characteristics of the geomagnetic field we refer to CHANNELL et al. (2004a), GUBBINS (2008) and JACOBS (1994).

3 Remanence acquisition

All information carried by the main elements of the geomagnetic field described above can be stored in almost every rock type and up to ages of more than billions of years. The carriers of the magnetic memory in rocks are ferrimagnetic and antiferromagnetic minerals. These are iron minerals, exclusively iron oxides/hydroxides

and sulphides, with special lattice properties that enable the storage of magnetic information over geological time at ambient temperature. For simplicity, we will call them magnetic minerals in the following (*sensu strictu*). We will give only a short introduction into the processes responsible for remanence acquisition. For a detailed description, we refer to the literature mentioned above (e.g., BUTLER 1992).

3.1 Magmatic rocks

In magmatic rocks, as well as in heated archaeological materials, a so-called thermoremanent magnetization (TRM) or thermoremanence carries the ancient signal of the EMF. A TRM is produced by cooling to temperatures below the Curie temperature (CT) in the presence of a magnetic field. The CT is a mineral specific temperature, generally lower than 700° C, below which the minerals become magnetic. The TRM at surface temperature can be stable over geological time and resistant to magnetic fields after original cooling. Best long-term recorders are flood basalt provinces with long-lasting volcanic activity such as the Deccan traps in India (COURTILLOT et al. 1986) or volcanites on Gran Canaria (LEONHARDT et al. 2002). In case of rapid and frequent eruptions, it is even possible to record a complete change of polarity (LEONHARDT et al. 2002; LEONHARDT & SOFFEL 2002). A TRM can also be the result of a metamorphic overprint, if the reached temperatures and/or time spans are sufficient.

3.2 Archaeological materials

Because rapid cooling of lava flows takes place on time scales of hours to years, a lava flow provides a spot reading of the EMF elements, which is stored as the TRM. The same happens when a hearth, oven, kiln or any other artificially fired structure is heated and cooled down in the EMF. They will obtain a TRM parallel to the EMF. For soil, clay or loam, apart from detrital or diagenetic Fe-bearing minerals, new magnetic minerals are formed under the influence of fire and hence the magnetism of the material is consider-

ably enhanced (LE BORGNE 1955). The TRM carried by young lavas or archaeological material is normally very stable and only minor secondary magnetisation components are observed.

3.3 Sediments

Sediments are able to record information about the EMF by a depositional process called acquisition of post-depositional remanent magnetisation (PDRM). During deposition and diagenesis, detrital magnetic minerals may become aligned parallel to the EMF and the sediment acquires the primary PDRM. Most of the detrital minerals involved in this process acquired their primary magnetic moment by thermal processes and carry therefore a stable TRM. In comparison to the thermal remanent magnetisation described earlier, the PDRM is normally less intensive and stable (for fundamentals see e.g., HAMANO (1980) and TUCKER (1980)). There are several processes that may weaken the primary (quasi-synsedimentary) magnetisation, for example bioturbation, compaction or alteration of magnetic minerals by changing chemical environments. As described by several authors (e.g., BLEIL & VON DOBENECK 1999; EVANS & HELLER 2003; ROBERTS & WINKLHOFFER 2004) the most effective handicap against recording of short lasting subchrons is the lock-in mechanism. This mechanism describes the fixing of the magnetic remanence in sediments. Remanence locking does not happen immediately during deposition at or near to the sediment water interface, but needs a specific amount of sediment (lock-in depth) superposed to force dewatering, which in turn hinders grain rotation. The required lock-in time in case of fine grained clastics varies depending on grain size and sedimentation rate and was found to be in the order of 150 years in lake sediments with high sedimentation rates (STOCKHAUSEN 1998). Due to this process, it may happen that the lock-in depth (height of sediment necessary to fix magnetic remanence) needs more time to be collected than a subchron lasts. In that case, such a subchron may be entirely suppressed or masked. This is even more valid for the shorter

excursions of the magnetic field, which - if recorded and well dated - are excellent time markers.

The precipitation and growth of magnetic minerals during diagenesis and weathering is also an important process of remanence acquisition. This chemical process may lead to a stable so-called chemical remanent magnetisation (CRM). A CRM may occur in all kind of rocks at any time, if the environmental conditions are appropriate.

4 Methods in palaeomagnetic dating

Palaeomagnetic dating includes all approaches dealing with the temporal variation of the direction as well as with the intensity of the EMF on time scales from 10^2 to 10^7 years. On time scales of only a few million years, the well-known temporal pattern of reversals of the EMF provides an excellent tool for the subdivision of sedimentary sequences as well as volcanic rocks. Short-lived changes of the EMF (in the order of 10^3 years) in direction and intensity are called geomagnetic excursions, as stated above. If their chronology is known, they are perfect time markers, especially in sequences that are hardly datable by other methods. Records of the intensity variations of the EMF retrieved from sediments (relative palaeointensity), volcanic rocks and archaeological items (absolute palaeointensity) may also serve as dating tools and their application in stratigraphic studies is continuously increasing.

4.1 Magnetic polarity stratigraphy

Magnetic polarity stratigraphy or magnetostratigraphy is based on the temporal pattern of the reversals of the EMF as documented in the geomagnetic polarity timescale (GPTS). In the 1960s, it was found that sea floor spreading at mid-ocean ridges led to the characteristic bilateral symmetric 'stripes' of normal and reversed polarised oceanic crust. This pattern is measurable by ship based magnetometers and provides the basis for the GPTS (e.g., COX et al. 1967). The GPTS serves as the main 'levelling rule'

for the magnetic polarity stratigraphy from the recent back to the Early Jurassic (Fig. 4, 5).

Magnetic polarity stratigraphy is one of the excellent and independent tools (among others, e.g., oxygen isotope stratigraphy and orbitally tuned cyclo-stratigraphy) available for retrieving chronometric information, especially from sedimentary sequences. Magnetic polarity stratigraphy capitalises on the fact that the polarity of the EMF is recorded in rocks shortly after deposition. A distinction is drawn between chrons ($10^5 - 10^7$ years duration) and subchrons ($10^4 - 10^5$ years long). The first four chrons are usually named after famous geomagnetists (Brunhes, Matuyama, Gauss, Gilbert) and subchrons are named after the geographic location of their first discovery (Jaramillo, Olduvai). Both chrons and subchrons are helpful time markers, if recorded and classifiable (CHANNELL et al. 2004a, Fig. 5). The chronologic system used in magnetostratigraphy was determined simultaneously with its development. A GPTS (Fig. 5) labelled with names was originally based on magnetic remanence data from continental volcanic sequences dated with K-Ar ages and it is mainly used in investigations of Quaternary and Early Tertiary rocks (COX et al. 1967; MANKINEN & DALRYMPLE 1979). In contrast, a GPTS with a numerical system, based on the scheme used for labelling the marine magnetic anomalies, is used for roughly the last 160 Ma, the time span with substantial portions of oceanic crust remaining in ocean basins (CANDE & KENT 1995). The magnetic field anomalies are interpreted to be due to reversals of the geomagnetic field as recorded in the rocks of the oceanic crust (Fig. 4). A more detailed discussion about the nomenclature and evolution of the different GPTSs in use can be found e.g. in JACOBS (1994), McELHINNY & McFADDEN (2000) or OPDYKE & CHANNELL (1996). Combining GPTS and Milankovitch climate cyclicity improved application and led to a powerful geochronometer called the astronomical polarity timescale (APTS). APTS has improved accuracy of chronology for the Pliocene and Pleistocene and is now even used for calibrating high-precision radiometric dating (e.g., BASSINOT et al. 1994; KENT 1999).

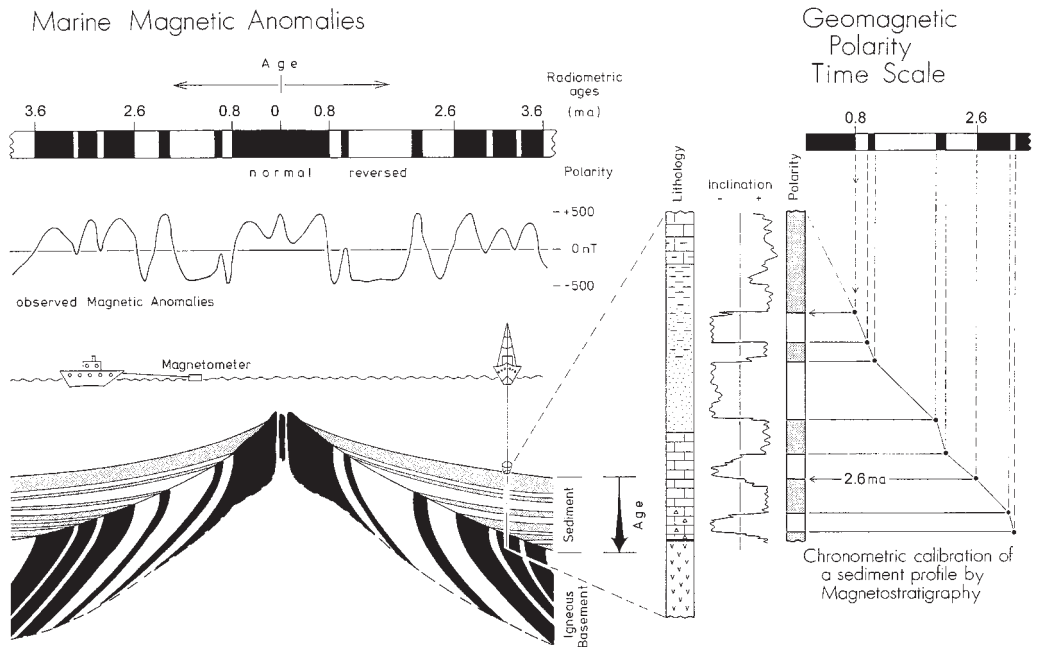


Fig. 4: Sea floor spreading at mid-ocean ridges leads to the characteristic bilateral symmetric 'stripes' of normal and reversed polarised oceanic crust. This pattern is measurable by ship-based magnetometers and provided the basis for the Geomagnetic Polarity Time Scale (GPTS). The GPTS serves as the 'levelling rule' for the magnetic polarity stratigraphy. From HAMBACH & KRUMSIEK (2000).

Abb. 4: Die Bildung von neuem Ozeanboden (sea floor spreading) an den mittelozeanischen Rücken führt zu den charakteristischen, bilateralen, symmetrischen 'Streifen' von normal und invers magnetisierter ozeanischer Kruste. Dieses Muster wird mit Seemagnetik gemessen und lieferte die Basis für die geomagnetische Polaritätszeitskala (GPTS). Die Magnetostratigraphie benutzt die GPTS zur Kalibrierung der Zeit (aus HAMBACH & KRUMSIEK, 2000).

4.1.1 Magnetic polarity stratigraphy in sedimentary archives

With respect to sediments, the situation is generally complex. Firstly, in limnic and marine sediments, conditions for recording the complete succession of changes in the EMF prevail frequently (CHANNELL 1999; CLEMENT & KENT 1987; FRANK et al. 2007; NOWACZYK et al. 1994), in contrast to fluvial continental sedimentation, which is very often far from being continuous. In their milestone publication about Chinese loess, HELLER & LIU (1982) were able to demonstrate the great potential of Quaternary continental sediments for magnetostratigraphy. This was the starting point for

a great variety of studies in Quaternary sediments all over the world (e.g., LANGEREIS et al. 1997; ROLPH 1993; RÖSLER et al. 1997; WORM et al. 1998; see textbooks by EVANS & HELLER 2003 and OPDYKE & CHANNELL 1996). Secondly, remanence lock-in of sediments (see chapter 3.1) does not happen immediately after deposition but needs a specific amount of sediment superposed to force dewatering (e.g., BLEIL & VON DOBENECK 1999; BUTLER 1992; EVANS & HELLER 2003; TAUXE 1998).

In the following, we will discuss some exemplary studies in which magnetic polarity stratigraphy was successfully applied to continental sediments. Though there have been many attempts to date Quaternary sediments

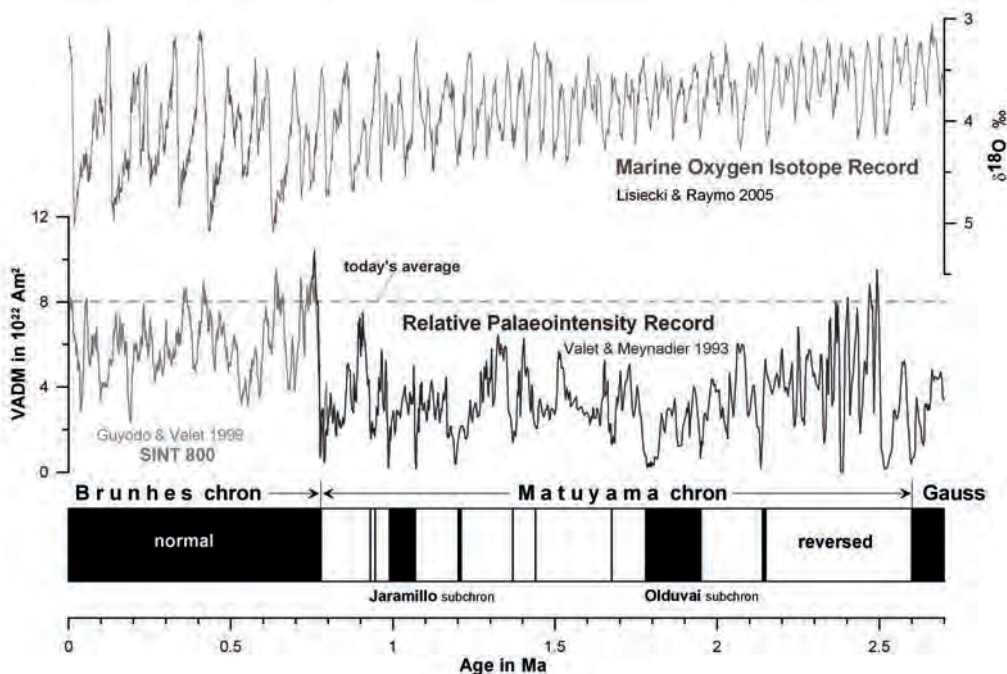


Fig. 5: The GPTS of the Quaternary is characterised by the normal Brunhes and the reversed Matuyama chron, of which the latter is interrupted by several subchrons. A record of Earth's magnetic axial dipole moment (VDAM) temporal variations is also shown. Note that the minima in RPI go together with polarity changes and that today's dipole moment is relatively high compared to most of the length of the Quaternary. The marine oxygen isotope record shows the evolution of the global ice volume and thus of the global climate. Data from CHANNELL et al. (2004a) [GPTS], VALET & MEYNADIER (1993) and GUYODO & VALET (1999) [relative palaeointensity, RPI] and LISIECKI & RAYMO (2005) [marine oxygen isotopes].

Abb. 5: Die GPTS des Quartärs unterteilt sich im Wesentlichen in die normale Brunhes- und die inverse Matuyama-Chrone, letztere ist von mehreren Subchronen unterbrochen. Eine Kurve der zeitlichen Änderung des Dipolmoments der Erde ist ebenfalls dargestellt. Man beachte das zeitgleiche Auftreten von Polaritätswechseln und Minima des Dipolmoments. Weiterhin beachtenswert ist die Stärke des heutigen Dipolmoments der Erde im Vergleich zu der während des Quartärs. Die marine Sauerstoffisotopenkurve zeigt die Entwicklung der globalen Eisvolumina und somit des globalen Klimas. Daten von CHANNELL et al. (2004a) [GPTS], VALET & MEYNADIER (1993) und GUYODO & VALET (1999) [relative Paläointensität, RPI] and LISIECKI & RAYMO (2005) [marine oxygen isotopes].

by palaeomagnetic methods in the last decades, the success story of magnetic polarity dating in continental Quaternary sequences started with the seminal work of Friedrich Heller and Tung-Sheng Liu assigning the Chinese loess to the Quaternary time table and correlating it with the global ice volume record (HELLER & LIU 1982, 1984). For further reading we refer to EVANS & HELLER (2003) and HELLER & EVANS (1996). In active orogenic belts such as the Alpine range, sedimentary sequences very often reveal

only incomplete records representing short time intervals. Even though a polarity change is identified, due to the indistinguishability of magnetic polarity patterns for the last 3 million years (mainly because of negligible small tectonic movements of the European plate) the acquired data cannot be unambiguously assigned to any interval of the GPTS without further chronological information. Magnetic polarity stratigraphy on glacial sediments of the Iller-Lech-Platte showed problems in determination

of palaeodirections for these sediments carrying a remanence of weak intensity and difficulties on stratigraphic interpretation due to incompleteness of the profiles. However, it was possible to find the Matuyama-Brunhes boundary in two profiles on the Zusam gravel plain and to date it to older than 780 ka (STRATTNER & ROLF 1995).

WORM et al. (1998) studied a more than 4 km thick succession of delta sediments from Bangladesh that was devoid of firm dating through biostratigraphy or other methods. Applying classical magnetic polarity stratigraphy, a Lower Pleistocene to Lower Pliocene age could be assigned for the whole sequence. This new and reliable age model inferred a sedimentation rate of 1.2 m ka^{-1} , which appears to be one of the highest sedimentation rates in Earth history that was sustained for millions of years.

Pleistocene fluvial sequences in the eastern part of the Pannonian Basin were studied in order to establish a palaeoclimatic framework for the region (NADOR et al. 2003). Magnetostratigraphy indicates that the sediment sequences span the last 2.6 Ma without significant hiatuses (COOKE et al. 1979). Comparison of systematic variations in magnetic susceptibility with changes in grain size distribution revealed Milankovitch-scale cyclicity in sedimentation. Comparison of pollen and gastropod paleoecological data with the magnetic susceptibility proxies suggests that climate was a major allogenic control factor on the cyclicity. This study convincingly demonstrates the potential of magnetic dating techniques even in fluvial sequences, which are important palaeoclimate archives on the continents.

The Quaternary stratigraphy of the North Sea and surrounding areas received essential support through magnetostratigraphic investigations. MAHER & HALLAM (2005) were able to determine stable magnetic polarities from discontinuous sediment sequences, of Plio/Pleistocene age, occurring onshore around the southern North Sea margins. The reliable palaeomagnetic polarity determinations were used, in combination with biostratigraphic data and in comparison with the GPTS, to ascribe correlations and absolute ages, which shed a

new light, especially on the Quaternary stratigraphy of East Anglia.

The lower Rhine Embayment and the Rhine-Meuse-Delta in the Netherlands are complex sedimentary systems which recorded environmental changes of the Rhine catchment including even parts of the Alps (PREUSSER 2008). Their chronology is largely based on magnetostratigraphic studies (e.g. BOENIGK & FRECHEN 2005; ZAGWIJN 1992), which provide the only time constraints especially for the Lower Pleistocene.

Magnetostratigraphic and rock magnetic investigations performed on fluvial sediments from the Ludwigshafen-Parkinsel drilling project (Upper Rhine Graben (URG), Germany) show the change from mainly locally controlled sedimentation from the graben margins to a more distinct alpine controlled sedimentation at a depth of 177 m by magnetic data (ROLF et al. 2008). Based on lithostratigraphic correlation with other sedimentary records from the URG and also based on palynological evidence, this event happened at the end of the Late Pliocene at the top of a normally polarised core interval (end of the Gauss Chron?). The well documented characteristic change in magnetomineralogy from goethite to greigite almost at the same stratigraphic level, was interpreted solely as a climatic signal, which can be correlated with the global climate change at ~ 2.6 Ma that is well documented in a wide range of sedimentary environments (e.g. deep-sea sediments, loess).

The chronology of early humans in Europe is intrinsically tied to the results of palaeomagnetic investigations at the excavation sites. From Dmanisi, Georgia, several findings of *Homo erectus* are reported from sediments containing a reversal and resting on normally polarised basalt. Supported by the faunal context, an age slightly younger than the Olduvai subchron was inferred making these human remains from Dmanisi the oldest in Eurasia (GABUNIA et al. 2000). At Atapuerca, Spain, human remains (*Homo erectus?*) were found in cave debris that are reversely polarised. Combined bio- and magnetostratigraphy prove here

the existence of early humans already well before the Matuyama-Brunhes boundary (PARES & PEREZ-GONZALEZ 1995). Also, the age of the earliest “German”, the *Homo heidelbergensis* was constrained by palaeomagnetism. The investigation of several sedimentary units above and below the sand beds in which the jaw was found exhibit only normal polarity, assigning this beds doubtless to the Brunhes chron (HAMBACH et al. 1992; WAGNER et al. 1997).

4.1.2 Magnetic polarity dating of magmatic rocks

Volcanic rocks are normally stable and firm recorders of the EMF. Yet due to the relatively short duration of volcanic activities compared to quasi-continuous recorders such as sediments, the magnetic information very often only represents spot-readings of the EMF and shorter reversals were frequently not recorded. If by chance a volcano was active during the life-time of an excursion or subchron, it might be possible to identify them (see chapter 4.1.3).

The volcanic oceanic crust represents by far the most important archive for the history of the EMF, going back to about 160 Ma at least. Sea floor spreading at mid-ocean ridges led to the characteristic bilateral symmetric ‘stripes’ of normal and reversed polarised oceanic crust. This pattern is measurable by ship-based magnetometers and provided the basis for the Geomagnetic Polarity Time Scale (GPTS) (Fig. 4). The GPTS serves as the ‘levelling rule’ for the magnetic polarity stratigraphy and its development started in the sixties of the last century. The beginning, however, was the work of Allen Cox who systematically measured volcanic rocks, which were dated by the contemporaneously improved K-Ar dating techniques. His early GPTS covered only the last 5 Ma, but allowed the determination of spreading rates, which in turn lead to the extrapolation of the GPTS back to the Early Jurassic (COX et al. 1967; OPDYKE & CHANNELL 1996).

There are numerous magnetostratigraphic studies of volcanic sequences on continents. We would like to highlight the extensive work that

was done on the Deccan traps in India (COURTILLOT et al. 1986). Only magnetic polarity was able to prove the unexpected short eruption interval for this immense amount of lava. In only about 1 Ma, exactly around the Cretaceous-Tertiary boundary, the complete succession of trap basalts were erupted and possibly contributed to the vast extinction event at the end of the Mesozoic.

LEONHARDT et al. (2002) studied a continuous series of 87 lava flows from the mid-Miocene shield basalts of Gran Canaria. The sequence covers a reversal of the EMF from reverse to normal. Intermediate directions of the field are recorded in 34 lava flows and in addition to the reversal, three excursions (see chapter 4.1.3) are recorded. The results impressively demonstrate the potential of magnetic polarity studies for temporally highly resolved volcanic sequences and their relevance for understanding the processes controlling the geodynamo. Conventional K-Ar dating techniques are unable to resolve short-lived volcanic events and very often suggest continuous volcanic activity in a given time span. Palaeomagnetic data from the Quaternary alkali basaltic volcanic field of the Perşani Mountains, Romania, revealed sites with normal, transitional (excursion) and reversed polarity (Fig. 6). The combination of K-Ar dating with palaeomagnetic polarity determinations allowed the discrimination of short-lived volcanic phases of only a few thousand years duration, well beyond the precision of K-Ar dating (PANAIOTU et al. 2004).

4.1.3 Geomagnetic Excursions as time markers

As discussed above, the shape of the EMF varies on time scales ranging from days to tens of millions of years. Long periodic amplitudinal and directional variations as polarity changes and even polarity transitions, as well as medium to long periodic secular variations (see chapter 4.2) are relatively well-known phenomena (MERRILL et al. 1996). However, prominent short-lived features (< 5 ka) such as magnetic polarity episodes and reversal excursions are only rarely

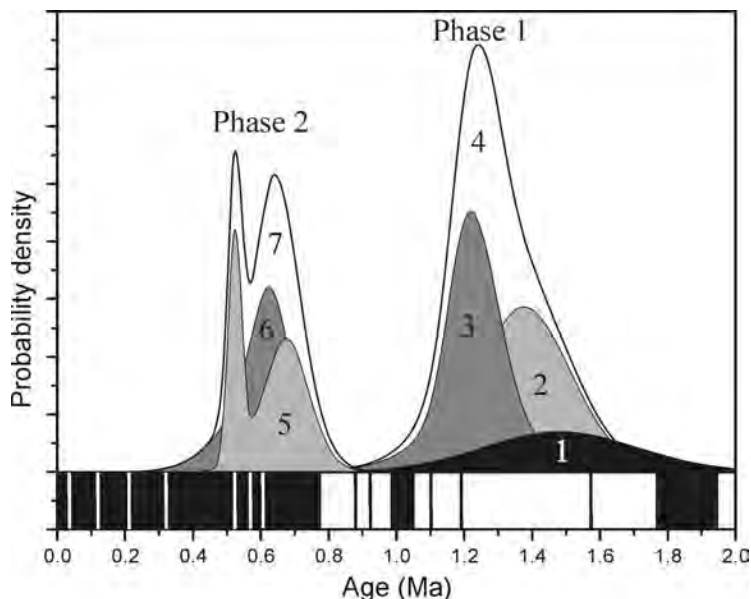


Fig. 6: Distribution of K-Ar ages from the Perșani Mountains, Romania. This alkali basaltic volcanic field revealed sites with normal, transitional (excursional) and reversed polarity. The individual density distributions display populations of normal, transitional (excursional) and reversed polarity sites separated (1-3, 5-6) and combined (4, 7). The combination of K-Ar dating with palaeomagnetic polarity determinations allows the discrimination of short-lived volcanic phases of only a few thousand years duration, well beyond the precision of K-Ar dating. Thin white and black bands are short lived polarity excursions (white) or subchrons (black, Matuyama). From PANAIOTU et al. (2004).

Abb. 6: Verteilung der K-Ar-Alter der Perșani Berge, Rumänien. Dieses alkalibasaltische Vulkanfeld enthält Vulkanite mit normaler, transitoinaler (exkursionaler) und inverser Polarität. Die Verteilungen zeigen Populationen mit normaler, transitoinaler (exkursionaler) und inverser Polarität im Einzelnen (1-3, 5-6) bzw. die Gesamtverteilungen (4, 7). Die Kombination von K-Ar-Altern und der Bestimmung der magnetischen Polarität erlaubt die Unterscheidung von kurzen vulkanischen Phasen mit nur einigen Tausend Jahren Dauer, die durch die K-Ar-Datierung allein nicht aufgelöst werden können. Schmale weiße und schwarze Bänder sind kurzlebige Polaritätsexkursionen (weiß) oder Subchrons (schwarz, Matuyama). Aus PANAIOTU et al. (2004).

documented. These rare features of the EMF, so-called geomagnetic events, are characterised by large deviations from the dipole direction often associated with a low relative palaeointensity. Most of them are found in the Brunhes chron (since 780 ka ago) where several have been documented in sedimentary and volcanic rocks. The reports include short-term reversals, which probably occur globally (e.g. Blake polarity episode), and reversal excursions whose extension is possibly regionally restricted (e.g. Fram Strait episode?). The significance and interpretation of geomagnetic events, and to a certain extent even

their interpretation as real manifestations of the EMF, is still open to debate (LAJ & CHANNELL, 2007). Geomagnetic excursions probably originate from incomplete reversals that employ only a polarity change in the liquid outer, but not in the solid inner core (GUBBINS 1999). Thus, the older picture of the geomagnetic field having long periods of stable polarity may not be correct; instead, the field appears to suffer many dramatic changes in direction and reduction in intensity.

The most significant progress in our understanding of temporal variations of the EMF

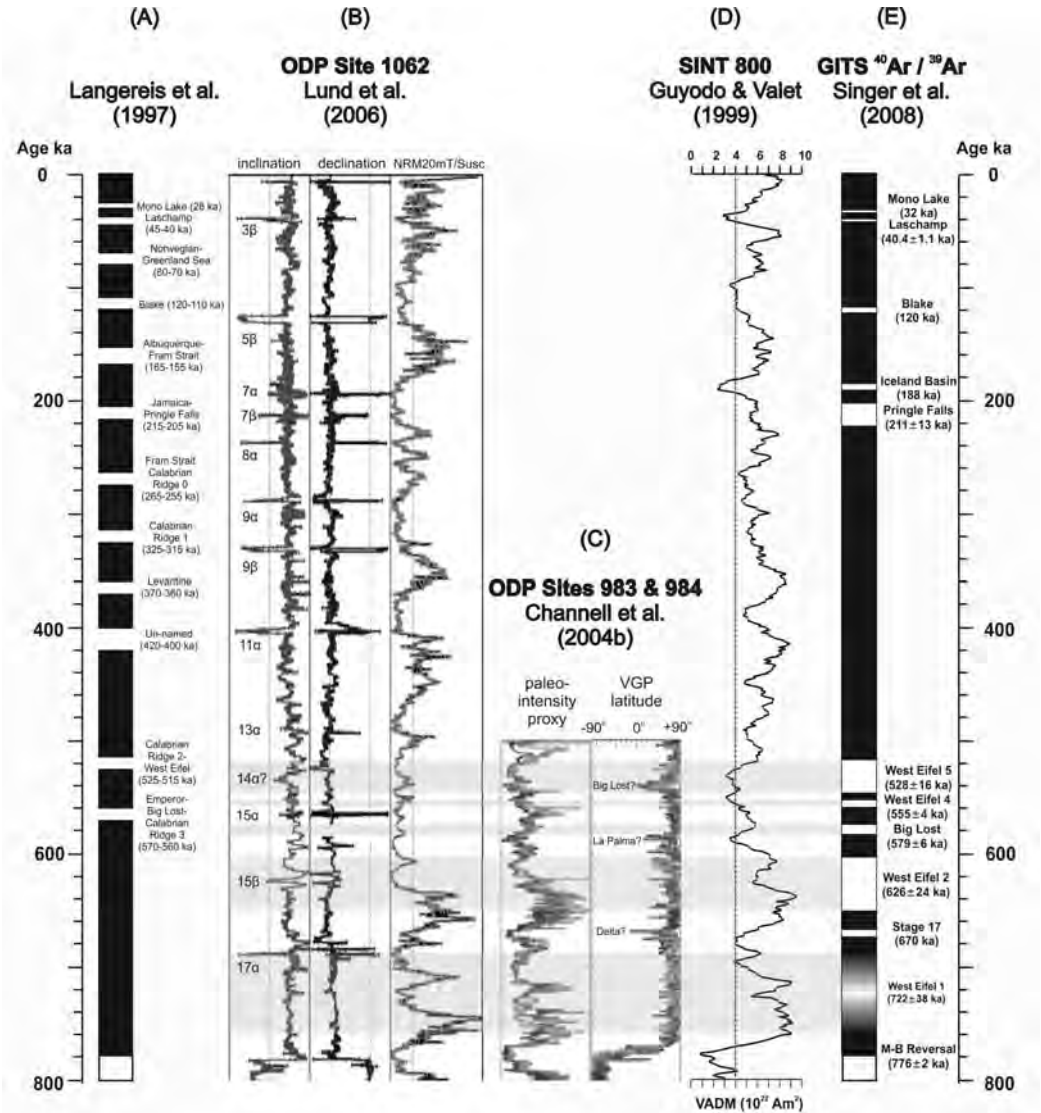


Fig. 7: Records of geomagnetic excursions and palaeointensity during the Brunhes Chron. Records are from: A) LANGEREIS et al. (1997), B) LUND et al. (2006), C) CHANNELL et al. (2004b), D) GUYODO & VALET (1999), and E) Geomagnetic Instability Time Scale (GITS) based on $^{40}\text{Ar}/^{39}\text{Ar}$ dated volcanic rocks plus excursions in marine sediment cores that have been dated via continuous oxygen isotope-based astrochronology (e.g. CHANNELL et al. 2004a). Redrawn from SINGER et al. (2008).

Abb. 7: Aufzeichnungen der geomagnetischen Exkursionen während der Brunhes-Chron. Die Kurven stammen von: A) LANGEREIS et al. (1997), B) LUND et al. (2006), C) CHANNELL et al. (2004b), D) GUYODO & VALET (1999), und E) Geomagnetische Instabilitätszeitskala (GITS) basierend auf $^{40}\text{Ar}/^{39}\text{Ar}$ datierten vulkanischen Gesteinen und Exkursionen aufgezeichnet in marinen Sedimentkernen, die mit einer kontinuierlichen astrochronologischen Sauerstoffisotopenkurve datiert wurden (z.B. CHANNELL et al. 2004a). Nach SINGER et al. (2008).

during the last decade stems from marine sedimentary records. This comes from relative palaeointensity curves in particular (see chapter 4.3), but some directional records also suggest that the processes that generate the EMF are highly dynamic and that temporal variations occur on a much shorter time scale than previously thought possible (GUYODO & VALET 1999; LUND et al. 2006; NOWACZYK et al. 1994). However, while changes of the virtual axial dipole moment (VADM) account for the global nature of apparently most of the (relative) minima of the relative palaeointensity variations, not all geomagnetic events or excursions in the Brunhes chron could be traced globally (Fig. 7). Nevertheless, combined evidence suggest that at least 10 polarity events or excursions exist in the Brunhes chron, which are often not primarily preserved and / or diagenetically overprinted in most of sedimentary environments due to the palaeoclimatic conditions under which they were recorded (e.g., BLEIL & VON DOBENECK 1999; LANGEREIS et al. 1997). However, an impressive record of well documented excursions has been published in the last years. LANGEREIS et al. (1997) gave a thorough review of Brunhes age excursions and reported several new ones from a detailed study of an eastern Mediterranean piston core. Precisely calibrated astronomical reversal boundaries and ages for short-lived subchrons and polarity episodes in the Matuyama chron were published by HORNG et al. (2002). The Ocean Drilling Program (ODP) recovered many long sediment sequences from around the world that contain medium- to high-resolution paleomagnetic records of Brunhes ages. LUND et al. (2006) collected the evidence for 17 excursions younger than the last reversal from these records and provided accurate ages based on oxygen isotope chronostratigraphies. From a marine core off Portugal going back to 400 ka, THOUVENY et al. (2004) documented eight lows in relative palaeointensity. Four of these lows mark already well-known events and were also accompanied by directional excursions, which points to their global nature. From the ODP site 919, located in the Northern Atlantic, CHANNELL (2006) found additional ev-

idence for four already well documented excursions of the last 250 ka. This interval contains at least five polarity episodes with global evidence. From young to old these are: Mono Lake (32-34 ka), Laschamp (40-42 ka), Blake (115-120 ka), Icelandic Basin (~185 ka) and Pringle Falls (205-225 ka) (CHANNELL 2006). In recent years, Brad Singer pointed out the instability of the dominant normal polarity of the Brunhes chron. Singer and his co-workers applied precise $^{40}\text{Ar}/^{39}\text{Ar}$ isochron dating on volcanic rocks containing excursions palaeomagnetic records. Combining their data with records from sediments, they constructed a Geomagnetic Instability Time Scale (GITS) for the Brunhes Chron (SINGER et al. 2008, Fig. 7).

Geomagnetic excursions have even been reported from loess profiles. The earliest evidence dates back to the seventies and was later confirmed by numerous papers published in the mid 1990s (e.g., REINDERS & HAMBACH 1995, and references therein; EVANS & HELLER 2001, for review and further reading). Recently, PILIPENKO et al. (2006) and ZHU et al. (2006) presented records containing excursions directions from loess sections in the Ukraine and the Chinese loess plateau, proving that even loess units can record short-term features of the EMF.

We will not provide any listing of Brunhes excursions that could be useful stratigraphic markers, because their exact ages and timing as well as their global or only regional nature are still under debate. Therefore, we would like to draw the reader's attention to some recently published papers (e.g., CHANNELL 2006; LUND et al. 2006; SINGER et al. 2008) and we encourage them to follow the forthcoming relevant publications.

In the context of geomagnetic excursions, the possible link between climate and EMF is being increasingly addressed. Already WORM (1997) suggested "a link between geomagnetic reversals and events and glaciations" following the observation of a concomitant incidence of geomagnetic polarity events and sediments indicating periods of cold climate. The occurrence of excursions and reversals with the obliquity signal of the Earth's orbit was tested by FULLER (2006). His results have shown that

the strongest minima in the palaeomagnetic intensity records correlate with obliquity minima and suggest that precession provides some power to the geodynamo. In their study of a well dated marine core off Portugal, THOUVENY et al. (2004) made the interesting observation that relative palaeointensity lows often coincide with the end of interglacial or interstadial stages. They found, furthermore, a dominant period in the relative palaeointensity record of about 100 ka and a phase shift between the $\delta^{18}\text{O}$ and the relative palaeointensity record in the order of 18 ka. Both findings may suggest the dependence of Earth's climate system and EMF upon the same driving process.

However, possible interactions between the Earth's climate system, the geodynamo and solar activity can presently not be proven. Further evidence and models have to be awaited. The papers of COURTILLOT et al. (2007) and FULLER (2006) give an up-to-date summary of the current state of discussion.

4.2 Secular variation

The shortest with palaeomagnetic techniques resolvable temporal variation of the EMF is a lower angular variation (10-30°) called secular variation (SV) occurring on time scales from 1 to 10^3 years. The SV of the EMF provides insight into processes in the outer liquid core of the Earth. Therefore, considerable effort has been undertaken to map the palaeomagnetic SV with high temporal resolution over long time-intervals from sites all over the world (e.g., LUND 1996; STOCKHAUSEN 1998). However, even for a relatively large area such as Europe, only few records exist that are considerable longer than the Holocene, or extend into the early Upper Pleistocene (BRANDT et al. 1999; THOUVENY et al. 1990).

SV is observed today by magnetic observatories, which are spread over the globe as well as by satellites, i.e. CHAMP or OERSTEDT (OLSEN et al. 2006). Historic measurements of SV go back in time only 400 years (JACKSON et al. 2000), whereas archaeomagnetic data cover the past two or three millennia for Europe (DE

MARCO et al. 2008a; GALLET et al. 2002; GÓMEZ-PACCARD et al. 2006a, b; MÁRTON & FERENCZ 2006; SCHNEPP & LANOS 2005, 2006; TEMA et al. 2006; ZANANIRI et al. 2007) and for America (BOWLES et al. 2002, STERNBERG 1989) and go back about 8000 years for a few places, i.e. Bulgaria (KOVACHEVA et al. 1998) (Fig. 9). Secular variation master curves have been obtained from many places on Earth with a broad range of time resolution and directional fidelity. An overview of the data sets obtained for the past 7000 years was given by (KORTE et al. 2005). Well-defined archaeomagnetic secular variation curves have time resolutions in the order of 50-100 years. SV data from sediments often cover much longer time intervals, but here the time resolution as well as preciseness of the direction is lower (Fig. 8). While the so-called archaeomagnetic dating, which is also possible for lavas (TANGUY et al. 2003), provides a date with error margins (LANOS 2004; LE GOFF et al. 2002), SV dating for lake sediments uses wiggle matching in order to correlate sedimentary profiles.

4.2.1 Dating with secular variation

Dating with secular variation (SV) is possible on time scales from decades to millennia for the past 50 ka. It can be applied to young volcanic rocks, lake sediments with high sedimentation rates, baked in-situ archaeological structures and, with limitations, to displaced ceramic material.

The dating process depends strongly on the material to be dated. Continuous data series obtained from sediments require wiggle matching techniques in order to replace depth by a time scale calibrated with SV patterns, whereas in contrast, volcanic rocks or archaeological materials provide spot readings of the direction (intensity), which allow calculation of a probability distribution of age, but does not necessarily provide a single date.

4.2.1.2 Archaeomagnetic dating

Archaeomagnetic dating uses detailed regional calibration curves with an error envelope (Fig.

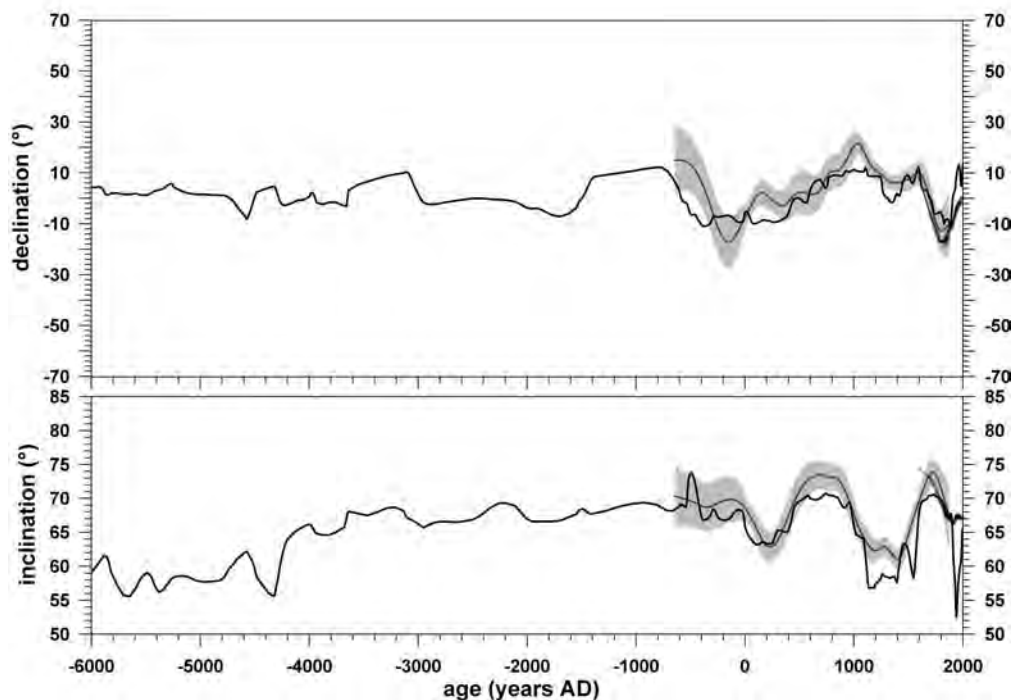


Fig. 8: Declination and inclination (black curves) for the past 6000 years obtained from paleomagnetic measurements of the Holzmaar sediments (STOCKHAUSEN 1998). The curves are shown in comparison with the German archaeomagnetic secular variation curve (SCHNEPP & LANOS 2005, see also Fig. 9, grey curves with 95% error envelope). In order to match the variation pattern, the time scale of the sediment curve was shifted by 125 years to younger dates.

Abb. 8: Deklination und Inklination (schwarze Kurven) der vergangenen 6000 Jahre, die aus paläomagnetischen Untersuchungen der Holzmaarsedimente gewonnen wurden (STOCKHAUSEN 1998). Die Kurven sind im Vergleich mit der deutschen archäomagnetischen Säkularvariationskurve (SCHNEPP & LANOS 2005, siehe auch Abb. 9, graue Kurven mit 95% Fehlerband) gezeigt. Damit die Variationsmuster der Sediment- und der archäomagnetischen Kurven zusammenpassen, wurde die Sedimentkurve um 125 Jahre zu jüngeren Altern hin verschoben.

8). This dating compares directions and intensities of unknown age against the curves and a probability distribution of age is obtained. Additional constraints (archaeological finds, stratigraphy) allow a reduction in ambiguity. Archaeomagnetic dating can be applied to in-situ fired structures and, with limitations, also to displaced material as potsherds or bricks. Whereas the polarity time scale is valid for the complete Earth, because a polarity reversal is a worldwide phenomenon, secular variations show regional patterns that cannot be transferred over very far distances (CASAS &

INCORONATO 2007). Accordingly, a reference curve is required, which is valid for the region in which the archaeological structure was found or in which the displaced material was produced; for archaeomagnetic dating applications. Reference curves must be established first by studying the direction and/or intensity of well dated archaeological material for a certain region, before archaeomagnetic dating is possible (e.g., DE MARCO et al. 2008b).

Due to the lock-in mechanism in sediments mentioned above, a shift between archaeomagnetic SV variation curves and sedimentary

secular variation curves is observed, which has to be taken into account, if time resolutions below 500 years are required.

Besides archaeomagnetic reference curves, which are made for dating in specific regions, global models of the EMF are available (KORTE & CONSTABLE 2005; KORTE et al. 2005), which allow the calculation of secular variation curves for any place. The use of such model curves for dating purposes results in very uncertain ages. This has to be avoided for all regions from which no or only few data exist for the requested time interval.

4.2.1.3 Secular variation dating in sedimentary archives

In limnic and marine sediments, conditions for recording the complete succession of temporal changes of the EMF prevail frequently over diagenesis and other secondary processes. Normally, two questions are addressed before a palaeomagnetic record is analysed with respect to the underlying physical processes. Firstly, the crucial determination of a depth to time transformation to construct a well-defined time-series usually requires a multi-disciplinary approach, and several years, too. The second question addresses the nature of the record: how and when was the natural remanent magnetisation acquired; is it contemporaneous with the sedimentary matrix? If not, what is the time lag between sedimentation and remanence acquisition? In other words: is it possible to verify that a geological archive carries a record of the past variations of the EMF? Conventional techniques such as the stacking of multiple data from one lake (sedimentary environment) are used for such an evaluation. Supplementary arguments are then drawn from rock magnetic studies to construct plausibility arguments as to the origin of the stacked signal.

Warved sediments from the Eifel Maar lakes have been successfully used to retrieve reliable SV records for geomagnetic as well as for stratigraphic studies. The best record of the Holocene SV for Central Europe was derived from a thorough study of three Maar lakes from the

West-Eifel (Germany) (STOCKHAUSEN 1998, see Fig. 8). ZOLITSCHKA et al. (2000) applied multiple dating methods and interlake comparison in order to establish a robust chronology for a Late Weichselian continental paleoclimate record from the Eifel Maar Lakes. Dating by means of SV serves here for the chronostratigraphic frame especially for the older part (10 – 23 ka) of the succession.

REINDERS et al. (submitted) presented the so far “oldest” SV record for Germany from cores of the dry Maar Jungfernweiher in the southern West Eifel Volcanic Field. The record covers the time interval from approx. 60 – 100 ka and revealed a time interval of low SV which could be used to correlate the record to the SV records of Lac du Bouchet in Central France as well as to Lago Grande de Montichio in South Italy by wiggle matching (BRANDT et al. 1999; THOUVENY et al. 1990).

4.3 Dating by means of relative palaeointensity records

Not only the direction, but also the intensity of the EMF at a given point at the Earth’s surface provides important information about the EMF itself. As the direction, the intensity also undergoes temporal variations. During a polarity reversal, the intensity goes down, probably to less than 10 % of the average values before or after the reversal (e.g., JACOBS 1994). The EMF also weakens dramatically (Fig. 5 and 7) during geomagnetic excursions (see chapter 4.1.3). The variation of the EMF intensity within the Brunhes normal chron is quite well-known and provides a tool for relative stratigraphic dating. Absolute palaeointensity can be determined from magmatic rocks as well as from heated archaeological materials by simulating the remanence acquisition process in the laboratory (e.g., SCHNEPP & HRADETZKY, 1994). In sediments, however, only a relative approximation for the intensity of the EMF at the time shortly after deposition is possible. For this purpose, the determined magnetic remanence is normalised by a measure for the concentration of magnetisable minerals in the sediment (TAUXE

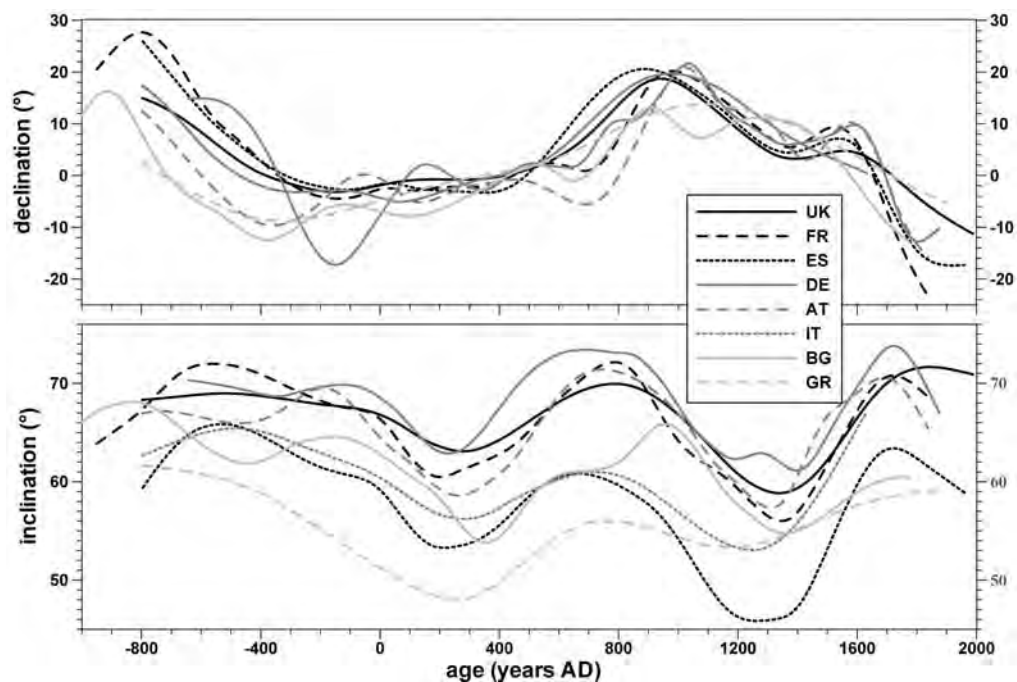


Fig. 9: Declination and inclination for archaeomagnetic secular variation reference curves are plotted versus time for several European countries: United Kingdom (UK, ZANANIRI et al. 2007), France (FR) and Spain (ES, GÓMEZ-PACCARD et al., 2006a, b), Germany (DE, SCHNEPP & LANOS 2005), Austria (AT, SCHNEPP & LANOS 2006), Italy (IT, TEMA et al. 2006), Bulgaria (BG, LANOS 2004) and Greece (GR, DE MARCO et al., 2008a).

Abb. 9: Deklination und Inklination aufgetragen gegen die Zeit der archäomagnetischen Säkularvariationsreferenzkurven für verschiedenen europäische Länder: Vereinigtes Königreich (UK, ZANANIRI et al. 2007), Frankreich (FR) und Spanien (ES, GÓMEZ-PACCARD et al. 2006a, b), Deutschland (DE, SCHNEPP & LANOS 2005), Österreich (AT, SCHNEPP & LANOS 2006), Italien (IT, TEMA et al. 2006), Bulgarien (BG, LANOS 2004), und Griechenland (GR, DE MARCO et al. 2008a).

1998). The result is a relative palaeointensity (RPI) record that shows variations in the order of ten thousands to thousands of years (Fig. 5). VALET & MEYNADIER (1993) published one of the first long RPI records, which goes back 4 Ma and covers the whole Quaternary. For the past 800 ka, GUOYODO & VALET (1999) built a stack of globally distributed marine RPI records. This synthetic intensity record (SINT 800) is the best 'master curve' presently available on this timescale (Fig. 5). Short-lived variations in SINT 800, however, seem to be suppressed due to the stacking process. A very detailed data set with high temporal resolution covering the last 75 ka for the North Atlantic

realm (North Atlantic Palaeointensity Stack, NAPIS) was published by LAJ et al. (2000) and was extended later on to a global stack (Global Palaeointensity Stack, GLOPIS, LAJ et al. 2004). High-resolution reconstruction of cosmogenic nuclide concentrations along sedimentary archives is an alternative approach for documenting past magnetic field changes. This approach is being increasingly applied in the study of marine as well as ice cores (e.g. CARCAILLET et al. 2004).

Recent studies of high-resolution RPI records from European loess sites contain evidence for multiple RPI lows between approximately 22 and 45 ka, including the "Mono Lake" and

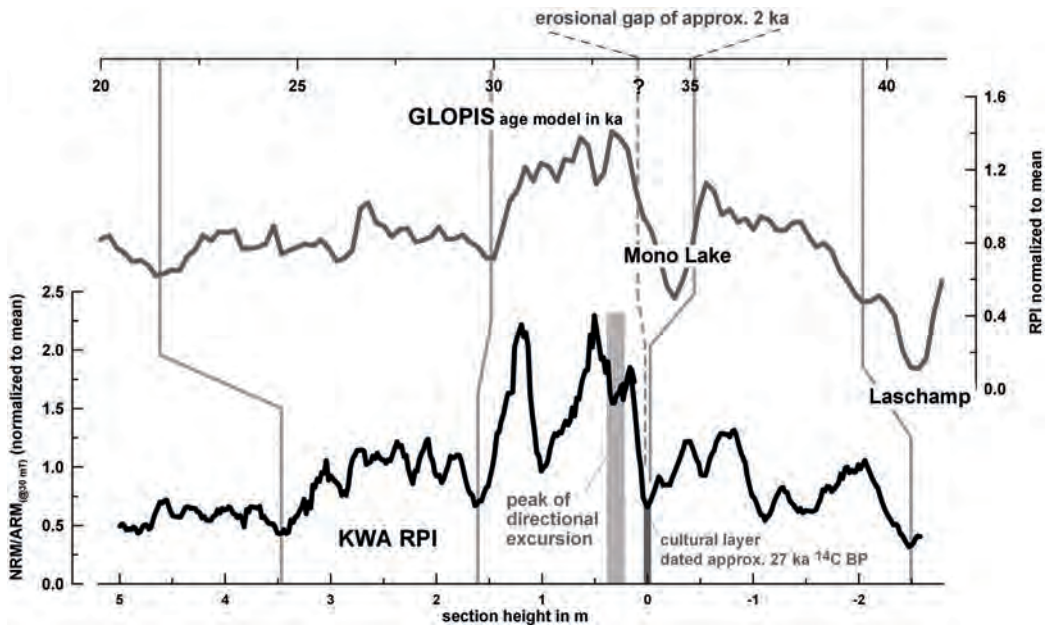


Fig. 10: A relative palaeointensity (RPI) record from a loess section in Krems (Lower Austria) and the Global Palaeointensity stack (GLOPIS). The Mono Lake and Laschamp geomagnetic excursions are characterised by pronounced intensity lows. Age (GLOPIS) and depth (from left: shallow, young to right: deep, old) are plotted on the abscissa vs. the RPI. Light grey lines indicate suggested correlations; a cultural layer dated by radiocarbon is indicated. From HAMBACH et al. (submitted).

Abb. 10: Die relative Paläointensität (RPI) gemessen an einem Lössprofil aus Krems (Niederösterreich) und der Global Palaeointensity stack (GLOPIS). Die geomagnetische Mono-Lake- und die Laschamp-Exkursion werden durch deutlich hervortretende Abschwächungen der Intensität charakterisiert. Alter (GLOPIS) bzw. Tiefe (von links (jung bzw. hangend) nach rechts (alt bzw. liegend)) sind auf der Abszisse gegen die RPI abgetragen. Hellgrau gestrichelte Linien verbinden die vorgeschlagene Korrelation der Kurven; eine mit Radiokohlenstoff datierte Kulturschicht ist angegeben. Aus HAMBACH et al. (submitted).

“Laschamp” geomagnetic excursions. The best documented RPI record stems from an archaeological site in Krems (Austria). The age model is based on AMS radiocarbon dates from cultural layers as well as on optical stimulated luminescence dating and RPI correlation to the GLOPIS stack. The investigation of the palaeomagnetic signal resulted in a reliable and comparable RPI record (Fig. 10). This high-resolution palaeomagnetic record clearly emphasises the yet largely undiscovered but high potential of quasi-continuously deposited loess sites, which contain archaeological materials for palaeomagnetic studies (HAMBACH et al. submitted; ZEEDEEN et al. 2008).

5 Magnetic susceptibility stratigraphy

Nowadays, magnetic properties of sediments and soils are widely used in Quaternary palaeoclimatic, as well as in geoarchaeological, research to characterise the palaeoenvironment and to indirectly date sedimentary sequences. Magnetic property variations with depth/time in sedimentary sequences are mainly climatically controlled and therefore can serve as relative dating tools if the timing of palaeoclimatic variations is known independently.

Palaeomagnetism and enviromagnetism use the same physical methods and similar technologies, but deal with fundamentally differ-

ent phenomena. Palaeomagnetism is based on the fact that rocks can provide a record of the temporal variations of the EMF: Sediments may be regarded in this case as 'tape recorders' of the past EMF which we can decipher with palaeomagnetic methods. By knowing its temporal variations, one can indirectly date these sediments while correlating their records with 'master curves' (e.g., BUTLER 1992, SCHNEPP et al. 2004).

Enviromagnetism, the magnetism of sediments and soils (rock and mineral magnetism applied to environmental questions), however, describes the occurrence, abundance and properties of iron-bearing minerals in the environment: When we study the magnetic properties of sediments and soils as proxies for environmental change, we study the physical (magnetic) properties of the 'tape' itself and not the 'music', which is recorded on it (e.g., EVANS & HELLER 2003).

Magnetic grains, exclusively iron oxides/hydroxides and sulphides, occur virtually ubiquitously in Quaternary sediments, soils, dusts and organisms, albeit often in minor or trace concentrations. After sedimentation or reworking, they undergo diagenesis and pedogenesis when, for example, more humid conditions predominate, which is reflected in physical-chemical alterations of the sediment or soil. These alterations can also result in transformation, depletion as well as enhancement of magnetic minerals. Especially ferrimagnetic minerals react in ambient laboratory magnetic fields (measurement of magnetic susceptibility, MS) several orders of magnitude more strongly than other iron-bearing minerals. Thus, already very small amounts of magnetic minerals control the magnetic properties of sediments or soils. Furthermore, they may act as sensitive magnetic recorders of palaeoclimatic and palaeoenvironmental change. Changes in climate as well as human occupations produce changes in the environment, including sedimentary and soil-forming environments. Thus, magnetic information from a wide range of marine as well as continental sedimentary archives reflects alternating warm/humid and cold/dry climates during the Quaternary.

Therefore, MS and other magnetic parameters as functions of depth in sedimentary archives can serve as proxies for palaeoclimatic variations also on the continents and allow for a close match with the isotope records from Greenland and Antarctic ice cores as well as with all kinds of high-resolution palaeoclimatic archives on millennial scales.

Loess is by far the most important terrestrial archive that provides detailed palaeoclimatic information for the whole Quaternary and in China goes back to even the Pliocene. HELLER & LIU (1984) first used magnetic susceptibility variations in Chinese loess to correlate the loess deposits to marine records. The MS variations in the loess-palaeosol couplets in the Chinese loess plateau resemble the pattern of the global ice volume record with higher values in palaeosols (interglacials) and lower values in loess (glacials). In China, magnetic susceptibility was found to reflect the intensity of pedogenesis, which in turn leads to enhancement of magnetic minerals in soils. However, in other parts of the world under different climatic conditions, even depletion of the magnetic fraction could be observed. Furthermore, the wind strength during dust transport and loess deposition also seems to control the magnetic mineralogy. With stronger winds, minerals with higher density such as iron oxides are enriched during aeolian transport. For a detailed discussion of these phenomena, we refer to EVANS & HELLER (2001, 2003).

The enhancement of magnetic minerals in loess during pedogenesis is by far the most important process that defines the magnetic properties of most loess deposits. The models that deal with the enhancement processes are essential for understanding the direct mineralogical reasons for magnetic susceptibility enhancement. The formation of ferrimagnetic minerals in the course of pedogenesis is the most important mechanism. Its rate and the equilibrium between the formation of magnetite/maghemite and other Fe-minerals are controlled by conditions in the soil environment such as temperature, moisture, pH and content of organic matter (EVANS & HELLER 2001). The most widely

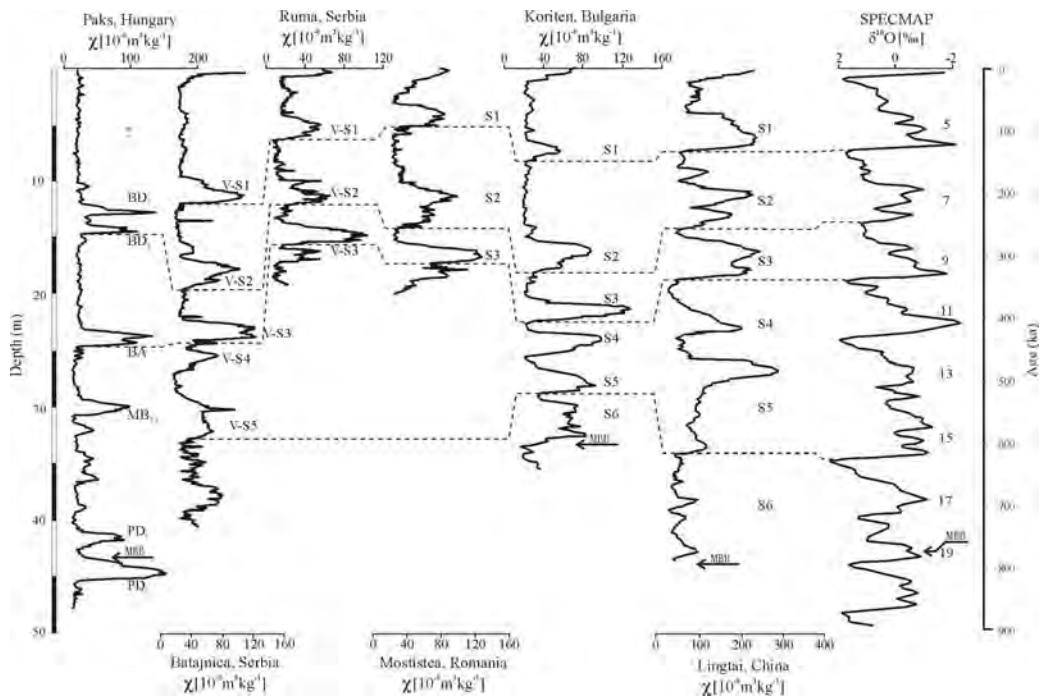


Fig. 11: Correlation of magnetic susceptibility records from Paks (SARTORI et al. 1999), Batajnica, Ruma (MARKOVIĆ et al. 2006), Mostistea (PANAIOU et al. 2001) and Koriten (JORDANOVA & PETERSEN 1999), with the astronomically calibrated MS curve of the Chinese loess site Lingtai (SUN et al. 2006) and with the SPECMAP oxygen isotope record (IMBRIE et al. 1984). Note that all SE-European sections are plotted on the depth scale of the loess profiles at Paks. From MARKOVIĆ et al. (submitted).

Abb. 11: Korrelation von magnetischen Suszeptibilitäts Kurven aus Paks (SARTORI et al. 1999), Batajnica, Ruma (MARKOVIĆ et al. 2006), Mostistea (PANAIOU et al. 2001) und Koriten (JORDANOVA & PETERSEN 1999) mit der astronomisch kalibrierten MS Kurve des chinesischen Lössprofils Lingtai (SUN et al. 2006) und mit der SPECMAP Sauerstoffisotopenkurve (IMBRIE et al. 1984). Zu beachten ist, dass alle SE-europäischen Profile mit dem Tiefenmaßstab des Lössprofils Paks gezeichnet wurden. Aus MARKOVIĆ et al. (submitted).

accepted model (CHEN et al. 2005; EVANS & HELLER 2001; MAHER 1998; THOMPSON & OLDFIELD 1986) assumes alternating reducing and oxidizing conditions in the beginning, which leads to a release of Fe^{2+} from the weathering of iron minerals. For the whole process, the relevance of iron-reducing bacteria is stressed by several authors (CHEN et al. 2005; EVANS & HELLER 2003; MAHER 1998). Finally, magnetite of extremely fine grain size is formed by dehydration and is still susceptible for dissolution. Only further oxidation to maghemite results in a more stable ferrimagnetic mineral giving the palaeosols in loess their magnetic

characteristics (BUGGLE et al. in press; MAHER 1998).

Many previous investigations of loess-palaeosol sequences around the world have used MS as a basis for differentiating widespread loess and palaeosol units, correlating them regionally and relating them to the deep-sea isotope stratigraphy (e.g., HELLER & EVANS 1996; EVANS & HELLER 2001). Recently thick loess deposits from the Carpathian region were also studied using magnetic proxies. The application of the Chinese loess stratigraphical scheme (HELLER & LIU 1984) necessitates a serious revision of the earlier chronostratigraphic interpreta-

tions of the loess-palaeosol sequences in the Danube basins. In Figure 11, MS records from loess sections from the Middle and Lower Danube Basin (Hungary and Serbia to Romania and Bulgaria) are presented. The more than 40 m thick loess-palaeosol sequences on the Danube banks near Batajnica, Serbia, are characterised by sharp environmental differences between high dust accumulation rates during the glacials and reduced dust fluxes in the periods of palaeosol formation (MARKOVIC *et al.*, submitted). The correlation of the MS record at Batajnica to other records from the region as well to the global ice volume record (IMBRIE *et al.* 1984), leads to a revised chronostratigraphic frame that yields deposition of loess during the last five interglacial-glacial cycles. This example demonstrates on the one hand the potential of MS stratigraphy as an indirect dating tool for the Middle and Upper Pleistocene across Eurasia and on the other hand its potential for analysing the long-term environmental dynamics within the context of regional, continental and global climate changes.

6 Summary and outlook

In our overview, we pointed out the potential of the largely underdeveloped application of magnetic dating techniques in the Quaternary. Though magnetic dating is already well established for marine sediments and volcanic rocks, in terrestrial sedimentary archives and for archaeological materials, its implementation is far from exhausted. Since the early 1980s, magnetostratigraphic and magnetic susceptibility dating has been extensively applied to Asian loess sequences. However, their applications to other loess provinces around the world and to other terrestrial sedimentary archives still lacks a breakthrough. While the refinement of the geomagnetic polarity time scale for the past 200 Ma has slowed down in recent times, a new potential seems to arise from the geomagnetic instability time scale for the past few Ma. High precision $^{40}\text{Ar}/^{39}\text{Ar}$ isochron dating now allows the correlation of geomagnetic

excursions recorded in volcanic rocks with the worldwide relative palaeointensity stack of marine sequences. This provides a new and additional possibility for magnetostratigraphic dating for the Quaternary. The use of geomagnetic excursions as time markers in the Brunhes and Matuyama chrons, in combination with relative palaeointensity records derived from the same archives, will open a new perspective for dating of Middle and Lower Pleistocene sequences which are hardly datable by other methods.

As the Quaternary is the first period in Earth's history in which the climate and the biosphere are influenced by non-system players (humans), the knowledge about past climates is essential for our understanding of future climate change. Magnetic dating techniques are among the few dating methods, which can be applied to a wide range of geological archives and that can help us to decipher what happened in the past and can help improve predictions of future climate changes.

We have hopefully demonstrated the success and achievements of palaeo- and rock magnetism in earth science in general and for Quaternary research in particular and we would be very pleased, if our contribution publicises magnetic dating techniques to a broader geoscientific audience.

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$^{230}\text{Th}/\text{U}$ -dating of fossil corals and speleothems

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Abstract: Both marine and terrestrial carbonates can be precisely dated by U-series disequilibrium methods in the age range <600 ka (thousands of years). Here we focus on $^{230}\text{Th}/\text{U}$ -dating of reef corals and speleothems. The requirements, potential but also the problems of $^{230}\text{Th}/\text{U}$ -dating of both archives are presented and discussed. Fossil reef corals are used as indicators for past sea level fluctuations and as high-resolution palaeoclimate archives. These applications require precise and accurate dating, which can be achieved using $^{230}\text{Th}/\text{U}$ -dating. However, many fossil corals show evidence for post-depositional open-system behaviour. This limits the accuracy of $^{230}\text{Th}/\text{U}$ -ages of fossil corals rather than the analytical precision. We present and discuss the currently available methods to identify altered corals and also review three recently developed open-system dating approaches. Speleothems are very important climate archives because they are found in most continental areas and can be used to investigate and directly compare spatially variable climate conditions. They usually show no evidence for open-system behaviour but may contain significant amounts of initial detrital ^{230}Th . We discuss the currently available correction techniques and methods to derive the most reliable ages. Furthermore, we give an overview of the state of the art techniques for U-series isotopes measurements.

[$^{230}\text{Th}/\text{U}$ -Datierung fossiler Korallen und Speläotheme]

Kurzfassung: Marine und terrestrische Karbonate können im Altersbereich von bis zu 600.000 Jahren mit Uranreihen-Ungleichgewichtsmethoden sehr präzise datiert werden. In diesem Artikel wird die $^{230}\text{Th}/\text{U}$ -Datierung von Riffkorallen und Speläothemen dargestellt. Anforderungen, Potenzial aber auch Probleme der Datierung beider Archive werden eingehend diskutiert. Fossile Riffkorallen werden sowohl als Anzeiger der Meeresspiegelschwankungen der Vergangenheit als auch als Paläoklimaarchiv mit sehr hoher Auflösung genutzt. Für beide Anwendungen werden äußerst präzise und zuverlässige Alter benötigt, die mit der $^{230}\text{Th}/\text{U}$ -Methode bestimmt werden können. Viele fossile Riffkorallen zeigen jedoch Anzeichen von nachträglicher diagenetischer Veränderung, welche die Genauigkeit der $^{230}\text{Th}/\text{U}$ -Alter mehr limitiert als die Messgenauigkeit. Die Methoden zur Identifikation diagenetisch veränderter Korallen werden dargestellt, und drei in den letzten Jahren entwickelte Methoden zur Datierung von Korallen, die sich als offene Systeme verhalten haben, werden diskutiert. Speläotheme sind bedeutende Klimaarchive, da sie in fast allen Gegenden vorkommen, was es ermöglicht, Klimaveränderungen in verschiedenen Gebieten zu untersuchen und direkt miteinander zu vergleichen. Sie zeigen in der Regel keine Anzeichen für nachträgliche Störungen des U-Th-Systems können aber in Einzelfällen signifikante Mengen von initialem ^{230}Th enthalten. Die gängigen Korrekturverfahren und Methoden, um zuverlässige Datierungen zu erhalten, werden dargestellt. Weiterhin geben wir einen Überblick über die derzeitigen Methoden zur Messung von Uranreihenisotopen.

Keywords: $^{230}\text{Th}/\text{U}$ -dating, fossil corals, speleothems, palaeoclimate reconstruction, mass spectrometry

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1 Introduction

Palaeoclimate archives such as ice cores, deep sea sediments or speleothems provide important insights about natural climate variability, which are urgently needed to understand the mechanisms driving our climate system and hence to quantify and potentially predict (anthropogenic) future climate change. A key aspect of palaeoclimate reconstruction is the accurate determination of the *timing* of past climate changes, which provides fundamental information about the phasing of change and, therefore, about potential links between climate in different regions or between climate and the forcing mechanisms driving it (HENDERSON 2006).

Carbonates such as corals or speleothems can be accurately and precisely dated using U-series disequilibrium methods. This method is based on the radioactive decay of radionuclides within the naturally occurring decay chains. There are three decay chains, each starts with an actinide nuclide (i.e., ^{238}U , ^{235}U , and ^{232}Th) having a long half live (all have $T_{1/2} > 7 \times 10^8$ a) and ultimately ends with different stable isotopes of lead (Fig. 1). In an undisturbed system all daughter nuclides reach a state of radioactive equilibrium with the parent nuclide after a few million years. Natural processes that fractionate the nuclides within a decay chain result in a disequilibrium between the activity of the parent and the daughter isotopes. The return to equilibrium then allows quantification of time and, thus, dating of the timing of chemical or physical fractionation (BOURDON et al. 2003).

U-series disequilibrium dating methods can, in principle, be applied to all materials that form accompanied by a constrained parent-daughter disequilibrium, such as marine carbonates (reef and deep sea corals, carbonate bank sediments, molluscs and foraminifera), lacustrine carbonates (tufa and inorganically precipitated sediments), terrestrial carbonates (speleothems and travertine) and peat deposits (GEYH 2008). However, U-series dating of each of these materials is associated with particular problems. For example, mollusc shells and foraminifera

were shown to take up and exchange U after deposition violating the basic closed-system assumption of U-series dating (next section). Hence, U-series dating cannot be reliably applied to these materials. Reef building corals and speleothems (i.e., secondary carbonates precipitated in caves), however, can be accurately and precisely dated using U-series methods. For these materials U-series dating methods are widely used because they provide by far the most reliable and precise ages for samples younger than 600 ka (thousands of years). Here, we focus on mass spectrometric U-series dating of corals and speleothems. A review of $^{230}\text{Th}/\text{U}$ -dating of other marine and terrestrial carbonates is given by EDWARDS et al. (2003).

Fossil reef corals were successfully dated for the first time with U-series methods by BARNES et al. (1956) using alpha-counting techniques, followed by numerous studies in the 1960's and 1970's (e.g., BROECKER et al. 1968; MESOLELLA et al. 1969). With the advent of modern thermal ionisation mass spectrometry (TIMS, section 2.3.2) the precision of U-series dating significantly improved. This led to increasing research on fossil reef corals and the application for sea level reconstruction in the late 1980's and early 1990's (e.g., BARD et al. 1990; CHAPPELL & POLACH 1991; CHEN et al. 1986; EDWARDS et al. 1986; GALLUP et al. 1994).

The potential of using U-series dated proxy records in speleothems, such as oxygen and carbon isotopes for palaeoclimate reconstruction was already indicated by the pioneering work in the 1960's and 1970's (e.g., HENDY & WILSON 1968). Within the past two decades improvements in analytical techniques allowed (i) more precise $^{230}\text{Th}/\text{U}$ -ages to be obtained from much smaller sample sizes and (ii) a variety of proxies to be measured at very high spatial resolution (i.e., in the range of μm). Because speleothems are found in most continental areas, spatially variable climate changes can be investigated and directly compared. Thus, it has recently been hypothesized that for palaeoclimate research, the next two decades might be „the age of the speleothem“ (HENDERSON 2006).

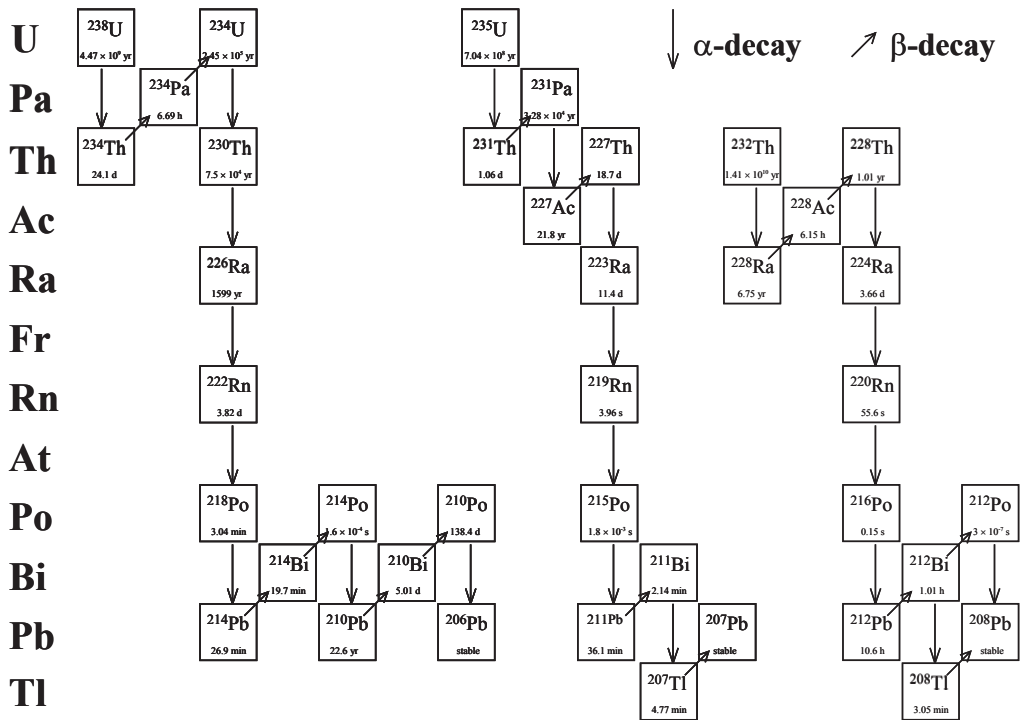


Fig. 1: Schematic drawing of the ^{238}U , ^{235}U and ^{232}Th decay chains (from SCHOLZ 2005). The half-lives of the nuclides are indicated in the boxes.

Abb. 1: Schematische Darstellung der Zerfallsreihen von ^{238}U , ^{235}U und ^{232}Th . Die Halbwertszeiten sind in den jeweiligen Boxen angegeben.

2 Basics

2.1 Geochemistry of U and Th

Typical U concentrations of the continental crust range from 0.1 to 6 $\mu\text{g/g}$. ^{238}U is the most abundant U isotope (99.2745 %), and ^{232}Th is the most abundant Th isotope (~100 %). The average Th/U weight ratio in crustal rocks is ~3.5 (WEDEPOHL 1995). The half-lives of ^{238}U ($T_{1/2} = 4.4683 \times 10^9$ a, JAFFEY et al. 1971), ^{235}U ($T_{1/2} = 0.70381 \times 10^9$ a, JAFFEY et al. 1971), and ^{232}Th ($T_{1/2} = 14.01 \times 10^9$ a, HOLDEN 1990) are much longer than those of their daughters in the decay chain (Fig. 1). Thus, the activities of parent and daughter isotopes reach a state of secular equilibrium in naturally occurring undisturbed materials within several million

years. However, natural processes can disrupt the state of equilibrium, which is the key of the U-series disequilibrium dating methods. A state of disequilibrium in the ^{238}U decay chain can either result from elemental fractionation of Th from U or from isotope fractionation between ^{234}U and ^{238}U .

The elemental fractionation is a result of the different geochemical behaviour of U and Th. U mainly exists in two oxidation states in nature (U^{4+} and U^{6+}), and at the Earth's surface it is dominant in its soluble U^{6+} form. It is soluble as uranyl ion $(\text{UO}_2)^{2+}$ and in various uranyl carbonate forms (IVANOVICH & HARMON 1992). In a reducing environment, however, it occurs mainly in the U^{4+} state where it is insoluble and, thus, far less mobile than U^{6+} . In contrast, Th,

which occurs in terrestrial material mainly in the 4+ oxidation state, is insoluble in natural waters (IVANOVICH & HARMON 1992), and, thus, under natural conditions usually transported in minerals or adsorbed onto particles. As a consequence, groundwater, fluvial water and seawater contain dissolved U but essentially no Th. Hence, in contrast to U, Th is not incorporated in secondary carbonates during their formation resulting in an initial Th/U disequilibrium.

Fractionation of ²³⁴U and ²³⁸U is produced by the α -recoil effect. An α -decay (e.g., decay of ²³⁸U, Fig. 1) results in the emission of a He nucleus with finite kinetic energy. This has a twofold effect: (i) The daughter nuclide is slightly displaced from its parent's original site and, thus, either directly ejected into an adjacent phase or more easily removed subsequently. (ii) The crystal lattice is damaged by the α -particle along the trajectory (BOURDON et al. 2003). Therefore, the daughter nuclide is subsequently more easily mobilized than its parent e.g., during weathering processes. As a consequence, ²³⁴U/²³⁸U isotope ratios of ground and river water are usually higher than the equilibrium value (CHABAUX et al. 2003; PORCELLI & SWARZENSKI 2003).

2.2 Calculation of ²³⁰Th/U-ages

The activity, *A*, of a number of atoms of a radioactive nuclide, *N*, is defined as the number of decay events per unit of time:

$$A = \frac{dN}{dt} = -\lambda N, \quad (1)$$

where λ is the decay constant defined as $\ln(2)/T_{1/2}$.

In the following, activities and activity ratios are indicated by parenthesis, e.g., (²³⁸U). The temporal development of the activities of the individual isotopes within a decay chain is described by a system of differential equations, which can be solved by standard methods (IVANOVICH & HARMON 1992).

In contrast to U, Th is not incorporated in secondary carbonates. Thus, the initial activity of ²³⁰Th, (²³⁰Th)_{init.}, is zero. If we further assume that the decay system remains closed after deposition (i.e., both U and Th isotopes are neither lost nor gained) and take into account that the half-life of ²³⁸U is much longer than that of ²³⁴U and ²³⁰Th (Fig. 1), the development of (²³⁴U/²³⁸U) and (²³⁰Th/²³⁸U) is described by equations (2) and (3) (see IVANOVICH & HARMON 1992, for derivation):

$$\left(\frac{^{234}\text{U}}{^{238}\text{U}}\right)(t) = \left(\left(\frac{^{234}\text{U}}{^{238}\text{U}}\right)_{\text{init.}} - 1\right) e^{-\lambda_{234} t} + 1 \quad (2)$$

$$\left(\frac{^{230}\text{Th}}{^{238}\text{U}}\right)(t) = (1 - e^{-\lambda_{230} t}) + \left(\left(\frac{^{234}\text{U}}{^{238}\text{U}}\right)(t) - 1\right) \frac{\lambda_{230}}{\lambda_{230} - \lambda_{234}} (1 - e^{-(\lambda_{230} - \lambda_{234}) t}), \quad (3)$$

where (²³⁴U/²³⁸U)_{init.} is the initial (²³⁴U/²³⁸U) activity ratio, and the λ_i are the decay constants for ²³⁰Th and ²³⁴U, respectively. The most recent values for the decay constants of ²³⁰Th and ²³⁴U are $\lambda_{230} = 9.1577 \times 10^{-6} \text{ a}^{-1}$ and $\lambda_{234} = 2.8263 \times 10^{-6} \text{ a}^{-1}$ (CHENG et al. 2000). Note that Eqs. (2) and (3) are only valid if two basic assumptions are fulfilled: (i) no presence of initial ²³⁰Th and (ii) the system remains closed after deposition. If one of these assumptions is violated, the resulting ²³⁰Th/U-age might be biased substantially and, thus, without significance. However, if both assumptions are fulfilled, the measurement of (²³⁴U/²³⁸U) and (²³⁰Th/²³⁸U) allows the calculation of the time since the formation of the sample. Fig. 2 shows the temporal development of (²³⁰Th/²³⁸U) and (²³⁴U/²³⁸U) for two different values of (²³⁴U/²³⁸U)_{init.}. (²³⁴U/²³⁸U) is often given in the δ -notation in per mil:

$$\delta^{234}\text{U} = \frac{\left(\frac{^{234}\text{U}}{^{238}\text{U}}\right) - \left(\frac{^{234}\text{U}}{^{238}\text{U}}\right)_{eq}}{\left(\frac{^{234}\text{U}}{^{238}\text{U}}\right)_{eq}} \cdot 1000$$

$$= \left(\left(\frac{^{234}\text{U}}{^{238}\text{U}} \right) - 1 \right) \cdot 1000, \quad (4)$$

where $(^{234}\text{U}/^{238}\text{U})_{eq}$ indicates $(^{234}\text{U}/^{238}\text{U})$ in secular equilibrium.

Eq. (2) alone cannot be used for age calculations because $(^{234}\text{U}/^{238}\text{U})_{init}$ is generally not known. Fortunately, Eq. (3) only requires the present $(^{230}\text{Th}/^{238}\text{U})$ and $(^{234}\text{U}/^{238}\text{U})$ of the sample to solve for age, which can both be measured. Since Eq. (3) cannot be analytically solved for t , the age calculation must be done either graphically or numerically. The corresponding error of the age can be calculated using numerical methods, such as Monte-Carlo simulation (LUDWIG 2003). A common way to visualize the temporal development of the activity ratios is to plot $(^{234}\text{U}/^{238}\text{U})$ vs. $(^{230}\text{Th}/^{238}\text{U})$ (Fig. 3).

2.3 Analytical techniques

$^{230}\text{Th}/\text{U}$ -dating requires precise and accurate measurement of the relevant U and Th isotope abundances to obtain the isotope activity ratios needed to calculate an age according to Eqs. (2) and (3). The isotopes ^{238}U , ^{234}U , ^{230}Th have a different atomic mass and all decay by alpha emission (Fig. 1). Two methods are commonly used for U and Th isotope measurements: alpha spectrometry and mass spectrometry. Both techniques require prior chemical separation and purification of U and Th from the CaCO_3 matrix (IVANOVICH & HARMON 1992). A detailed description of the sample preparation chemistry is given by HOFFMANN (in press). Since U and Th behave differently during separation and purification, a tracer (or „spike“) is required, such as a solution of ^{236}U and ^{229}Th with known accurately calibrated U-Th ratio. A defined

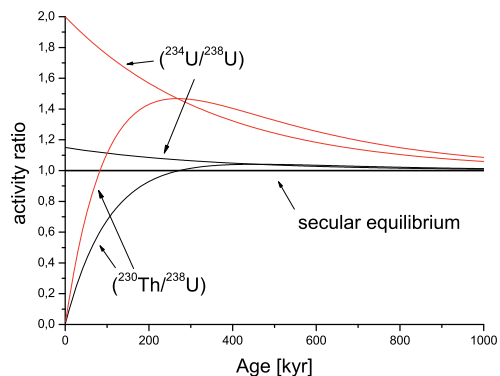


Fig. 2: Temporal development of $(^{234}\text{U}/^{238}\text{U})$ and $(^{230}\text{Th}/^{238}\text{U})$ under closed system conditions with no initial Th. Two cases of initial $(^{234}\text{U}/^{238}\text{U})$ are shown: $(^{234}\text{U}/^{238}\text{U})_{init} = 1.15$ (black line) and $(^{234}\text{U}/^{238}\text{U})_{init} = 2.0$ (red line). The development of $(^{230}\text{Th}/^{238}\text{U})$ depends on that of $(^{234}\text{U}/^{238}\text{U})$ (Eq. (3)), which in turn depends on $(^{234}\text{U}/^{238}\text{U})_{init}$ (Eq. 2). The limit of the dating range is reached when secular equilibrium is established. It is obvious that the dating range depends on $(^{234}\text{U}/^{238}\text{U})_{init}$. For marine samples, which usually have $(^{234}\text{U}/^{238}\text{U})_{init} = 1.15$ (black line, see next section), the dating range is $\sim 600,000$ a (EDWARDS et al. 2003). For other samples, such as speleothems, higher values of $(^{234}\text{U}/^{238}\text{U})_{init}$ can be found, and older samples may still be datable.

Abb. 2: Zeitliche Entwicklung der Aktivitätsverhältnisse $(^{234}\text{U}/^{238}\text{U})$ und $(^{230}\text{Th}/^{238}\text{U})$ unter Annahme eines geschlossenen Systems. Es werden zwei Fälle unterschieden: $(^{234}\text{U}/^{238}\text{U})_{init} = 1.15$ (schwarze Kurve) und $(^{234}\text{U}/^{238}\text{U})_{init} = 2.0$ (rote Kurve). Die Entwicklung von $(^{230}\text{Th}/^{238}\text{U})$ hängt von der von $(^{234}\text{U}/^{238}\text{U})$ ab, welche wiederum von $(^{234}\text{U}/^{238}\text{U})_{init}$ abhängt. Die Grenze der Datierbarkeit ist erreicht, wenn sich das säkulare Gleichgewicht eingestellt hat. Es ist offensichtlich, dass diese Grenze von $(^{234}\text{U}/^{238}\text{U})_{init}$ abhängt. Für marine Proben, die in der Regel ein initiales $(^{234}\text{U}/^{238}\text{U})$ Aktivitätsverhältnis von 1.15 haben (schwarze Kurve, s. nächster Abschnitt), ist das Limit der Datierungsmethode ca. 600 000 Jahre (EDWARDS et al. 2003). Für andere Proben, wie z.B. Speläotheme, sind höhere Werte für $(^{234}\text{U}/^{238}\text{U})_{init}$ nicht ungewöhnlich. In diesen Fällen können auch ältere Proben noch datiert werden.

quantity of the tracer needs to be added to the sample prior to the separation procedure (isotope-dilution technique). Hence, additionally to the above mentioned isotopes, the abundances of ^{236}U (and/or ^{233}U) and ^{229}Th also have to be determined. Furthermore, ^{232}Th is measured as a proxy for detrital components.

2.3.1 Alpha spectrometry

Radioactive decay of the isotopes mentioned above is accompanied by the emission of an alpha particle with isotope specific energy. Thus, one possibility of measuring the relative isotope abundances is alpha spectrometry. However, alpha spectrometry is meanwhile of

minor importance for $^{230}\text{Th}/\text{U}$ -dating of corals and speleothems due to the limits in precision and requirements in sample sizes. In brief, after separation, U and Th fractions are deposited as a thin layer on a flat surface such as metal planchet, e.g., by electrodeposition. The planchet is then placed in a sample chamber at high vacuum beneath a detector, which detects the occurrence and energy of alpha particles. A U or Th isotope measurement takes a few days, and the precision is in the range of a few percent, which limits the dating range to 300 ka. Another disadvantage of alpha spectrometry compared to mass spectrometry is the rather big sample size required for one measurement (usually more than 1 μg total U).

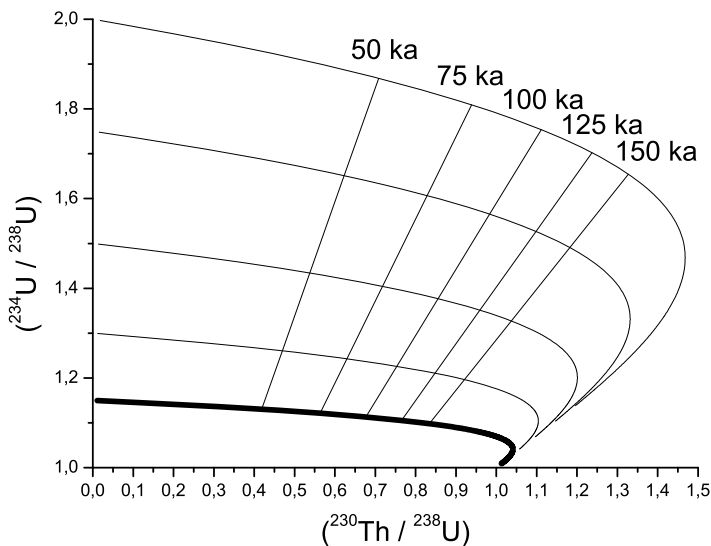


Fig. 3: $(^{234}\text{U}/^{238}\text{U})$ vs. $(^{230}\text{Th}/^{238}\text{U})$ plot for different values of $(^{234}\text{U}/^{238}\text{U})_{\text{init}}$. Each curve starts with $(^{230}\text{Th}/^{238}\text{U}) = 0$ but with another value of $(^{234}\text{U}/^{238}\text{U})_{\text{init}}$. The bold curve corresponds to $(^{234}\text{U}/^{238}\text{U})_{\text{init}} = 1.15$, the value measured on modern sea water, and represents the seawater evolution curve. All curves finally converge to secular equilibrium. The vertically tilted straight lines represent isochrons for a specific age as indicated by the corresponding labels. All combinations of activity ratios of an isochron correspond to the same age. This plot is very useful to detect post-depositional open-system behaviour (next section).

Abb. 3: $(^{234}\text{U}/^{238}\text{U})$ vs. $(^{230}\text{Th}/^{238}\text{U})$ Diagramm für verschiedene Werte von $(^{234}\text{U}/^{238}\text{U})_{\text{init}}$. Jede Kurve startet bei $(^{230}\text{Th}/^{238}\text{U}) = 0$ jedoch mit einem anderen Wert für $(^{234}\text{U}/^{238}\text{U})_{\text{init}}$. Die fettgedruckte Kurve gilt für $(^{234}\text{U}/^{238}\text{U})_{\text{init}} = 1.15$, welches der heutige Wert von Meerwasser ist, und heißt daher *seawater evolution curve*. Alle Kurven konvergieren gegen das säkulare Gleichgewicht. Die vertikalen Gerade sind Isochronen für die angegebenen Alter. Alle Kombinationen von Aktivitätsverhältnissen auf einer Isochrone entsprechen dem gleichen Alter. Dieses Diagramm ist sehr nützlich, um nachträgliche Störungen des Systems zu erkennen (s. nächster Abschnitt).

2.3.2 Mass spectrometry

$^{230}\text{Th}/\text{U}$ -dating is now largely done using mass spectrometry. Thus, this technique is presented in more detail here. Basically, a mass spectrometer directly measures the relative isotope abundances via the mass to charge ratio of ionised isotopes in a sample. The concept was first introduced almost a century ago in 1913 by Thompson. It is based on the deflection of charged particle beams in a magnetic field depending on the mass to charge ratio. A review of early developments of mass spectrometry can be found in DE LAETER (1998). Technical advances in thermal ionisation mass spectrometry (TIMS) in the 1980's led to a significant improvement in precision and detection limits (EDWARDS et al. 1987). In the last 10–15 years further improvements in plasma source mass spectrometry, especially the advent of multi collector inductively coupled plasma mass spectrometry (MC-ICPMS) led to a further shift to this technique (e.g., GOLDSTEIN & STIRLING 2003; HALLIDAY et al. 1998). Here we focus on TIMS and MC-ICPMS because those are currently the most important techniques for high precision U and Th isotope measurements. The basic concept of mass spectrometry is: (i) ionisation of isotopes, (ii) acceleration of the charged particles using a high potential difference, and (iii) the separation of charged particle beams with different mass to charge ratio in a magnetic field. The intensities of the separated ion beams then have to be detected to obtain the relative abundance. Thus, there are three main parts of a mass spectrometer: ion source, mass analyser and detector system.

A TIMS is a solid source mass spectrometer. The purified U and Th solutions are placed on a filament, e.g., made of Rhenium, which is placed in the ion source of the mass spectrometer. The sample chamber is evacuated to high vacuum, and the filament then heated by an electric current. Atoms are thermally released from the filament and a certain proportion (<1 %) also ionised. The ions are accelerated by a high potential in the ion source, and the ion beam is focused into the mass analyser section. The mass analyser of a TIMS is a magnetic

field. Typically, a U or Th isotope measurement takes a few hours. The total U needed for one analysis is in the range of a few tens to hundreds of nanograms, and precisions of a few per mil can be achieved.

A MC-ICPMS is a plasma source mass spectrometer. The ion source consists of a torch, in which an Ar plasma is generated by a radio frequency (RF) coil. The purified U and Th sample is dissolved in a weak acid, and these solutions are nebulised in a spray chamber and mixed with Ar sample gas. The mixture is then injected into the plasma, which has a temperature of about 8000 K, and almost completely ionised (>90 %). The ions are transferred from the plasma at atmospheric pressure into the mass spectrometer at high vacuum via the interface region, where more than 90 % of the ions are lost. Inside the mass spectrometer an extraction voltage accelerates the ions via electrical lenses into the mass-analyser section. A modern MC-ICPMS is usually equipped with a combination of an electrostatic analyser (ESA) and magnetic analyser (Nier-Johnson geometry). In contrast to TIMS, the ESA, which is essentially an energy filter, is needed because of the larger energy spread of the ions. The ionisation and transfer efficiency of a modern MC-ICPMS is in the range of 1 %. The measurement time of one U or Th sample is 10–20 minutes with precisions comparable to TIMS. For MC-ICPMS $^{230}\text{Th}/\text{U}$ -dating typically a total U load of about 5 – 10 ng is needed (HOFFMANN et al. 2007). Further details can be found in GOLDSTEIN & STIRLING (2003).

The detector system is largely the same for TIMS and MC-ICPMS. It is usually a combination of Faraday cup detectors and ion counter devices. Ion beam intensities larger than 1 mV can be measured on Faraday cups. Small ion beams are detected using ion counters, such as secondary electron multipliers or Daly detectors. U isotopes, for example, are usually measured with a combination of an ion counting system for the minor ^{234}U and ^{236}U isotopes and Faraday cups for the more abundant ^{235}U and ^{238}U isotopes. The isotope beams are ideally measured simultaneously (static) to avoid

biases by unstable beam intensities. If the detector system has only one ion counter, two static measurements are done, one with ^{234}U on the ion counter and ^{238}U on a Faraday cup and another with ^{236}U on the ion counter and ^{238}U on a Faraday cup. The measured raw isotope ratios need to be corrected for several instrumental biases, such as ion-counter-Faraday-cup gain, ion counter darknoise, Faraday cup baseline, peak tailing („abundance sensitivity“) and mass fractionation (e.g., HOFFMANN et al. 2007).

In summary, precise and accurate U-Th isotope ratio measurements can be done with TIMS and MC-ICPMS, with MC-ICPMS allowing the use of smaller sample sizes. An accurate correction of instrumental biases and accurately calibrated spikes are essential for reliable dating results. Currently, the limit for $^{230}\text{Th}/\text{U}$ -dating is in the range of approximately 600 ka.

3 $^{230}\text{Th}/\text{U}$ -dating of reef corals

3.1 ^{238}U , ^{234}U and ^{230}Th in seawater

River runoff from the continents is the dominant supply of U to the oceans with an estimated flux of $\sim 11 \times 10^9$ g/a ($\pm 35\%$) (IVANOVICH & HARMON 1992). Additional sources of U in the ocean are, e.g., wind-blown dust and groundwater discharge. While the former is unlikely to be significant ($< 3\%$ of the riverine U flux), the latter is very difficult to estimate because both the total flux and the average U concentration of groundwater are poorly known. U is mainly removed from the oceans into reducing marine sediments where U becomes insoluble in the U^{4+} state. The oceanic U budget is in balance within the level of uncertainty, and seawater has a near constant U concentration of 3.3 ppb (CHEN et al. 1986) indicating a residence time of ~ 400 ka.

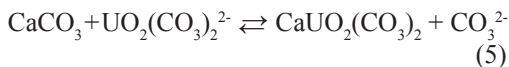
Seawater ($^{234}\text{U}/^{238}\text{U}$) is elevated compared to secular equilibrium, which is related to the α -recoil effect in minerals on the continents and in marine sediments. Most measured seawater ($^{234}\text{U}/^{238}\text{U}$) values are around 1.146 (CHEN et al. 1986; ROBINSON et al. 2004a), one study yielded a slightly higher value (1.149 ± 0.002 , DELANGHE et al. 2002). All these values are calculated using the half-lives reported by CHENG et al. (2000).

Potential changes in palaeo seawater ($^{234}\text{U}/^{238}\text{U}$) with *time* are most likely to be induced by changes in the riverine input, and several studies used models to place limits on how much marine ($^{234}\text{U}/^{238}\text{U}$) could have changed over Late Quaternary time scales (e.g., CHEN et al. 1986; HAMELIN et al. 1991; RICHTER & TUREKIAN 1993). These studies revealed that seawater $\delta^{234}\text{U}$ should not have changed more than 10–20 ‰ compared to its modern value during the last several hundred ka. This is further confirmed by U-series analysis of U-rich slope sediments suggesting that seawater $\delta^{234}\text{U}$ remained within 15 ‰ of its modern value within the last 360 ka (HENDERSON 2002). Recent studies indicate shifts in seawater ($^{234}\text{U}/^{238}\text{U}$) at times of major glacial-interglacial transitions involving large variations in sea levels (ESAT & YOKOYAMA 2006; ROBINSON et al. 2004b), but during periods of high sea level (i.e., interglacials) seawater $\delta^{234}\text{U}$ should have been in the range of $146 \pm 10\%$.

Th generally exists as a neutral hydroxide species in the oceans and is highly insoluble. Because of its high particle reactivity, it has a distinct tendency to become incorporated in colloids or adsorbed onto particle surfaces (IVANOVICH & HARMON 1992). Thus, ^{230}Th produced by decay of ^{234}U in the water column is rapidly removed by sorption to particles, which settle from the water column. The average residence time of Th in seawater is estimated as ~ 20 a (HENDERSON & ANDERSON 2003) resulting in ($^{230}\text{Th}/^{238}\text{U}$) values that are 105 times lower than secular equilibrium (MOORE 1981).

3.2 Incorporation of U-series isotopes into reef corals

The dominant form of U in seawater is the large uranyl carbonate anion $\text{UO}_2(\text{CO}_3)_3^{4-}$, and with descending pH, the species $\text{UO}_2(\text{CO}_3)_3^{2-}$ and $\text{UO}_2(\text{CO}_3)_3^0$ prevail. A comparison of several U substitution mechanisms showed that U incorporation into reef corals occurs most likely according to the following reaction (SHEN & DUNBAR 1995; SWART & HUBBARD 1982):



A variety of studies showed that the U/Ca ratio of scleractinian corals is controlled by the absolute U concentration of seawater (SHEN & DUNBAR 1995; SWART & HUBBARD 1982), water temperature (MIN et al. 1995; SHEN & DUNBAR 1995), coral growth rate, and coral species (CROSS & CROSS 1983). However, despite of these effects molar U/Ca ratios of surface corals are within about 30% of the seawater value of 1.3×10^{-6} (EDWARDS et al. 2003). Isotope fractionation between ^{234}U and ^{238}U seems not to occur during coral growth, which is demonstrated by the good agreement of the average $\delta^{234}\text{U}$ value measured on modern corals (i.e., 146.6 ± 1.4 ‰, DELANGHE et al. 2002) with the seawater value.

Molar $^{232}\text{Th}/^{238}\text{U}$ values of young surface corals are typically slightly lower than seawater values (CHEN et al. 1986). This shows that U and Th are not largely fractionated during aragonite formation and are incorporated into coral skeletons in their proportions in seawater. Consequently, the low ($^{230}\text{Th}/^{238}\text{U}$) activity ratio in seawater leads to close to zero initial ($^{230}\text{Th}/^{238}\text{U}$) activity ratio in corals, which is one of the basic premises for U-series dating.

3.3 Reliability of $^{230}\text{Th}/\text{U}$ -coral ages

The reliability of U-series ages of fossil reef corals depends on the validity of the two basic premises of $^{230}\text{Th}/\text{U}$ -dating. As discussed in the previous section, most reef building surface corals do not contain substantial initial ^{230}Th . Comparison of $^{230}\text{Th}/\text{U}$ -ages of corals with ages known a priori from counting of annual density bands showed that initial ^{230}Th levels are negligible in corals containing less than 100 pg/g of ^{232}Th (EDWARDS et al. 2003). ^{232}Th concentrations between several hundred and 1000 pg/g (COBB et al. 2003) or even higher (SCHOLZ et al. 2004; STIRLING et al. 1998) have been reported. However, values of this magnitude still do not result in significant shifts in $^{230}\text{Th}/\text{U}$ -ages for corals older than several ka, because the initial

^{230}Th will be negligible compared to that produced by decay of ^{234}U . Only for very young corals (i.e., younger than a few thousand years) high ^{232}Th content indicates the presence of significant levels of initial ^{230}Th and makes an appropriate correction necessary. This requires knowledge of the initial ($^{230}\text{Th}/^{232}\text{Th}$) activity ratio. Investigation of samples with known age showed isotopic $^{230}\text{Th}/^{232}\text{Th}$ ratios between 0 and 2×10^{-5} for marine and $\sim 4 \times 10^{-6}$ for terrestrial sources (COBB et al. 2003). With these values upper and lower limits for the correction can be estimated.

The validity of the closed-system assumption is more difficult to assess. During periods of low sea level (e.g., glacial periods) most fossil reef corals were exposed to meteoric water for a substantial period of time. This bears the risk of the aragonitic coral skeletons being recrystallised to calcite by dissolution or reprecipitation processes. Because calcite was shown to incorporate much less U than aragonite (REEDER et al. 2000), such processes are supposed to be accompanied with significant loss of U. Therefore, the calcite content of fossil corals is used as an indication for post-depositional recrystallisation and should not exceed 1%. The calcite content of a coral matrix can be precisely determined by X-ray diffraction (XRD).

Another test for open-system behaviour is the comparison of the total U content of fossil corals and their modern counterparts. Numerous studies on various coral species showed that the primary U concentration ranges from 1.5 to 4 $\mu\text{g/g}$ (e.g., AMIEL et al. 1973; CROSS & CROSS 1983; GVIRTZMAN et al. 1973; SWART & HUBBARD 1982), and within one coral specimen variations of several percent are possible (MIN et al. 1995; SHEN & DUNBAR 1995). Due to the rather large variability of U concentration in modern corals the total U content can only serve as a broad indicator for open system behaviour. Nevertheless, post-depositional uptake and/or loss of U might result in substantial age bias. For example, SCHOLZ et al. (2007) presented evidence for post-depositional U redistribution in coral reefs on Barbados, which leads to a maximum age bias of 30 ka for corals

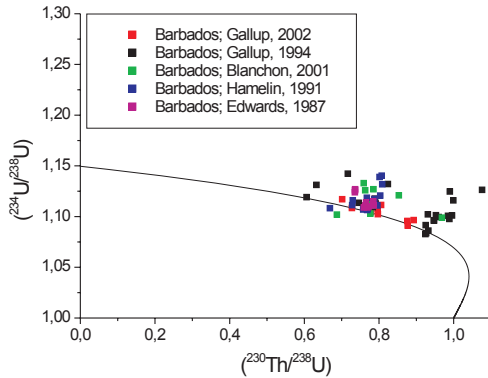


Fig. 4: A compilation of the activity ratios measured on fossil reef corals from Barbados, West Indies. The error bars are smaller than the plot-symbols. All corals are of the genus *Acropora palmata*. It is evident that most corals plot above the seawater evolution curve and, thus, have elevated initial ($^{234}\text{U}/^{238}\text{U}$). Data are taken from BLANCHON & EISENHAEUER (2001), EDWARDS et al. (1987), GALLUP et al. (2002; 1994) and HAMELIN et al. (1991).

Abb. 4 : Zusammenstellung von gemessenen Aktivitätsverhältnissen an fossilen Riffkorallen von Barbados. Die Fehlerbalken sind kleiner als die Symbole. Alle Korallen gehören zu der Gattung *Acropora palmata*. Es ist offensichtlich, dass die meisten Datenpunkte oberhalb der *seawater evolution curve* liegen und somit erhöhte initial ($^{234}\text{U}/^{238}\text{U}$)_{init} haben. Die Daten stammen aus BLANCHON & EISENHAEUER (2001), EDWARDS et al. (1987), GALLUP et al. (2002; 1994) und HAMELIN et al. (1991).

from Marine Isotope Stage (MIS) 6.5 (~175 ka before present). Elevated ^{232}Th can also be related to post-depositional contamination from detrital sources and, therefore, be an indication for open-system behaviour.

However, post-depositional open-system behaviour needs to be reliably identified in corals. Because corals incorporate ^{234}U and ^{238}U in the same ratio as they occur in modern seawater, the initial coral ($^{234}\text{U}/^{238}\text{U}$), which can be calculated using the $^{230}\text{Th}/\text{U}$ -age in Eq. (2), should agree with modern seawater ($^{234}\text{U}/^{238}\text{U}$). Equivalently, the ($^{230}\text{Th}/^{238}\text{U}$) and ($^{234}\text{U}/^{238}\text{U}$) activity ratios of a fossil coral should plot on the seawater evolution curve, which describes the temporal development of activity ratios

under closed-system conditions with the initial ($^{234}\text{U}/^{238}\text{U}$) of modern seawater (bold line in Fig. 3). A significant deviation of the initial ($^{234}\text{U}/^{238}\text{U}$) from the seawater value indicates open-system behaviour and the $^{230}\text{Th}/\text{U}$ -age has to be considered non-reliable.

However, the analytical precision of mass spectrometric analyses indicates that initial ($^{234}\text{U}/^{238}\text{U}$) varies significantly between different corals and that in fact most fossil reef corals display initial ($^{234}\text{U}/^{238}\text{U}$) in excess of modern seawater and recent corals (e.g., BARD et al. 1992; BARD et al. 1991; EDWARDS et al. 1987; HAMELIN et al. 1991; HENDERSON et al. 1993; KU et al. 1990) (Fig. 4).

All coral data shown in Fig. 4 correspond to past sea level highstands, i.e., MIS 5 and 7. As discussed in Section 3.1, seawater ($^{234}\text{U}/^{238}\text{U}$) should have been within 1 % of the modern value during such periods. Thus, the large variation in initial ($^{234}\text{U}/^{238}\text{U}$) of corals cannot reflect changes in the marine U isotopic composition and is a clear evidence for open-system behaviour. The fact that even samples, which are apparently pristine according to mineralogical criteria, show elevated initial ($^{234}\text{U}/^{238}\text{U}$) suggests that the isotopic system is more sensitive to post-depositional changes than any petrographic or general geochemical parameter (CHEN et al. 1991; ZHU et al. 1993). Therefore, the premise of closed-system behaviour is not generally warranted for fossil reef corals, and the accuracy of $^{230}\text{Th}/\text{U}$ -ages is more limited due to the isotopic anomalies than analytical precision (BARD et al. 1992; STIRLING et al. 1995).

To detect diagenetically altered corals, various reliability criteria were developed (e.g., compare STIRLING et al. 1998), and only $^{230}\text{Th}/\text{U}$ -ages that fulfil these criteria are believed to be *strictly* reliable:

- Initial ($^{234}\text{U}/^{238}\text{U}$) within a specific range of modern seawater ($^{234}\text{U}/^{238}\text{U}$)
- U content within the range of modern analogues from the same region
- ^{232}Th concentrations lower than 1 ppb
- Calcite content lower than 1 %
- Primary aragonitic textures

An additional rigorous test for open-system behaviour is combined $^{231}\text{Pa}/\text{U}$ and $^{230}\text{Th}/\text{U}$ -dating (EDWARDS et al. 2003). The application of both dating methods to the same coral sample allows identification of isotopically altered corals, which otherwise would have passed all criteria. Recent measurements on 14 corals with initial $\delta^{234}\text{U}$ values within 8% of modern seawater from Barbados showed that six had discordant $^{231}\text{Pa}/\text{U}$ and $^{230}\text{Th}/\text{U}$ -ages (CUTLER et al. 2003; GALLUP et al. 2002). This demonstrates processes that have affected Pa, Th, and/or U, but not $\delta^{234}\text{U}$ (EDWARDS et al. 2003).

3.4 Open-system dating of fossil reef corals

Since obviously most fossil corals show evidence for post-depositional diagenesis, an approach to reliably date these corals would represent a major improvement. Therefore it is a current research focus to understand the mechanisms of coral diagenesis on U-Th isotopes and develop correction techniques for open-system dating.

Several authors have proposed diagenetic scenarios, such as post-depositional U and Th gain/loss to explain the observed isotopic anomalies in fossil reef corals (e.g., BAR-MATTHEWS et al. 1993; CHEN et al. 1991; FRUIJTIER et al. 2000; HAMELIN et al. 1991; HENDERSON et al. 1993). Most diagenetically altered fossil reef corals show elevated initial $\delta^{234}\text{U}$. Several studies detected a correlation between $\delta^{234}\text{U}$ and the $^{230}\text{Th}/\text{U}$ -age (FRUIJTIER et al. 2000; STEIN et al. 1993; ZHU et al. 1993) and also between $\delta^{234}\text{U}$ and $(^{230}\text{Th}/^{238}\text{U})$ (GALLUP et al. 1994; SCHOLZ et al. 2004; STIRLING et al. 1998; THOMPSON et al. 2003; VILLEMANT & FEUILLET 2003). Other studies, however, found no general correlations (CHEN et al. 1991).

BENDER et al. (1979) were the first who presented a diagenetic open-system model for fossil reef corals. They assumed that all corals suffered continuous addition of ^{234}U and ^{230}Th . Subsequently, KU et al. (1990) proposed a model, based on gain or loss of both ^{234}U and ^{238}U through continuous exchange with U in groundwater or soil water. GALLUP et al.

(1994) detected a rough correlation between initial $\delta^{234}\text{U}$ and $^{230}\text{Th}/\text{U}$ -age in Barbados corals. On the basis of the observed relation between $(^{234}\text{U}/^{238}\text{U})$ and $(^{230}\text{Th}/^{238}\text{U})$ (Fig. 3), they modelled the diagenetic processes assuming continuous addition of ^{234}U and ^{230}Th . However, this model has two serious problems: (i) it is difficult to show that the diagenetic trends are such as described by the model when there are only a limited number of coeval corals. Furthermore, in many cases corals do not show the same diagenetic trends or no trend at all (EDWARDS et al. 2003; POTTER et al. 2004; SCHOLZ et al. 2004; SCHOLZ et al. 2007). (ii) It is not reasonable that ^{234}U and ^{230}Th should be added at similar rates because of their very different chemical behaviour (Section 2.1). In theory, the redistribution of these isotopes should rely on transport by groundwater or porewater, but the model results suggest that the different solubilities of U and Th do not play a significant role in how they are transported and deposited. GALLUP et al. (1994) proposed that the nuclides are transported on particles in the water, such as organic colloids. Many authors have proposed mechanisms to explain this problem. CHEN et al. (1991) proposed that some ^{238}U , ^{234}U , and ^{230}Th may be leached out of one part of the coral by chemical reactions and deposited onto another part. As a mechanism for ^{234}U addition they proposed that ^{234}Th as the recoil daughter nuclide of ^{238}U (Fig. 1) is likely to be hydrolysed and adsorbed onto a solid surface before decaying to ^{234}U .

More recently, three models dealing with open-system behaviour of fossil reef corals were published (SCHOLZ et al. 2004; THOMPSON et al. 2003; VILLEMANT & FEUILLET 2003). The model of VILLEMANT & FEUILLET (2003) takes into account possible initial ^{230}Th excess and continuous selective redistribution (i.e., gain and/or loss) of ^{234}U , ^{234}Th , and ^{230}Th controlled by α -recoil processes. The THOMPSON et al. (2003) model explains the positive correlation between $(^{234}\text{U}/^{238}\text{U})$ and $(^{230}\text{Th}/^{238}\text{U})$ by coupled addition of particle-reactive ^{234}Th and ^{230}Th , which is produced by decay of dissolved U and α -recoil mobilisation of U daughters. In

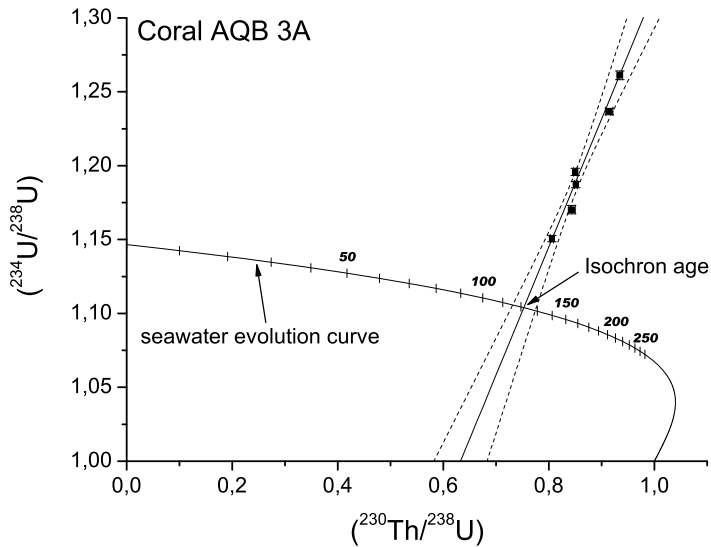


Fig. 5: (taken from SCHOLZ & MANGINI 2006): Isochron plot for coral AQB 3A from Aqaba, Jordan (SCHOLZ et al. 2004). The activity ratios measured in six different sub-samples show a high linear correlation between $(^{234}\text{U}/^{238}\text{U})$ and $(^{230}\text{Th}/^{238}\text{U})$ ($R = 0.99$). The labels on the seawater evolution curve represent the corresponding $^{230}\text{Th}/\text{U}$ -ages in ka. The isochron age is calculated from the intersect of the isochron with the seawater evolution curve. The dashed curves represent confidence bands at 95% confidence level. Isochron age errors can be calculated from the intersects of the confidence bands with the seawater evolution curve.

Abb. 5: (übernommen aus SCHOLZ & MANGINI 2006): Isochronen Diagramm für Koralle AQB 3A aus Aqaba, Jordanien (SCHOLZ et al. 2004). Die Aktivitätsverhältnisse, die an sechs verschiedenen Teilproben der Koralle gemessen wurden, zeigen eine starke lineare Korrelation zwischen $(^{234}\text{U}/^{238}\text{U})$ und $(^{230}\text{Th}/^{238}\text{U})$ ($R = 0.99$). Die Beschriftungen an der *seawater evolution curve* zeigen die entsprechenden $^{230}\text{Th}/\text{U}$ -Alter in ka. Das Isochronen Alter wird aus dem Schnittpunkt der Isochrone mit der *seawater evolution curve* berechnet. Die gestrichelten Kurven sind die entsprechenden 95%-Konfidenzbänder. Der Fehler des Isochronenalters wird aus den Schnittpunkten des Konfidenzbänder mit der seawater evolution curve bestimmt.

contrast to GALLUP et al. (1994), the authors of both models suggest that their models can be used to correct $^{230}\text{Th}/\text{U}$ -ages of samples with initial $\delta^{234}\text{U}$ deviating from the modern seawater value. They term these ages *open-system ages*. Because both models assume very similar redistribution mechanisms, the calculated $^{230}\text{Th}/\text{U}$ -ages are similar. Corals that were affected by other diagenetic processes (e.g., gain/loss of U) cannot be dated because the models take only into account the described redistribution process. Thus, such corals must be rejected, e.g., by application of standard screening criteria (Section 3.3). However, there are problems with the α -recoil based models: (i)

The trends between $(^{234}\text{U}/^{238}\text{U})$ and $(^{230}\text{Th}/^{238}\text{U})$ observed at different localities worldwide are not the same. In general, the data points from a given terrace are not all co-linear within analytical error, as required for an exact $^{230}\text{Th}/\text{U}$ -age correction with these models (EDWARDS et al. 2003). (ii) Impossibly large volumes of carbonate are necessary to maintain the concentration of dissolved U required to produce the isotopic anomalies observed in most fossil reef corals (THOMPSON et al. 2003).

SCHOLZ et al. (2004) proposed a different model to date diagenetically altered reef corals. They assumed that different sub-samples of an individual coral gained different amounts of

U with high ($^{234}\text{U}/^{238}\text{U}$), possibly followed by U loss proportional to U gain. This results in a linear correlation between ($^{230}\text{Th}/^{238}\text{U}$) and ($^{234}\text{U}/^{238}\text{U}$), similar to the models of THOMPSON et al. (2003) and VILLEMANT & FEUILLET (2003). In contrast to these models, however, the slope of the addition line (referred to as isochron) is not defined by the model but depends on the timing of U gain and loss, respectively, and hence, on the specific diagenetic history of the analysed coral. The isochron is obtained from a linear fit of the data on a ($^{234}\text{U}/^{238}\text{U}$) vs. ($^{230}\text{Th}/^{238}\text{U}$) diagram, and the coral isochron age is calculated from the intersect of the isochron with the seawater evolution curve (Fig. 5).

The isochron model (SCHOLZ et al. 2004) can also explain the activity ratios of fossil corals plotting below the seawater evolution curve if one assumes that the gained U has a lower ($^{234}\text{U}/^{238}\text{U}$) activity ratio than the coral. Because coral isochron dating does not account for elevated ^{232}Th and aragonite recrystallisation, prior screening with standard screening criteria is still required. The isochron age error is calculated by propagation of the uncertainties of the isochron (SCHOLZ & MANGINI 2006).

Meanwhile, the three open-system models have been applied in several studies to derive palaeoclimatic information (ANDERSEN et al. 2008; FELIS et al. 2004; FRANK et al. 2006; POTTER et al. 2004; THOMPSON & GOLDSTEIN 2005; THOMPSON & GOLDSTEIN 2006; WAELBROECK et al. 2008). However, both conventional $^{230}\text{Th}/\text{U}$ -dating and the three open-system models are associated with specific problems. For example, investigation of several sub-samples from the same coral specimen has shown that the real age uncertainty of both conventional $^{230}\text{Th}/\text{U}$ -ages and the open-system ages calculated by the THOMPSON et al. (2003) model is substantially larger than suggested by the quoted uncertainties (SCHOLZ & MANGINI 2007). In addition, the widely used „strict“ reliability criteria are not sufficient to identify all corals that were altered by other diagenetic processes than assumed by the models (SCHOLZ & MANGINI 2007). In general, all models should

only be applied to coral data, which have been demonstrated to be altered by the underlying processes. Application to published coral data may result in both wrong $^{230}\text{Th}/\text{U}$ -ages and substantial underestimation of the age uncertainty (SCHOLZ & MANGINI 2007).

After all, despite of almost 40 years of intensive examination of the U-series systematics of fossil reef corals, a number of aspects that produce the diagenetic alterations have not been satisfactorily explained so far. Thus, it has been extremely difficult to develop a general correction technique for diagenetically altered fossil reef corals, although several promising models have been proposed.

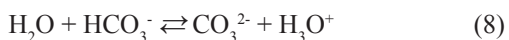
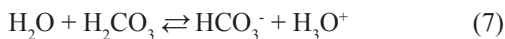
4 $^{230}\text{Th}/\text{U}$ -dating of speleothems

4.1 Growth and occurrence of speleothems

Speleothems such as stalagmites, stalactites or flowstones are secondary cave deposits and grow in karst areas in almost all parts of the world. A detailed overview of the different forms of speleothems is given e.g. by HILL & FORTI (1997). In brief, speleothem growth can be explained as follows: Meteoric water seeps through the soil above a cave and becomes enriched in CO_2 due to the high pCO_2 of the soil zone, which results from the respiration of plant roots and decay of organic material. In the soil zone pCO_2 levels can reach values of up to 100,000 ppm (McDERMOTT 2004). This results in the production of carbonic acid according to Eq. (6):



which dissociates according to Eqs. (7) and (8):



Calcium carbonate is dissolved as described by Eq. (9), when the highly corrosive percolating water comes into contact with the carbonate bedrock:



The resulting equilibrium concentration of Ca^{++} and HCO_3^- in the drip water depends on several parameters such as temperature, pCO_2 and whether dissolution occurs in a closed or an open system (KAUFMANN 2003; McDERMOTT 2004). The pCO_2 of cave air is usually higher than atmospheric pCO_2 but, depending of the degree of cave ventilation, much lower than in the soil zone. Thus, the CO_2 dissolved in the water entering the cave degases to the cave atmosphere. The drip water becomes supersaturated with respect to calcite, and CaCO_3 precipitates forming speleothems.

Stalagmite growth was first quantitatively described by DREYBRODT (1988; 1999). More recently, several models were developed describing the growth rate and shape of stalagmites depending on cave temperature, drip rate, pCO_2 of both the soil zone and the cave and also the mixing parameter, which accounts for mixing of the impinging drop with the existing solution film on top of the stalagmite (KAUFMANN 2003; MÜHLINGHAUS et al. 2007; ROMANOV et al. 2008b).

4.2 U incorporation into speleothems

Dissolution of the carbonate bedrock above the cave also results in accumulation of traces of several elements in the drip water, such as Mg, Sr, Ba and also U. Their concentration in the drip water mainly depends on their concentration in the host rock and the corresponding partitioning coefficient. The trace elements are then co-precipitated with speleothem calcite. Thus, the U-concentration in speleothems ranges from near zero to more than $100 \mu\text{g/g}$ (FORD & WILLIAMS 2007).

The U and Th isotopes, which are relevant for $^{230}\text{Th}/\text{U}$ -dating (Fig. 1, Section 2.2), are in secular equilibrium in the host rock (Fig. 2) because the karst bedrock usually has an age of several million years. However, due to the different geochemical behaviour of U and Th (Section 2.1) the activity ratios established in the percolating cave drip water are substantially different from secular equilibrium. Th is insoluble in natural water and, thus, the

($^{230}\text{Th}/^{238}\text{U}$) of drip water is zero - one of the basic premises for $^{230}\text{Th}/\text{U}$ -dating. In contrast, the ($^{234}\text{U}/^{238}\text{U}$) of drip water is usually larger than secular equilibrium due to the preferential leaching of ^{234}U resulting from α -recoil (Section 2.1), unless the host rock is already depleted in ^{234}U . The ($^{234}\text{U}/^{238}\text{U}$) of drip water depends on (i) the age of the bedrock and (ii) the time available for the dissolution of the bedrock. In theory, slowly percolating water and, thus, a long residence time should result in high drip water ($^{234}\text{U}/^{238}\text{U}$). In contrast, high rainfall events and, as a consequence, fast percolating waters and, thus, a short residence time should result in ($^{234}\text{U}/^{238}\text{U}$) close to secular equilibrium. However, unambiguous interpretation of ($^{234}\text{U}/^{238}\text{U}$) and quantification of the responsible processes remain difficult (PORCELLI & SWARZENSKI 2003). In any case, drip water ($^{234}\text{U}/^{238}\text{U}$) can vary substantially for different cave systems and is also usually not constant in time for an individual cave. Thus, there is no uniform initial ($^{234}\text{U}/^{238}\text{U}$) for speleothems like e.g. for marine carbonates, such as reef corals (Section 3). This does not affect the application of $^{230}\text{Th}/\text{U}$ -dating to speleothems because the initial ($^{234}\text{U}/^{238}\text{U}$) is not needed for the age calculation (Eq. 3).

4.3 Reliability of speleothem $^{230}\text{Th}/\text{U}$ -ages

Again, the speleothem sample has to meet the two basic premises of $^{230}\text{Th}/\text{U}$ -dating. The validity of the first assumption (i.e., no initial ^{230}Th) is usually assessed by measuring the concentration of detrital ^{232}Th . The second assumption is more difficult to verify. As mentioned in the previous section, there is no general initial ($^{234}\text{U}/^{238}\text{U}$) as for marine carbonates, which could be used as a reliability criterion. However, due to their natural occurrence in caves speleothems are not as exposed to meteoric waters as e.g. reef corals. Thus, even very old stalagmites are usually very well preserved and show no evidence for post-depositional diagenesis (WOODHEAD et al. 2006).

However, violation of one of the basic premises cannot be a priori excluded and the reliability of $^{230}\text{Th}/\text{U}$ ages needs to be assessed. Initial ^{230}Th

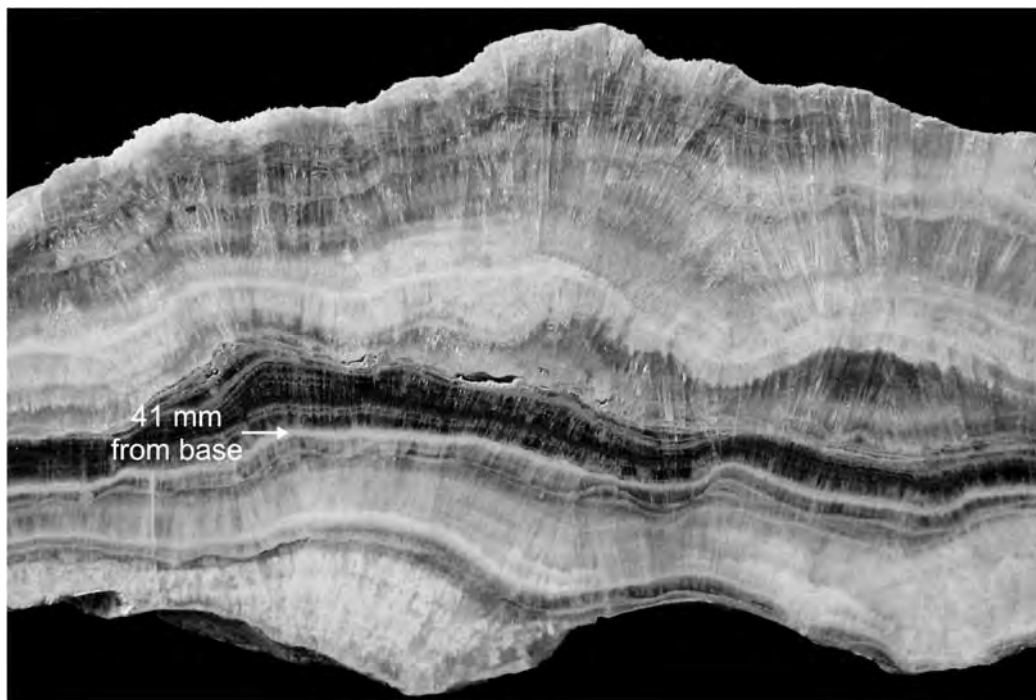


Fig. 6: Flowstone SPA 59 from Spannagel cave, Austrian Alps. There are clearly visible distinct growth layers. The chronology of SPA 59 is shown in Fig. 7. The picture was provided by C. Spötl.

Abb. 6: Flowstone SPA 59 aus der Spannagel Höhle in den österreichischen Alpen. Unterschiedliche Wachstumslagen sind sehr gut zu erkennen. Das Altersmodell für SPA 59 ist in Abb. 7 dargestellt. Das Foto wurde von C. Spötl zur Verfügung gestellt.

(i.e., not produced by in situ decay of ^{234}U and ^{238}U in the sample) is generally accompanied by a larger amount of ^{232}Th because the $^{230}\text{Th}/^{232}\text{Th}$ isotope ratio in most crustal materials is around 5×10^{-6} (WEDEPOHL 1995). For $^{230}\text{Th}/\text{U}$ -dating the ^{232}Th concentration of a sample is routinely also measured in addition to the isotopes required for the age calculation (Section 2.3). Samples containing more than an acceptable value of initial ^{230}Th can be identified based on the assumption that detrital ^{232}Th is accompanied by ^{230}Th in a defined ratio. However, it is not possible to correct the measured ($^{230}\text{Th}/^{238}\text{U}$) using a universally applicable ($^{230}\text{Th}/^{232}\text{Th}$) ratio because this ratio varies substantially in possible detrital source materials. Two correction procedures were suggested: (i) correction for detrital contamination using an a priori estimate of the isotopic composition of the detrital phase and the corresponding

uncertainty (HELLSTROM 2006; LUDWIG 2003), and (ii) direct determination of the isotopic composition of the detrital phase using isochron techniques (BECK et al. 2001; LUDWIG 2003; LUDWIG & TITTERINGTON 1994). The $^{230}\text{Th}/\text{U}$ -ages of speleothems with ($^{230}\text{Th}/^{232}\text{Th}$) within a range that indicates significant Th contamination (i.e., ($^{230}\text{Th}/^{232}\text{Th}$) $< 100 - 300$, RICHARDS & DORALE 2003) should either be corrected for the effect of contamination or rejected (HELLSTROM 2006). A criterion to test the reliability of the $^{230}\text{Th}/\text{U}$ -ages of speleothems is that the ages must be in stratigraphic order. Basically, the $^{230}\text{Th}/\text{U}$ -age of the upper layers must be younger than that of those below because stalagmites grow from the bottom up depositing growth layers (KAUFMANN 2003; MÜHLINGHAUS et al. 2007; ROMANOV et al. 2008b) (Fig. 6). Thus, the ages down the growth axis of a stalagmite must

become progressively older. Age inversions of dated layers, i.e., a $^{230}\text{Th}/\text{U}$ -age of a sub-sample that is significantly older or younger than suggested by the stratigraphy, indicate that at least one of the determined ages is wrong. This may have several reasons: (i) Post-depositional open-system behaviour, i.e., violation of the closed-system assumption, (ii) inadequate correction for detrital contamination, (iii) underestimation of the age uncertainty resulting either from the analysis itself or the applied correction for contamination, or (iv) a mistake during the analysis or the sample preparation. In any case, age inversions have to be cross-checked by analysis of additional samples from the same horizon. Otherwise, they must be accounted for by assigning the corresponding ages with an appropriately enlarged age uncertainty.

4.4 Speleothem age-distance model

To determine the age of a stalagmite at the depths of the proxy measurements, an age model is constructed, which mathematically describes the relationship between the distance from the top/bottom of the stalagmite and the age of the stalagmite. However, although an increasing number of papers presenting high precision $^{230}\text{Th}/\text{U}$ ages of speleothems were published in the last decade, a standard approach to derive an age model on the basis of the measured ages (compare Fig. 6) has not been established yet. Some authors use linear interpolation of the $^{230}\text{Th}/\text{U}$ -ages between the depth of the dated samples (WANG et al. 2005), others apply least squares polynomial fits (SPÖTL & MANGINI 2002). Other studies, in turn, apply various kinds of smoothing splines (SPÖTL et al. 2006; VÖLLWEILER et al. 2006) or even more sophisticated methods (DRYSDALE et al. 2005; DRYSDALE et al. 2004; GENTY et al. 2006) based on the general growth systematics of speleothems (KAUFMANN 2003). The age model is crucial because the timing or duration determined for a specific event, e.g., indicated by a peak in a stable isotope curve, depends on the method used to calculate the age model. This is especially problematic because $^{230}\text{Th}/\text{U}$ -dating

is usually not performed at the depth of the corresponding peak. Thus, a generalised method to construct speleothem age models is desirable, which would not only guarantee the comparability of stalagmite records from different laboratories but also make the most efficient use of the U-series ages. A general approach to estimate the uncertainty of stalagmite age models is also needed (DRYSDALE et al. 2004; GENTY et al. 2006; HEEGAARD et al. 2005; SPÖTL et al. 2006).

4.5 $^{230}\text{Th}/\text{U}$ -dating of speleothems and palaeoclimate reconstruction

The chronology of speleothem growth provides important palaeoclimatic information. For example, the timing of speleothem growth itself is related to the climatic conditions above the cave. In cold and/or dry environments even a slight cooling/drying may result in a cessation of speleothem growth (HOLZKÄMPER et al. 2004; HOLZKÄMPER et al. 2005; SPÖTL et al. 2006). Timing of speleothem formation can also be related to global climate and sea level changes (BARD et al. 2002; RICHARDS et al. 1994). Speleothems found in submerged caves indicate that the caves were not flooded at some time in the past because speleothems only form when the cave is air filled. Therefore, in tectonically stable regions the timing of the growth of speleothems collected from submerged caves unambiguously indicates that the cave was above sea level and constrains times of lower sea levels.

Furthermore, the speleothem growth rate is related to precipitation intensity, temperature and vegetation cover above the cave, which in turn is related to climate (DRYSDALE et al. 2005; DRYSDALE et al. 2004; McDERMOTT 2004; MÜHLINGHAUS et al. 2007).

An accurate and precise chronology is essential for using speleothems as palaeoclimate archives. Speleothems provide numerous proxies of palaeoclimate, which can be measured with very high spatial resolution (HENDERSON 2006). Most palaeoclimate studies using speleothems apply precisely dated high-resolution stable isotope profiles (i.e., $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$), which have contributed much information to the un-

derstanding of the climate system within the last decade (BAKER et al. 2007; CRUZ JR et al. 2005; DRYSDALE et al. 2005; FLEITMANN et al. 2003; GENTY et al. 2003; HOLZKÄMPER et al. 2004; MANGINI et al. 2005; NEFF et al. 2001; NIGGEMANN et al. 2003b; NIGGEMANN et al. 2003a; PARTIN et al. 2007; SPÖTL & MANGINI 2002; SPÖTL et al. 2002; WANG et al. 2004; WANG et al. 2005; ZANCHETTA et al. 2007). The $\delta^{13}\text{C}$ signal recorded in speleothems depends on several parameters both in the soil zone and in the cave (McDERMOTT 2004) and is, therefore, complicated to interpret. However, the understanding of the effects influencing stalagmite $\delta^{13}\text{C}$ has improved in recent years (MICKLER et al. 2004; MICKLER et al. 2006; MÜHLINGHAUS et al. 2007; ROMANOV et al. 2008a). In combination with a thorough monitoring of the studied

cave system the application of $\delta^{13}\text{C}$ for palaeoclimate reconstruction is possible (BAR-MATTHEWS et al. 2000; DRYSDALE et al. 2004; FRISIA et al. 2006; GENTY et al. 2006; GENTY et al. 2003; MÜHLINGHAUS et al. 2007). High resolution trace element profiles may reveal variation in precipitation intensity (HUANG & FAIRCHILD 2001; JOHNSON et al. 2006; ROBERTS et al. 1998; TREBLE et al. 2003; VERHEYDEN et al. 2000).

4.6 $^{230}\text{Th}/\text{U}$ -dating of speleothem SPA 59

We present a case study of $^{230}\text{Th}/\text{U}$ -dating of a flow stone (SPA 59) collected in the Spannagel cave, Austrian Alps. The present mean annual temperature in the cave is around 2°C and, thus, just above the freezing point. Since speleothems do not form below this point, periods of speleo-

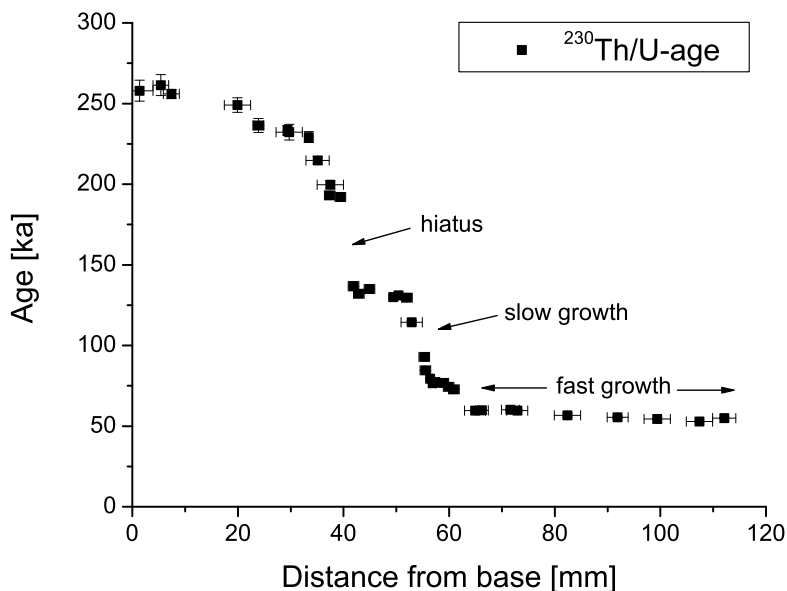


Fig. 7: TIMS $^{230}\text{Th}/\text{U}$ -dating results for flowstone SPA 59 from Spannagel cave (modified after HOLZKÄMPER et al. 2005). The speleothem grew in several phases between 50 and 270 ka, which correspond to interglacials as defined in the SPECMAP chronology (IMBRIE et al. 1984). Periods of fast and slow growth, respectively, as well as a longer hiatus between ~ 140 and ~ 190 ka are indicated.

Abb. 7: TIMS $^{230}\text{Th}/\text{U}$ -Datierungsergebnisse für Flowstone SPA 59 aus der Spannagel Höhle (geändert nach HOLZKÄMPER et al. 2005). Dieser Speläothem wuchs in mehreren Phasen zwischen 50 und 270 ka, die entsprechenden Interglazialen in der SPECMAP Chronologie zugeordnet werden können (IMBRIE et al. 1984). Phasen schnellen bzw. langsamen Wachstums sowie ein längerer Hiatus zwischen 140 und 190 ka sind eingezeichnet.

them formation correspond to past warm phases (HOLZKÄMPER et al. 2005; SPÖTL et al. 2007). Thus, the timing of the beginning and end of speleothem growth, respectively, is of particular interest. A chronology based on TIMS $^{230}\text{Th}/\text{U}$ -dating of SPA 59 was previously published by HOLZKÄMPER et al. (2005). More recently a high spatial resolution MC-ICPMS $^{230}\text{Th}/\text{U}$ -dating study of the hiatus between MIS 7 and 5 was performed by HOFFMANN et al. (submitted).

The spatial resolution of $^{230}\text{Th}/\text{U}$ dating that can be achieved depends on the sampling as well as the mass spectrometry technique that is used. There is a variety of different techniques to obtain sub-samples from speleothems. Usually a stalagmite is cut in two halves along the growth axis and the surface is then polished to identify growth layers. Samples for $^{230}\text{Th}/\text{U}$ -dating can be taken from a growth layer by a handheld drill. A disadvantage of this sampling technique is that the obtained powders are prone to contamination and cannot be cleaned prior to chemical treatment. Another possibility is to cut slices from the speleothem and then cut subsamples, e.g., using a band saw. The obtained solid pieces of carbonate can be chemically cleaned prior to further analytical work. This technique, however, is more destructive than using a handheld drill.

Fig. 6 shows the section of flowstone SPA 59 from Spannagel cave, Austrian Alps, analysed by HOLZKÄMPER et al. (2005). The corresponding TIMS $^{230}\text{Th}/\text{U}$ -dating results, based on 37 analyses along the 11 cm of growth are presented in Fig. 7. TIMS results were obtained on sample sizes between 50 and 200 mg, which were sampled using a band saw. The flowstone formed between 260 and 55 ka, mostly during the warm phases of Marine Isotope Stages (MIS) 7, 5 and 3. There are substantial differences in growth rates, with fast growth around 250, 135 and 55 ka. In contrast growth was very slow between 125 and 75 ka. Furthermore, there is a distinct 55 ka long hiatus between 190 and 135 ka.

SPA 59 has an exceptionally high U concentration (between 10 and 168 $\mu\text{g}/\text{g}$, HOLZKÄMPER et al. 2005) allowing the use of samples smaller

than 1 mg for MC-ICPMS analysis. Hence, $^{230}\text{Th}/\text{U}$ -dating can be performed at very high spatial resolution. HOFFMANN et al. (submitted) cut a 1 mm thick sample slice off the flowstone section previously analysed by HOLZKÄMPER et al. (2005) for high spatial resolution MC-ICPMS $^{230}\text{Th}/\text{U}$ dating. Six carbonate pieces of 0.5 – 1 mg were then cut from SPA 59 between 39 and 44 mm from the base at a spatial resolution of 0.8 mm using a diamond coated wire saw (Fig. 8). The sample pieces were cleaned by ultrasonication in ultra-pure water to remove potential surface contamination or residuals from the cutting process. The dried small pieces were stripped off the section with a razorblade and weighed before processing through U and Th separation and purification chemistry and MC-ICPMS analysis (HOFFMANN 2008). Fig. 9 shows the high resolution dating results in comparison with the previous TIMS chronology. The dating results agree well both below and above a white layer at the position of the hiatus indicated in Fig. 7, though the data were obtained in two different laboratories using different spikes and mass spectrometric techniques. However, the higher spatial resolution obtained using MC-ICPMS shows a dating problem associated with the white layer according to the stratigraphy criterion. Samples taken from the white layer are not in stratigraphic order. The $^{230}\text{Th}/\text{U}$ -age at 40 mm from base is apparently too old and another sample, taken at 41 mm, is out of range for $^{230}\text{Th}/\text{U}$ dating (Fig. 9). This indicates U loss, which is supported by lower U concentration in the white area (39 and 13 $\mu\text{g}/\text{g}$, respectively) compared to the clear and dense crystal below and above the white layer (64 and 134 $\mu\text{g}/\text{g}$, respectively). Thus, this section cannot be reliably dated by the $^{230}\text{Th}/\text{U}$ -method. The problem was not recognised in the study of HOLZKÄMPER et al. (2005) because they used larger sample sizes and TIMS.

Current research is concerned with MC-ICPMS $^{230}\text{Th}/\text{U}$ dating of microsamples, e.g., using a micromill or the laser ablation technique to further increase spatial resolution. Microsamples of SPA 59 with masses of about 0.09 - 0.1 mg can be prepared using a micromill. Micromill sam-

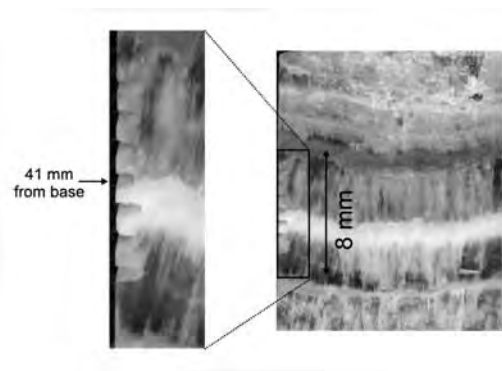


Fig. 8: A 1 mm thick slice of flowstone SPA 59. The white layer in the middle is at 41 mm from base at the position of the hiatus between MIS 7 and MIS 5. Six samples of a weight of about 1 mg were cut using a wire saw. The wire saw cuts are shown in the enlarged section. The small pieces between the cuts were stripped off and analysed for U-Th using MC-ICPMS.

Abb. 8: 1 mm dicke Scheibe von Flowstone SPA 59. Die weiße Lage in der Mitte befindet sich in 41 mm Abstand von der Basis in der Position der Wachstumsunterbrechung zwischen MIS 7 und MIS 5. Mit einer Drahtsäge wurden sechs Proben von ca. 1 mg herausgesägt. Die Schnitte der Säge sind in der vergrößerten Darstellung zu sehen. Die kleinen Karbonatstücke zwischen den Schnitten wurden entnommen und mittels MC-ICPMS auf ihren U- und Th-Gehalt untersucht.

pling procedures for $^{230}\text{Th}/\text{U}$ -dating are similar to those outlined in CHARLIER et al. (2006) for Sr isotope measurements (HOFFMANN et al. submitted). The laser ablation (LA) technique allows fast and direct $^{230}\text{Th}/\text{U}$ dating of speleothems without prior chemical separation at a spatial resolution of a few hundred μm . The precision of LA-MC-ICPMS can be similar to alpha spectrometry with samples sizes orders of magnitudes smaller (HOFFMANN et al. submitted).

5 Conclusions

Both fossil reef corals and speleothems are important archives of the climate of the past and can be reliably dated using U-series disequilibrium methods. The dating range of the $^{230}\text{Th}/\text{U}$ -method is between 500 and 600 ka depending

on both analytical precision and initial ($^{234}\text{U}/^{238}\text{U}$). Using state of the art mass spectrometric techniques such as TIMS and MC-ICPMS, accurate and very precise ages can be obtained. However, several criteria must be fulfilled to obtain reliable ages.

Most fossil corals show evidence for post-depositional diagenetic alteration, which is an indication for open-system behaviour. There have been several reliability criteria established to identify unaltered corals, but these are not able to detect all altered corals (SCHOLZ & MANGINI 2007). Several open-system dating models have been developed to calculate reliable ages from altered corals (SCHOLZ et al. 2004; THOMPSON et al. 2003; VILLEMANT & FEUILLET 2003), but each model is associated with specific problems and should only be applied if the underlying assumptions are valid (SCHOLZ & MANGINI 2007). The understanding of the mechanisms of coral diagenesis and the development of appropriate correction approaches is a major goal of current research.

Most speleothems show no evidence for post-depositional open-system behaviour but some may contain significant amounts of initial ^{230}Th , indicated by elevated ^{232}Th content. There are two correction approaches available to account for detrital contamination: (i) Direct determination of the isotopic composition of the detrital phase using isochron techniques or (ii) correction using an a priori estimate of the $^{230}\text{Th}/^{232}\text{Th}$ composition of the detrital phase with the corresponding uncertainty. The application of both approaches results in increased age uncertainty. Although an increasing number of speleothem studies with high precision $^{230}\text{Th}/\text{U}$ dating have been published in the last decade, a standard approach for the derivation of an age model on the basis of the determined ages has not been established yet. This is one of the goals of current research concerning $^{230}\text{Th}/\text{U}$ -dating of speleothems. Very high spatial resolution dating using laser ablation and micromill techniques with precisions comparable to alpha spectrometry are currently being developed (HOFFMANN 2008; HOFFMANN et al. submitted).

If the above mentioned criteria are met, it is

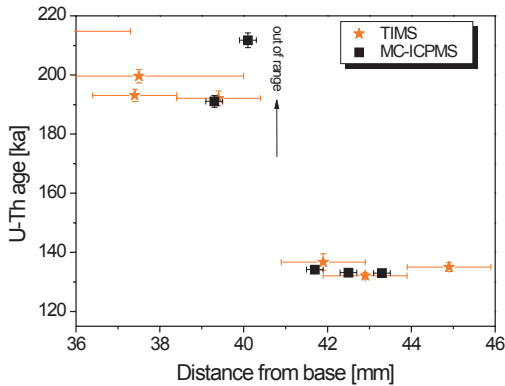


Fig. 9: TIMS and MC-ICPMS $^{230}\text{Th}/\text{U}$ -dating results for SPA 59. Below and above the white layer the results are in good agreement. However, sub-samples taken from the white layer are not in stratigraphic order indicating U loss.

Abb. 9: Ergebnisse der TIMS bzw. MC-ICPMS $^{230}\text{Th}/\text{U}$ -Datierung von SPA 59. Oberhalb und unterhalb der weißen Lage stimmen die Ergebnisse beider Methoden gut überein. Proben, die aus der weißen Lage entnommen wurden, stimmen nicht mit der Stratigraphie überein, was ein Anzeichen für nachträglichen U Verlust ist.

possible to derive very accurate and precise $^{230}\text{Th}/\text{U}$ -ages of both reef corals and speleothems. Their accuracy depends on several factors, such as the accuracy of the spike calibration and the correction of instrumental biases.

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$^{230}\text{Th}/\text{U}$ dating of interglacial and interstadial fen peat and lignite: Potential and limits

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Abstract: The state-of-the-art and potential of the $^{230}\text{Th}/\text{U}$ disequilibrium method are discussed for the dating of fen peat and lignite. Recommendations are given for the collection of suitable samples. The numerous interfering factors in $^{230}\text{Th}/\text{U}$ dating of fen peat show that a rigorous examination of the reliability of the measured data sets is required if reliable $^{230}\text{Th}/\text{U}$ ages are to be obtained. The accuracy of such $^{230}\text{Th}/\text{U}$ ages allows a reliable correlation of interglacial and interstadial deposits to the warm periods documented in the SPECMAP timescale but does not yet allow detailed temporal resolution.

[$^{230}\text{Th}/\text{U}$ -Altersbestimmung interglazialer und interstadialer Niedermoortorfe und Ligniten: Potential und Grenzen]

Kurzfassung: Der Stand und die Zukunft der Altersbestimmung von Niedermoortorf und Lignit mit der $^{230}\text{Th}/\text{U}$ -Ungleichgewichtsmethode werden diskutiert. Empfehlungen zur Auswahl der Probeentnahmestellen und zur Entnahme von geeigneten Proben werden gegeben. Aufgrund der zahlreichen negativen Einflussmöglichkeiten ist eine gründliche Eignungsprüfung der gemessenen Aktivitätsverhältnisse von Uran und Thorium solcher Proben notwendig, um zuverlässige Alter bestimmen zu können. Die Datierungsgenauigkeit ist ausreichend, um interglaziale und interstadiale Ablagerungen sicher den Warmperioden der SPECMAP-Zeitskala zuzuordnen, noch nicht aber, um sie zeitlich auflösen zu können.

Keywords: $^{230}\text{Th}/\text{U}$ dating, fen peat, lignite, minimum age

1 Basic principles of $^{230}\text{Th}/\text{U}$ dating

Peat deposits are important archives of Pleistocene climate change but correlation of individual records with long-term archives such as marine oxygen isotope records (SPECMAP; MARTINSON et al. 1987) remains tentative. Correlations based on palynological and stratigraphic studies are usually based on certain pre-assumptions and can hence not provide any independent age constraints. Sound numerical dating methods seem to be suitable tools to achieve a reliable correlation between the marine and terrestrial climatic records. One of them is the $^{230}\text{Th}/\text{U}$ dating

method. It is suitable for all materials that accumulated uranium during their formation and have not been affected by post-sedimentary processes that would mobilise uranium and thorium. When this is the case, the sampled material can be expected to have behaved as a closed system with respect to uranium and/or thorium during ageing. Moreover, it should not be older than 350-500 ka (IVANOVICH & HARMON 1992; BOURDON et al. 2000).

The $^{230}\text{Th}/\text{U}$ dating method is based on the radioactive decay series of ^{238}U . The first long-lived daughter isotope ^{234}U ($\tau = 2.4525 \times 10^2$ ka, CHENG et al. 2000) decays into the isotope ^{230}Th ($\tau = 75.69$ ka, CHENG et al. 2000) with the

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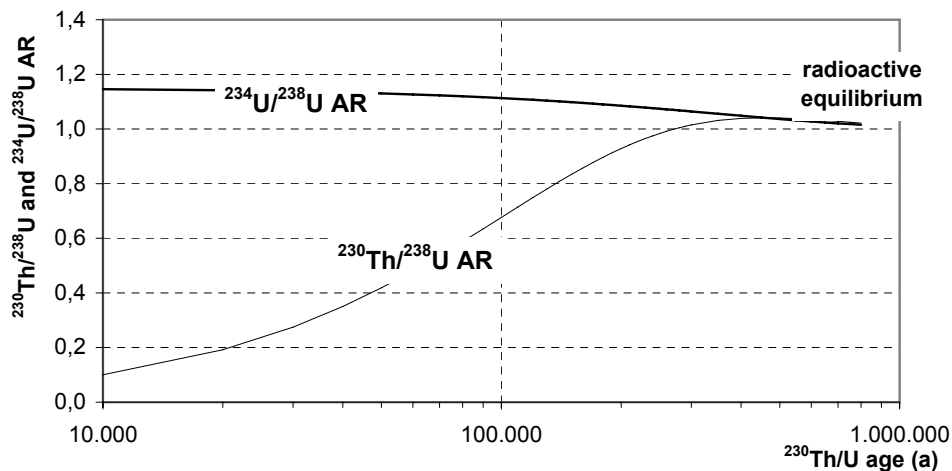


Fig. 1: Change in the $^{230}\text{Th}/^{238}\text{U}$ and $^{234}\text{U}/^{238}\text{U}$ activity ratios with increasing age for an initial $^{234}\text{U}/^{238}\text{U}$ AR of 1.15 (corresponding to the mean of marine sediments).

Abb. 1: Änderung der $^{230}\text{Th}/^{238}\text{U}$ - und $^{234}\text{U}/^{238}\text{U}$ -Aktivitätsverhältnisse mit der Alterung für ein initiales $^{234}\text{U}/^{238}\text{U}$ -Aktivitätsverhältnis von 1,15 (entsprechend dem Mittelwert mariner Sedimente).

emission of an alpha particle. The half-life of ^{238}U ($\tau = 4.468 \times 10^9$ a) is considerably longer than that of ^{234}U . As a consequence, radioactive equilibrium is established after about one million years in most geologically old rocks and sediments. At radioactive equilibrium, all members of the decay series have the same specific activity (which is the decay rate per mass unit), i.e., the activity ratios of any two of the series members are equal to one.

Radioactive equilibrium can be disturbed by geochemical processes if the members of the radioactive series have certain geochemical properties. An example is the uranium and thorium isotopes of the uranium-238 decay series. U(VI) ions are soluble in oxygenated water while thorium is practically insoluble. Hence, during weathering, uranium tends to be dissolved in water and thorium bound by clay minerals.

Uranium dissolved in groundwater can be incorporated into new systems. Examples are speleothems, foraminifera and fen peat. The most simple $^{230}\text{Th}/\text{U}$ dating model assumes that the new system initially contains only uranium. Radioactive disequilibrium exists between ^{238}U and ^{230}Th during ageing. The numerical $^{230}\text{Th}/\text{U}$ clock starts at zero and a $^{230}\text{Th}/^{238}\text{U}$ activity

ratio (AR) = 0. The radioactive disequilibrium between ^{230}Th and ^{238}U continues and evolves during ageing. A new radioactive equilibrium is approached after 350-500 ka. This process can be used to date sample material and is described by Equation 1, which has to be solved iteratively. Activity ratios are given in square brackets and $\lambda = \ln 2/\tau$:

$$(1) \quad \left[\frac{^{230}\text{Th}}{^{238}\text{U}} \right] = (1 - e^{-\lambda_{230} \cdot t}) + \left(\frac{^{234}\text{U}}{^{238}\text{U}} - 1 \right) \cdot \frac{\lambda_{230}}{\lambda_{230} - \lambda_{234}} \cdot (1 - e^{-(\lambda_{230} - \lambda_{234}) \cdot t})$$

The change in the $^{230}\text{Th}/^{238}\text{U}$ AR with age t is shown in Figure 1 assuming an initial $^{234}\text{U}/^{238}\text{U}$ AR of 1.15, which is characteristic for most marine carbonates. The $^{234}\text{U}/^{238}\text{U}$ AR of samples from terrestrial environments covers a wide range between 1 and 20. The most rapid change in the $^{230}\text{Th}/^{238}\text{U}$ AR takes place between 50,000 and about 200,000 years. The maximum age that can be measured by any dating method is the minimum age of the sample. For the radiometric and mass spectrometric $^{230}\text{Th}/\text{U}$ dating methods, this maximum age is about 350 ka and about 500 ka, respectively (Chapter 7).

KAUFMAN & BROECKER (1965) introduced an isotope-ratio evolution plot in which the $^{234}\text{U}/^{238}\text{U}$ AR is plotted versus the $^{230}\text{Th}/^{238}\text{U}$ AR. It shows the variation of these two ARs with increasing age. Each initial $^{234}\text{U}/^{238}\text{U}$ AR yields a separate evolution line. All meet the point (1,1) at radioactive equilibrium, i.e., at infinite age. The ARs of coeval samples fit straight lines of their corresponding ages, which are termed "isochrons" (Fig. 2).

2 Fen peat and lignite for $^{230}\text{Th}/\text{U}$ dating

The history of $^{230}\text{Th}/\text{U}$ dating of fen peat began with a study by TITAYEVA (1966). Her efforts failed as the analyzed Holocene peat was too young for $^{230}\text{Th}/\text{U}$ dating. But TITAYEVA recognised that "excess" (detrital) thorium and uranium have to be taken into account for $^{230}\text{Th}/\text{U}$ dating of fen peat. The first $^{230}\text{Th}/\text{U}$ ages of this material were published by VOGEL & KRONFELD (1980). Because the ages were not corrected for detrital thorium and uranium, as suggested by TITAYEVA (1966), their results were not acceptable by Quaternary geologists. The first chronostratigraphically satisfactory $^{230}\text{Th}/\text{U}$ ages of fen peat were determined by VAN DER WIJK et al. (1986) and HEINJES & VAN DER PLICHT (1992). Fen peat and lignite have a potential for $^{230}\text{Th}/\text{U}$ dating because the fulvic and humic acids they

contain have an extremely large capacity for forming complexes with uranium dissolved in groundwater. Moreover, in the reducing milieu of peat, soluble U(VI) is reduced to U(IV) and bound in very stable, immobile uranyl organic complexes. As a result fen peat may have uranium concentrations of up to 100 ppm.

The following scenario of fen peat formation is the basis for $^{230}\text{Th}/\text{U}$ dating of fen peat and lignite. Figure 3 shows a schematic section of a growing fen moss in a shallow lake. During its growth, uranium dissolved in groundwater enters the moss and is tightly bound to fulvic and humic acids. Before the peat has been compacted, groundwater can enter even deeper parts of the moss. In the terminal stage, organic material fills the entire volume of the lake. In the succeeding glacial period, the organic deposits are covered by mineral soil and compacted.

Figure 4 shows a section of interglacial fen peat sandwiched between detrital material above and below. The uranium dissolved in the groundwater seeping through the bottom and top mineral cover layers during the period of ageing is continuously absorbed in the bottom and top parts of the peat. It has been empirically found that the corresponding rim layers are seldom thicker than 10 cm. The organic material in the rim layers behaves as an open system with respect to uranium and is not datable. As a

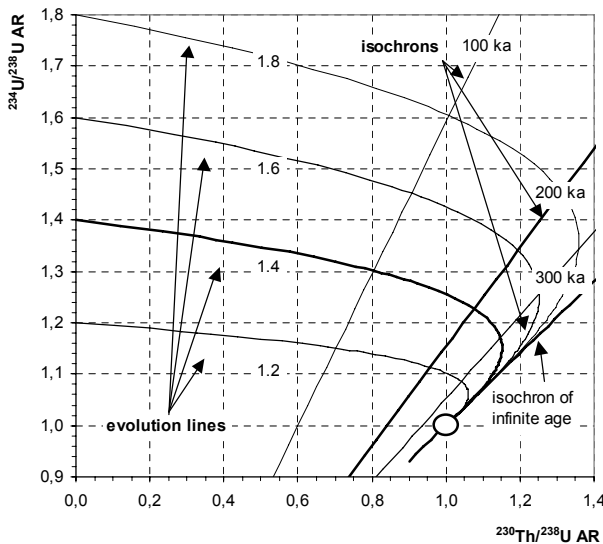


Fig. 2: Variation of the $^{234}\text{U}/^{238}\text{U}$ AR and the $^{230}\text{Th}/^{238}\text{U}$ AR with increasing age. Every initial $^{234}\text{U}/^{238}\text{U}$ AR yields an evolution line. All of them meet at the point (circle at 1,1) at infinite age, i.e. at radioactive equilibrium. The ARs of coeval samples plot on straight lines called isochrons (KAUFMAN & BROECKER 1965).

Abb. 2: Entwicklung der $^{234}\text{U}/^{238}\text{U}$ - und $^{230}\text{Th}/^{238}\text{U}$ -Aktivitätsverhältnisse mit wachsendem Alter. Jedes $^{234}\text{U}/^{238}\text{U}$ AR liefert eine eigene Entwicklungslinie, die wie alle anderen bei unendlichem Alter im Punkt 1,1 (Kreis) enden. Die Aktivitätsverhältnisse gleich alter Proben liegen auf Geraden, den Isochronen (KAUFMAN & BROECKER 1965).

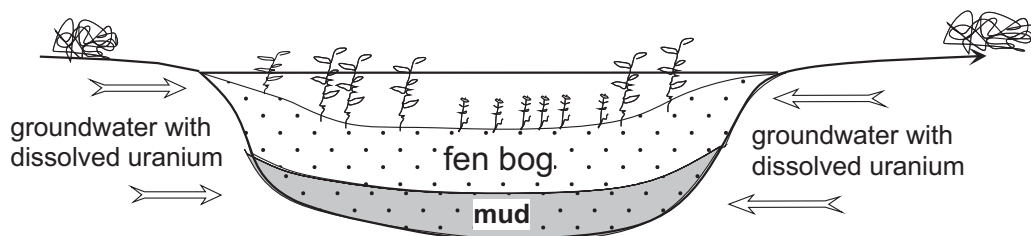


Fig. 3: Accumulation of uranium bound to fulvic and humic acids in fen peat during the peat formation in a shallow lake.

Abb. 3: Akkumulation von im Grundwasser gelöstem Uran an Fulvo- und Huminsäuren des Niedermoor torfs während der Entwicklung eines Niedermoores im flachen See.

consequence, groundwater entering the central part of fen peat more than 20-cm-thick should be free of uranium and the central part remains a closed system with respect to uranium. It is suitable for $^{230}\text{Th}/\text{U}$ dating.

The uranium and thorium data determined in a chronological study of the Netiesos interglacial site in Lithuania by GAIGALAS et al. (2005) is used in the following as a case study to illustrate the $^{230}\text{Th}/\text{U}$ dating of fen peat and lignite. The authors determined an ESR age on freshwater mollusc shells of 101.5 ± 11.5 ka. Six samples were collected between 30 and 55 cm depth. All were analyzed using the L/L (leachate/leachate) method (SCHWARCZ & LATHAM 1989; KAUFMAN 1993); the two outer samples (from 30 and 55 cm depth) were not analyzed with

the total sample dissolution (TSD) method (BISCHOFF & FITZPATRICK 1991; LUO & KU 1991). The U and Th isotope ratios were measured radiometrically at St. Petersburg University and summarized with 2σ standard deviations in a research report (Table 1). The standard deviations of these results deviate slightly from those published by GAIGALAS et al. (2005). The reason is unknown.

3 Factors that interfere with $^{230}\text{Th}/\text{U}$ dating of fen peat and lignite

There are two main factors which render $^{230}\text{Th}/\text{U}$ age determination difficult or even impossible:

1. Open-system conditions with respect to uranium and thorium, as well as

Tab. 1: Isotopenergebnisse der Niedermoorproben von Netiesos in Litauen (GAIGALAS et al. 2005).

No.	name	depth (cm)	ash (%)	$^{238}\text{U} \pm 2\sigma$ (dpm/g)	$^{230}\text{Th}/^{238}\text{U} \pm 2\sigma$	$^{234}\text{U}/^{238}\text{U} \pm 2\sigma$	$^{230}\text{Th}/^{232}\text{Th} \pm 2\sigma$
5133	L-1	30-35	50.4	0.537 ± 0.011	0.655 ± 0.032	1.194 ± 0.068	1.824 ± 0.081
5134	L-2	35-40	51.9	0.572 ± 0.018	0.617 ± 0.022	1.079 ± 0.047	1.555 ± 0.043
5135	L-3	40-45	49.7	0.494 ± 0.017	0.666 ± 0.029	1.119 ± 0.053	1.437 ± 0.059
5136	L-4	45-50	49.3	0.728 ± 0.022	0.657 ± 0.030	1.088 ± 0.046	1.790 ± 0.100
5137	L-5	50-55	47.3	0.640 ± 0.018	0.672 ± 0.031	1.136 ± 0.044	1.748 ± 0.107
5138	L-6	55-60	45.8	0.477 ± 0.008	0.755 ± 0.021	1.105 ± 0.026	1.545 ± 0.053
5129	T-2	35-40	51.9	1.582 ± 0.049	0.825 ± 0.032	1.001 ± 0.044	1.584 ± 0.075
5130	T-3	40-45	49.7	1.446 ± 0.034	0.900 ± 0.025	1.014 ± 0.033	1.240 ± 0.026
5131	T-4	45-50	49.3	2.009 ± 0.052	0.833 ± 0.025	1.030 ± 0.038	1.448 ± 0.036
5132	T-5	50-55	47.3	1.786 ± 0.039	0.843 ± 0.026	1.029 ± 0.032	1.352 ± 0.043

2. Detrital contamination with allochthonous ^{230}Th .

Open system conditions: Fen peat and lignite theoretically offers a chemically ideal milieu for closed-system conditions with respect to uranium. In contrast to carbonates (e.g., corals, foraminifera, speleothem), the reducing milieu of the peat means that any uranium present is reduced to insoluble U(IV), which form stable complexes with inorganic and organic ligands, such as humic and fulvic acids. Moreover, movement of humic acids over large distances and, therefore, post-depositional displacement of uranium can be largely excluded in the confined conditions of interglacial peat deposits. Likewise, α -recoil only moves the uranium daughter isotopes over distances that are smaller than the common sample volume of several cm^3 (HENDERSON & SLOWEY 2000). According to the above-described scenario for the formation of interglacial fen peat deposits, the central part may be considered as a closed system with respect to uranium and thorium. However, there are several possible situations in which this may not be true. Some fen peat deposits contain thin sand layers which may be conduits for oxygenated groundwater to the middle of the deposit. In the peat adjacent to these layers, open-system conditions with respect to uranium may have been present. Occasionally samples from the central part of a deposit without visible stratigraphic peculiarities yield outlier $^{230}\text{Th}/\text{U}$ ages. A possible explanation is that permafrost might have expanded the frozen fen peat and on warming voids were created into which groundwater containing uranium could have penetrated before the peat was recompacted. Detailed descriptions of the sampling site may help correlate outlier $^{230}\text{Th}/\text{U}$ ages to stratigraphic peculiarities. There are other scenarios which may occur in permafrost regions (SCHIRRMESTER et al. 2002). For example, superficial peat layers may become dry down to a depth of 1-2 m during the summer months, allowing oxygenated melt water to enter the deep parts of the deposit.

Many dated interglacial peat deposits in western Asia have been exposed along river banks. If the river rises above the level of the peat layers,

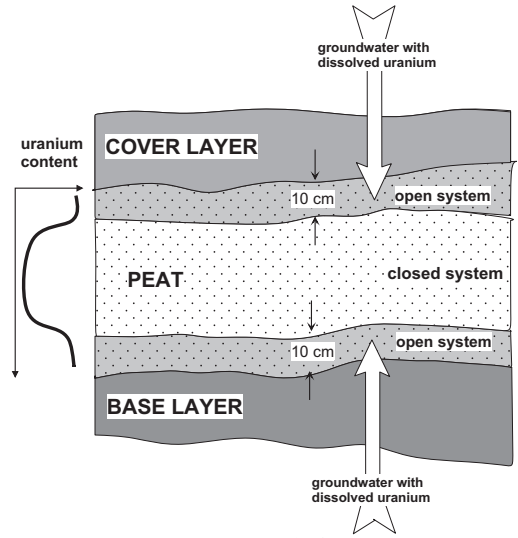


Fig. 4: Schematic of a section through a layer of interglacial fen peat: Samples suitable for $^{230}\text{Th}/\text{U}$ dating can only be expected in the central part of the organic deposit. The top and bottom 10 cm in most cases behaved as open systems with respect to uranium during ageing and are, therefore, unsuitable for $^{230}\text{Th}/\text{U}$ dating.

Abb. 4: Schematisiertes interglaziales Niedermoorprofil: Geeignete Proben für die $^{230}\text{Th}/\text{U}$ -Altersbestimmung können nur im zentralen Bereich des Torfes erwartet werden. Die Randbereiche von 10 cm Mächtigkeit im Hangenden und Liegenden des Torfes bilden gewöhnlich offene Systeme für Uran und erweisen sich deshalb für die $^{230}\text{Th}/\text{U}$ -Datierung als ungeeignet.

samples collected from up to several decimetres depth may have been affected by river water. Exposed fen peat deposits well above the present river level might also have been affected by seeping rainwater. Only drill cores of sufficient length seem to guarantee suitable peat samples. Thorium typically exists in the +4 and +5 oxidation states. The ions are readily hydrolyzed and either precipitate or are adsorbed on detrital particulates (inorganic or organic), clay minerals and iron (oxy)hydroxides. Thorium ions can be transported with humic acids (RICHARDS & DORALE 2003). But movement of humic acids in confined peat beds can be excluded.

Detrital contamination with allochthonous ^{230}Th : Airborne dust or water-borne fine-grained material may become incorporated during the formation of fen peat. These contaminants usually contain thorium, which is often bound to detrital clay. The presence of ^{230}Th in the detrital material means that the $^{230}\text{Th}/\text{U}$ clock shows $^{230}\text{Th}/\text{U}$ ages that are too old. The allochthonous (detrital) ^{230}Th decays during ageing while the content of radiogenic ^{230}Th increases as a result of the decay of ^{234}U . Hence the $^{230}\text{Th}/\text{U}$ age error due to detrital contamination decreases with increasing age. The age error caused by an initial detrital ^{230}Th content is often large and may amount to several 1000 years up to 100,000 years. Therefore, the two or more sources of ^{230}Th (one radiogenic and at least one detrital) have to be distinguished and quantified for correction of the $^{230}\text{Th}/\text{U}$ ages.

Limits on the $^{230}\text{Th}/\text{U}$ method for the dating of peat and lignite: The above-described scenario of both accumulation and binding of uranium in fen peat limits the use of the $^{230}\text{Th}/\text{U}$ dating

method for this material. Accurate $^{230}\text{Th}/\text{U}$ ages of sublayers cannot be expected. During the growth of the moss the peat remains very porous and groundwater or lake water containing dissolved uranium may enter even deeper layers. Hence the $^{230}\text{Th}/\text{U}$ ages will always be younger than the actual ones. Moreover, as fen peat formation is restricted to shallow lakes, which exist mainly at the end of the interglacial or interstadial periods, the corresponding organic deposits often represent the end of the peat-formation period or the interglacial period rather than the beginning. Hence, suitable material of palynologically distinct sub-layers of the first part of the interglacial period can seldom be provided.

Exclusion of raised-bog peat from $^{230}\text{Th}/\text{U}$ dating: Raised-bog peat receives uranium mainly from particulate matter in precipitation. Therefore, the concentration is usually low and there may be different sources of thorium and uranium with different isotopic compositions. Moreover, as raised bog peat is often only

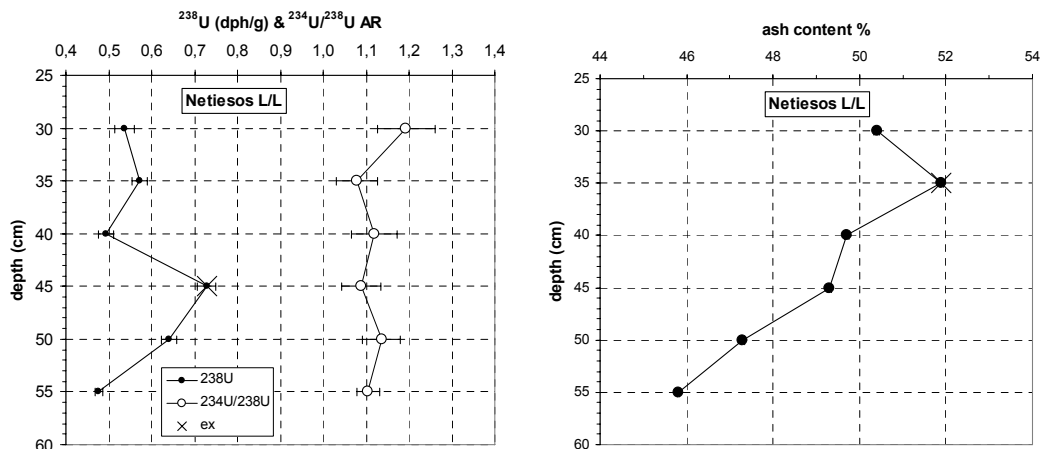


Fig. 5: Depth profiles of the specific ^{238}U activity, the $^{234}\text{U}/^{238}\text{U}$ AR and the ash content at the Netiesos site in Lithuania (GAIGALAS et al. 2005). Samples L-4 and L-5 (crossed dots) have a slightly elevated specific ^{238}U activity. This indicates that the premises of $^{230}\text{Th}/\text{U}$ dating are potentially violated.

Abb. 5: Die Tiefenprofile der spezifischen ^{238}U -Aktivität, des $^{234}\text{U}/^{238}\text{U}$ -Aktivitätsverhältnisses und des Aschegehalts vom Profil Netiesos in Litauen (GAIGALAS et al. 2005). Die Proben L-4 und L-5 (angekreuzte Punkte) haben eine leicht erhöhte ^{238}U -Konzentration und damit ein geringes Potential, für die $^{230}\text{Th}/\text{U}$ -Datierung geeignet zu sein.

weakly decomposed and the content of humic acids is low the ability to absorb actinides is limited. As a consequence, all attempts to date raised-bog peat using the $^{230}\text{Th}/\text{U}$ dating method have failed.

4 Determination of the reliability of data sets for the $^{230}\text{Th}/\text{U}$ dating of fen peat and lignite

Based on the above-described scenario for fen peat formation and the numerous factors that interfere with $^{230}\text{Th}/\text{U}$ dating of fen peat, it is necessary to identify and to discard data sets from samples that are not suitable for $^{230}\text{Th}/\text{U}$ dating. Such sample material does not fulfil the two basic premises of this dating method: (1) It has behaved as an open system with respect to uranium (and thorium) and/or (2) it contained more than one detrital contaminant.

Three simple tests are used for the initial reliability determination: depth profiles of (i) uranium concentration or specific ^{238}U activity, (ii) $^{234}\text{U}/^{238}\text{U}$ AR, and (iii) ash content.

Uranium-depth profiles may show up samples which behaved as an open system with respect to uranium on the basis of elevated uranium concentration relative to the base level in the central part of the peat profile. The $^{234}\text{U}/^{238}\text{U}$ AR in the top and bottom rim layers can be expected to differ from the central part if ^{234}U was preferentially leached or accumulated. The third test is determination of the ash content of the sample: an elevated ash content may indicate the presence of sand layers.

Figure 5 shows the results of the three tests for the Netiesos site (GAIGALAS et al. 2005). The ^{238}U activity of samples L-4 and L-5 (45 and 50 cm) are 30-40 % above the other samples and therefore these samples potentially violate the two basic premises for $^{230}\text{Th}/\text{U}$ dating. The authors do not describe any disturbance of the stratigraphy, and therefore other information is necessary to determine which samples need to be excluded from the age determination. For example, the decrease in ash content with depth points to the possibility of open-system conditions in the upper part of the peat.

Examination of 222 data sets obtained from 33 fen peat sections in Eurasia (e.g. ARSLANOV 2005) has yielded evidence that $^{230}\text{Th}/\text{U}$ ages can be satisfactorily correlated with the SPEC-MAC chronology even from samples with considerably elevated or depleted uranium concentrations, or from samples with anomalous $^{234}\text{U}/^{238}\text{U}$ ARs or that contain a high ash content. Hence, the three tests do not reliably identify samples that do not fulfil the basic premises for $^{230}\text{Th}/\text{U}$ dating.

More sophisticated tests are required. The theoretical background was developed in the 1960s.

KAUFMAN & BROECKER (1965) introduced the concept of a binary mixture of radiogenic ^{230}Th and allochthonous detrital ^{232}Th . If the latter has only one source, the proportion of detrital ^{230}Th can be estimated via the thorium concentration or the ^{232}Th activity. As the half-life of ^{232}Th is very large ($\tau = 14.01 \times 10^9$ a), it behaves similar to a stable isotope. Hence, its activity has not changed during the aging of the sample and hence is a measure of the initial detrital ^{230}Th content in the sample. One of the main tasks of $^{230}\text{Th}/\text{U}$ dating of fen peat and lignite is to determine the initial $^{230}\text{Th}/^{232}\text{Th}$ AR (thorium index) in order to correct the corresponding ages for the detrital contamination (Chapter 5). The principal feature of binary mixing is that any two properties of the two components show a linear correlation. Hence, the plot of any two isotope ratios of uranium and/or thorium in peat samples – according to the KAUFMAN & BROECKER concept – must also yield a straight line. If this requirement is not fulfilled, the basic premises for $^{230}\text{Th}/\text{U}$ dating are not fulfilled: The material did not behave as a closed system with respect to uranium and thorium and/or there were more than two sources of detrital ^{230}Th .

In 1970, OSMOND et al. (1970) developed two mixing plots: $^{230}\text{Th}/^{238}\text{U}$ AR versus $^{232}\text{Th}/^{238}\text{U}$ AR (Osmond-I plot) and $^{234}\text{U}/^{238}\text{U}$ AR versus $^{232}\text{Th}/^{238}\text{U}$ AR (Osmond-II plot) (Figs. 6 and 7 bottom). ROSHOLT (1976) introduced two additional plots, the Rosholt-I ($^{230}\text{Th}/^{232}\text{Th}$ AR versus $^{238}\text{Th}/^{232}\text{Th}$ AR) and Rosholt-II plots

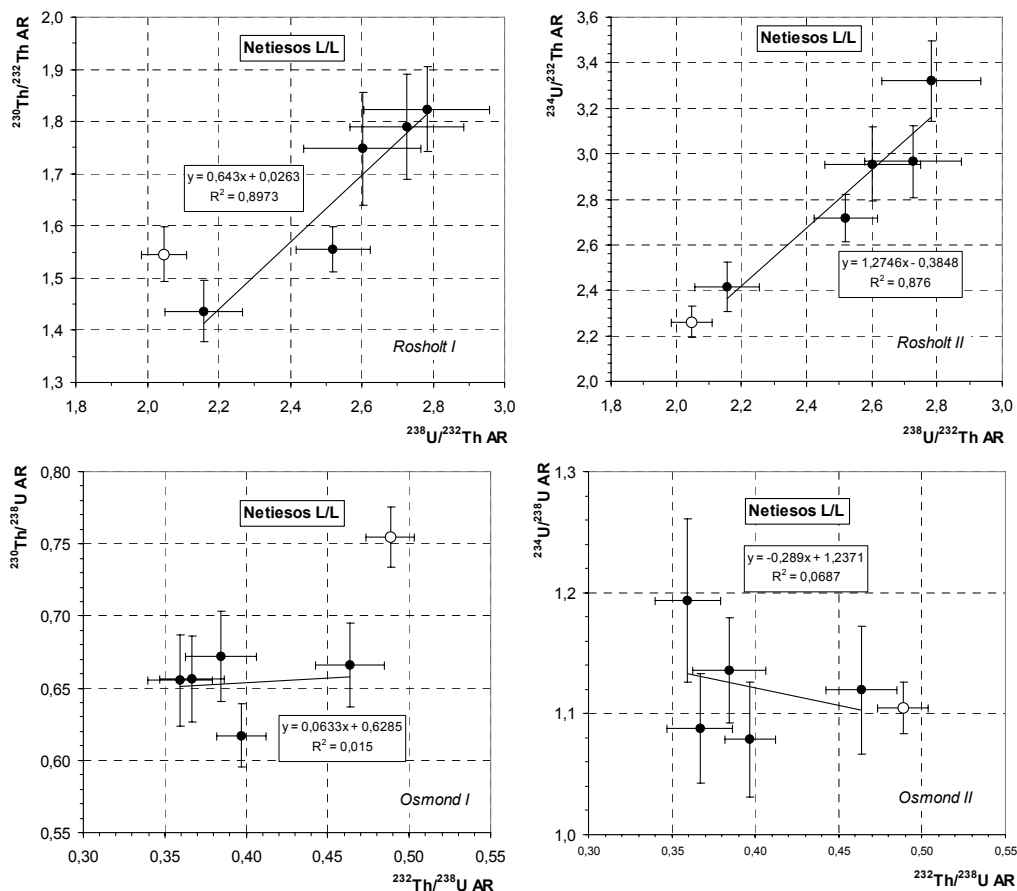


Fig. 6: Top: Rosholt I and Rosholt II plots (ROSHOLT 1976) of the L/L data from the Netiesos site in Lithuania (GAIGALAS et al. 2005): The slopes of the mixing lines equal the $^{230}\text{Th}/^{234}\text{U}$ AR and the $^{234}\text{U}/^{238}\text{U}$ AR, respectively, and the y-intercepts give the $^{230}\text{Th}/^{232}\text{Th}$ AR and $^{234}\text{U}/^{232}\text{Th}$ ARs. The data for sample L-6 (open circle) does not fit the “isochron” and was not used for the $^{230}\text{Th}/\text{U}$ age calculation.

Bottom: Osmond I und Osmond II plots (OSMOND et al. 1970) of the L/L data from the Netiesos site in Lithuania (GAIGALAS et al. 2005): The slopes of the mixing lines give the $^{230}\text{Th}/^{232}\text{Th}$ AR and the $^{234}\text{U}/^{232}\text{Th}$ AR, respectively, and the y-intercepts provide the $^{230}\text{Th}/^{238}\text{U}$ AR and $^{234}\text{U}/^{238}\text{U}$ AR. The data for sample L-6 (open circle) does not fit the mixing lines and was not used for the $^{230}\text{Th}/\text{U}$ age calculation.

Abb. 6: Oben: Rosholt-I und Rosholt-II-Diagramme (ROSHOLT 1976) der L/L-Daten vom Netiesos-Profil in Litauen (GAIGALAS et al. 2005): Die Steigungen der beiden Mischgeraden liefern die Aktivitätsverhältnisse $^{230}\text{Th}/^{234}\text{U}$ und $^{234}\text{U}/^{238}\text{U}$ sowie die Y-Achsenabschnitte die $^{230}\text{Th}/^{232}\text{Th}$ - und $^{234}\text{U}/^{232}\text{Th}$ -Aktivitätsverhältnisse. Der Datensatz der Probe L-6 (weißer Kreis) liegt jenseits der „Isochrone“ und wurde bei der Berechnung des $^{230}\text{Th}/\text{U}$ -Alters nicht berücksichtigt.

Unten: Osmond-I und Osmond-II-Diagramme (OSMOND et al. 1970) der L/L-Daten vom Netiesos-Profil in Litauen (GAIGALAS et al. 2005): Die Steigungen der beiden Mischgeraden liefern die Aktivitätsverhältnisse $^{230}\text{Th}/^{232}\text{Th}$ und $^{234}\text{U}/^{232}\text{Th}$ sowie die Y-Achsenabschnitte die $^{230}\text{Th}/^{238}\text{U}$ - und $^{234}\text{U}/^{238}\text{U}$ -Aktivitätsverhältnisse. Der Datensatz der Probe L-6 (weißer Kreis) liegt jenseits der Mischgerade und wurde nicht bei der Berechnung des $^{230}\text{Th}/\text{U}$ -Alters berücksichtigt.

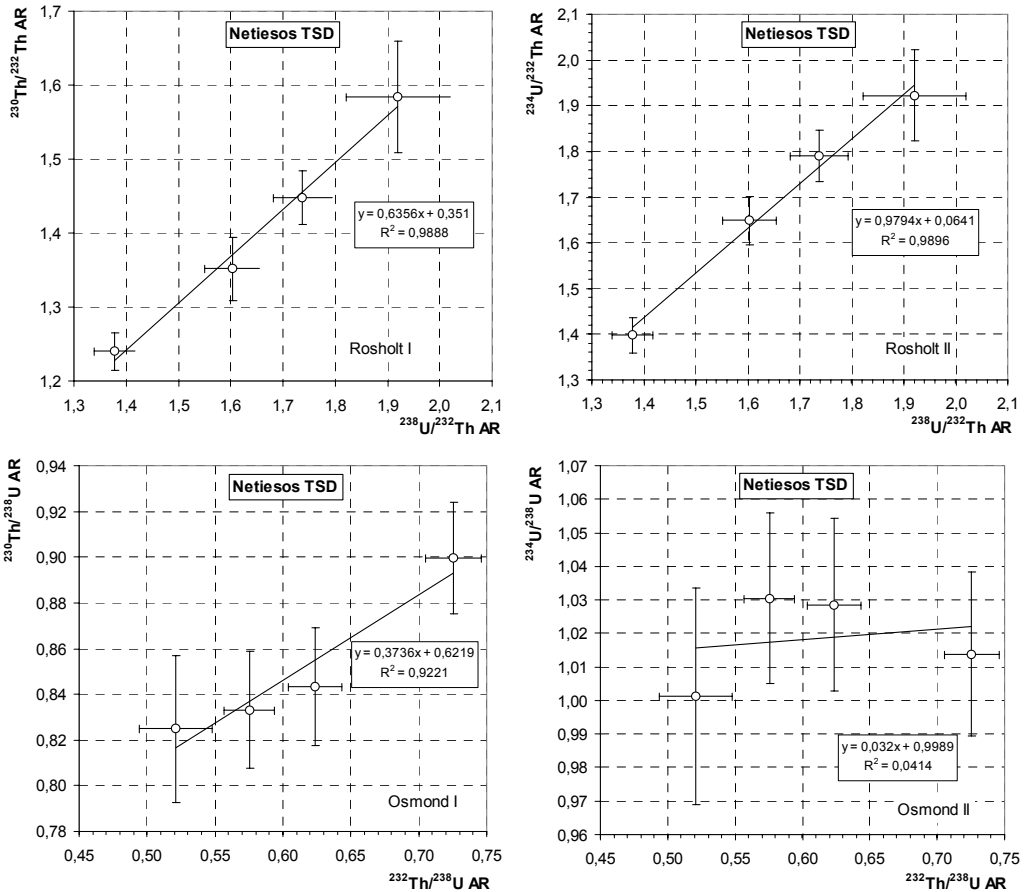


Fig. 7: Top: Rosholt I and Rosholt II plots (ROSHOLT 1976) of the TSD data from the Netiesos site in Lithuania (GAIGALAS et al. 2005): The slopes of the mixing lines equal the $^{230}\text{Th}/^{234}\text{U}$ AR and the $^{234}\text{U}/^{238}\text{U}$ AR, respectively, and the y-intercepts give the $^{230}\text{Th}/^{232}\text{Th}$ AR and $^{234}\text{U}/^{232}\text{Th}$ AR. All data points are on the mixing lines within their uncertainty intervals and are used to calculate $^{230}\text{Th}/\text{U}$ ages.

Bottom: Osmond I and Osmond II plots (OSMOND et al. 1970) of the TSD data from the Netiesos data in Lithuania (GAIGALAS et al. 2005): The slopes of the mixing lines give the $^{230}\text{Th}/^{232}\text{UTh}$ AR and the $^{234}\text{U}/^{232}\text{Th}$ AR, respectively, and the y-intercepts provide the $^{230}\text{Th}/^{238}\text{U}$ AR and $^{234}\text{U}/^{238}\text{U}$ AR. All data points are on the mixing lines within their uncertainty intervals and are used to calculate $^{230}\text{Th}/\text{U}$ ages.

Abb. 7: Oben: Rosholt-I und Rosholt-II-Diagramme (ROSHOLT 1976) der TSD-Daten vom Netiesos-Profil in Litauen (GAIGALAS et al. 2005): Die Steigungen der beiden Mischgeraden liefern die Aktivitätsverhältnisse von $^{230}\text{Th}/^{234}\text{U}$ und $^{234}\text{U}/^{238}\text{U}$ sowie die Y-Achsenabschnitte die $^{230}\text{Th}/^{232}\text{Th}$ - und $^{234}\text{U}/^{232}\text{Th}$ -Aktivitätsverhältnisse. Alle Datensätze genügen den Mischgeraden und den Kriterien der $^{230}\text{Th}/\text{U}$ -Methode.

Unten: Osmond-I und Osmond-II-Diagramme (OSMOND et al. 1970) der TSD-Daten vom Netiesos-Profil in Litauen (GAIGALAS et al. 2005): Die Steigungen der beiden Mischgeraden liefern die Aktivitätsverhältnisse $^{230}\text{Th}/^{232}\text{Th}$ und $^{234}\text{U}/^{232}\text{Th}$ sowie die Y-Achsenabschnitte die $^{230}\text{Th}/^{238}\text{U}$ - und $^{234}\text{U}/^{238}\text{U}$ -Aktivitätsverhältnisse.

($^{234}\text{U}/^{232}\text{Th}$ AR versus $^{238}\text{U}/^{232}\text{Th}$ AR; Figs. 6 and 7 top). The Rosholt-I plot is often incorrectly referred to as an “isochron” plot. The slope of the least-squares fitted straight line in a plot of the $^{230}\text{Th}/^{232}\text{Th}$ versus $^{238}\text{U}/^{232}\text{Th}$ activity ratios of coeval samples equals the $^{230}\text{Th}^*/^{234}\text{U}$ AR of the radiogenic $^{230}\text{Th}^*$ and its intercept on the y-axis equals the decay-corrected initial $^{230}\text{Th}/^{232}\text{Th}$ AR (detrital correction factor = f). The Rosholt-II plot yields the present $^{234}\text{U}/^{238}\text{U}$ AR. Both ARs are required to calculate the $^{230}\text{Th}/\text{U}$ ages (Eq. 1). An “isochron” slope of 0 corresponds to a $^{230}\text{Th}/\text{U}$ age of zero; an “infinite” age has a slope of 1 (radioactive equilibrium).

Figures 6 and 7 show two sets of the four Rosholt-Osmond plots of the ARs obtained from the uranium and thorium analyses of the fen peat at the Netiesos site using the L/L and TSD methods. The displacement of any pair of AR values from any of the four mixing lines indicates that the corresponding sample may not fulfill the basic premises for $^{230}\text{Th}/\text{U}$ dating. Especially, the Rosholt I and II plots give “a misleading impression of an well-defined isochron for data of little statistical power” (LUDWIG 2003). LUDWIG mentions three factors as the reason. (1) The $^{238}\text{U}/^{232}\text{Th}$ and $^{234}\text{U}/^{232}\text{Th}$ ARs tend to reflect the U/Th element ratio. The abundances of both ^{234}U and ^{230}Th of non-zero-age materials are generally highly correlated with the concentration of uranium. Therefore, the scatter of the $^{238}\text{U}/^{232}\text{Th}$ AR greatly amplifies even a dubious isochron trend. As a result the correlation coefficient of the Rosholt isochrons are usually higher than 0.90 and also considerably greater than those of the Osmond plots. (2) The ARs, especially if measured radiometrically, are highly correlated if the denominator isotope ^{232}Th activity is low. Due to the large analytical error the data scatter along lines subparallel to most isochrons and paradoxically strengthen the visual impression of an isochron. (3) The data points of radiometrically measured ARs plotted with error ranges appear to fall more precisely on the regression line than the error ranges imply, even if there is much more scatter than the errors would otherwise indicate

(“zero”-correlation problem; CHAYES 1949, 1971).

To obtain a well defined mixing line, (LUDWIG & TITTERINGTON 1994), data sets from at least two coeval samples with widely differing detrital ^{230}Th content must be available. In the case of fen peat, a much larger number of samples should be analysed.

Coeval samples with similar detrital contamination yield a data cluster, which excludes the possibility of a mixing line. Moreover, the y-axis intercept of the mixing line must be positive, as a decay-corrected negative initial $^{230}\text{Th}/^{232}\text{Th}$ AR is theoretically excluded. The mean of negative decay-corrected AR values can be accepted only if its confidence interval includes zero.

The four mixing plot tests – especially those by Osmond – are a sensitive check for identifying unsuitable data sets. However, it is recommended to include the three simple tests described above in a rigorous examination of the reliability of U/Th age data. Moreover, supplementary tests should be found which also allow an objective and reproducible identification of suitable and unsuitable data sets.

5 Detrital contamination and the correction factors

According to the KAUFMAN & BROECKER (1965) concept, the radiogenic ^{230}Th activity ($[^{230}\text{Th}]^*$) can be calculated from the measured activity $[^{230}\text{Th}]$ as follows:

$$(2) \quad [^{230}\text{Th}]^* = [^{230}\text{Th}] - [^{232}\text{Th}] \cdot f_o \cdot e^{-\lambda_{230} \cdot t} \\ = [^{230}\text{Th}] - f \times [^{232}\text{Th}],$$

where f_o is the initial $^{230}\text{Th}/^{232}\text{Th}$ AR and f is the decay-corrected f_o .

Any correction of radiometrically determined $^{230}\text{Th}/\text{U}$ ages for detrital ^{230}Th is negligible if the measured $^{230}\text{Th}/^{232}\text{Th}$ AR of any sample is smaller than 20 because in this case the detrital ^{230}Th activity is very low.

The detritus-corrected ^{234}U activity ($[^{234}\text{U}]^*$) can be calculated from the Rosholt II plot using an analogous equation:

$$(3) \quad \begin{aligned} [^{234}\text{U}]^* &= [^{234}\text{U}] - [^{232}\text{Th}] \cdot g_o \cdot e^{-\lambda_{234} \cdot t} \\ &= [^{234}\text{U}] - g \cdot [^{232}\text{Th}], \end{aligned}$$

where g_o is initial $^{234}\text{U}/^{232}\text{Th}$ AR and g is the decay-corrected g_o .

Both of these equations to correct for detrital thorium and detrital uranium assume there was only source of contamination.

No detrital correction is needed if most of the samples contain little or no detrital contamination and inferring effects can be excluded, like in the case for speleothems and or corals. In such cases the slope of the mixing lines of the Rosholt-I and Rosholt-II plots equal the $^{230}\text{Th}/^{238}\text{U}$ AR and $^{234}\text{U}/^{238}\text{U}$ AR, respectively, which are required to calculate the $^{230}\text{Th}/\text{U}$ age (Equation 1). The computer program ISOPLLOT (LUDWIG 2003) can be used to calculate the corrected $^{230}\text{Th}/\text{U}$ age and its standard deviation. The theoretical background for calculating the “error-correlated” standard deviation is given by LUDWIG & TITTERINGTON (1994) and LUDWIG (2001; 2003).

In the case of fen-peat dating a statistical check of the individual $^{230}\text{Th}/\text{U}$ ages is recommended in order to identify outliers. This cannot be done with the ISOPLLOT program. Therefore, Equations 2 and 3 should be applied to each data set of peat and lignite samples (GEYH 1994; 2001). The required f and g values and their standard deviations are obtained from the intercept of the mixing lines with the y-axis of the Rosholt-I and Rosholt-II plots or the iterative approximation of the decay-corrected initial $^{230}\text{Th}/^{232}\text{Th}$ AR described below. The standard deviation of the detritus-corrected ^{230}Th activity is calculated using the usual error propagation equations. In most cases the correction for detrital uranium is not necessary because the $^{234}\text{U}/^{238}\text{U}$ AR and $^{232}\text{Th}/^{238}\text{U}$ AR do not correlate for most peat samples. This is also the case for the Netiesos site (Figs. 6 and 7 bottom).

An independent approach to the determination of the detrital correction factor f is to approximate the decay-corrected initial $^{230}\text{Th}/^{232}\text{Th}$ AR iteratively. The $^{230}\text{Th}/^{232}\text{Th}$ AR and $^{234}\text{U}/^{232}\text{Th}$ AR

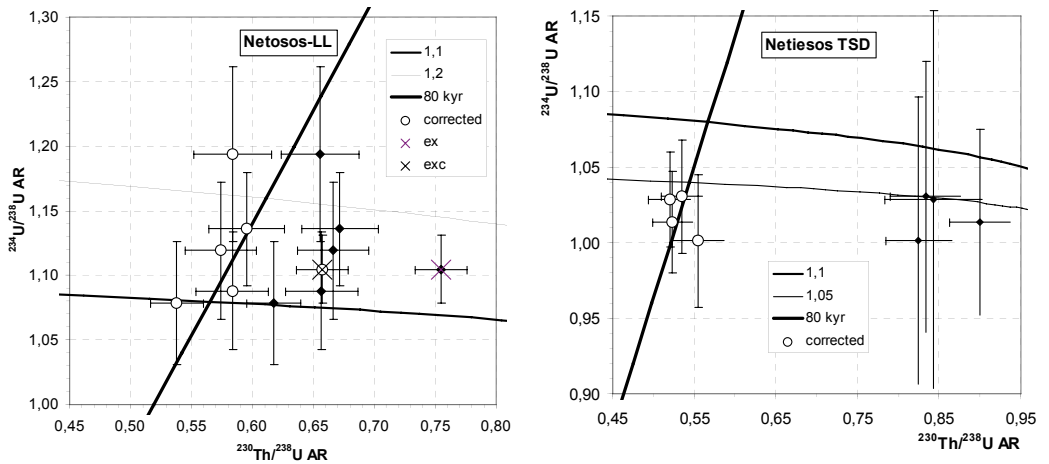


Fig. 8: Isotope-ratio evolution plot: Scatter of the L/L (left) and TSD ARs and their standard deviations in the $^{230}\text{Th}/^{232}\text{Th}$ - $^{234}\text{U}/^{232}\text{Th}$ diagram for the Netiesos site in Lithuania (GAIGALAS et al. 2005). This scatter decreases with increasing detrital correction factor. The L/L data of sample L-6 (crossed symbols) remains far from the cluster.

Abb. 8: Isotopen-Evolutionsdiagramm: Die Streuweite der L/L- und TSD-Aktivitätsverhältnisse des Netiesos-Profiles in Litauen (GAIGALAS et al. 2005) nimmt im $^{230}\text{Th}/^{232}\text{Th}$ - $^{234}\text{U}/^{232}\text{Th}$ -Diagramm mit steigendem detritischen Korrekturfaktor ab. Die L/L-Daten der Probe L-6 (gekreuzter Punkt) bleibt der Punkthäufung fern.

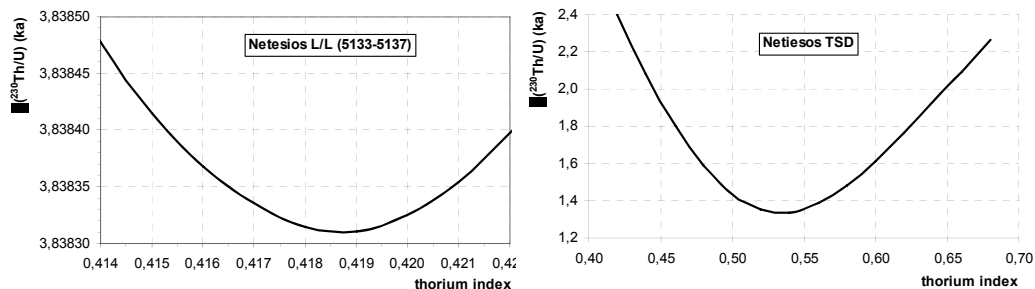


Fig. 9: The scatter represented by the standard deviations of the iteratively corrected L/L and TSD $^{230}\text{Th}/\text{U}$ ages of the Netiesios site in Lithuania (GAIGALAS et al. 2005) approaches a minimum at $f(\text{L/L}) = 0.412 \pm 0.080$ and $f(\text{TSD}) = 0.534 \pm 0.011$. The values for sample L-6 were discarded.

Abb. 9: Streuweite der iterativ-korrigierten $^{230}\text{Th}/\text{U}$ -Alter vom Netiesios-Profil in Litauen (GAIGALAS et al. 2005) erreicht Minima bei $f(\text{L/L}) = 0.412 \pm 0.080$ bzw. $f(\text{TSD}) = 0.534 \pm 0.011$. Der Datensatz der Probe L-6 wurde ausgeschlossen.

of coeval samples containing contamination from different sources scatter widely. Iteratively increasing the detrital correction factor f causes the points to move to the left in the isotope-ratio evolution plot of KAUFMAN & BROECKER (1965). The higher the detrital contamination the faster the points move to the left. The optimum detrital correction factor f is obtained when the points of the cluster are the closest together. The points in the cluster move further apart when the f value is increased beyond the value for the minimum width of the cluster. An elliptical shape of the cluster is caused by differences in the initial $^{234}\text{U}/^{238}\text{U}$ AR (Fig. 8). An effect resulting from α -recoil as observed in carbonates (HENDERSON & SLOWEY 2000) cannot be completely excluded for fen peat.

A plot of the standard deviation of the $^{230}\text{Th}/\text{U}$ age versus the f value provides a measure of the scatter of the individual ages. For the case study from the Netiesios site, the scatter of the L/L and TSD $^{230}\text{Th}/\text{U}$ ages approaches a minimum at $f(\text{L/L}) = 0.412 \pm 0.080$ and $f(\text{TSD}) = 0.534 \pm 0.011$ (Fig. 9). The plots again show that sample L-6 does not fulfill the premises for $^{230}\text{Th}/\text{U}$ dating.

An iterative method developed to determine the decay-corrected initial $^{230}\text{Th}/^{232}\text{Th}$ AR provides an additional test for a rigorous examination of the reliability of the data sets (GEYH 2001). The iteratively detritus-corrected $^{230}\text{Th}/\text{U}$ ages

plotted against the iteratively determined detrital correction factor yields lines with different slopes. If all samples behaved as closed systems with respect to uranium and only one detritus component was present in the samples, the lines should intersect at only one point – the actual f value. If one or more lines do not pass through this point the corresponding sample is identified as unsuitable.

For the case study of the Netiesios site, the bold line for sample L-6 in the L/L plot belongs to outlier data which do not fulfill the premises of the $^{230}\text{Th}/\text{U}$ dating method. This line does not pass through the common intersection point.

6 $^{230}\text{Th}/\text{U}$ age and random uncertainty

The data sets suitable for calculation of a $^{230}\text{Th}/\text{U}$ age and its standard deviation are selected on the basis of the rigorous examination of their reliability described above. The detrital correction is applied to each measured ^{230}Th activity (Equation 2; Chapter 5) and the $^{230}\text{Th}/\text{U}$ ages are calculated using Equation 1. The χ^2 test is then applied to the corrected $^{230}\text{Th}/\text{U}$ ages, outliers are discarded, and the mean $^{230}\text{Th}/\text{U}$ age is calculated. The standard deviation of the mean $^{230}\text{Th}/\text{U}$ age is obtained using the Gaussian method for error determination of the $^{230}\text{Th}/\text{U}$ ages of the samples.

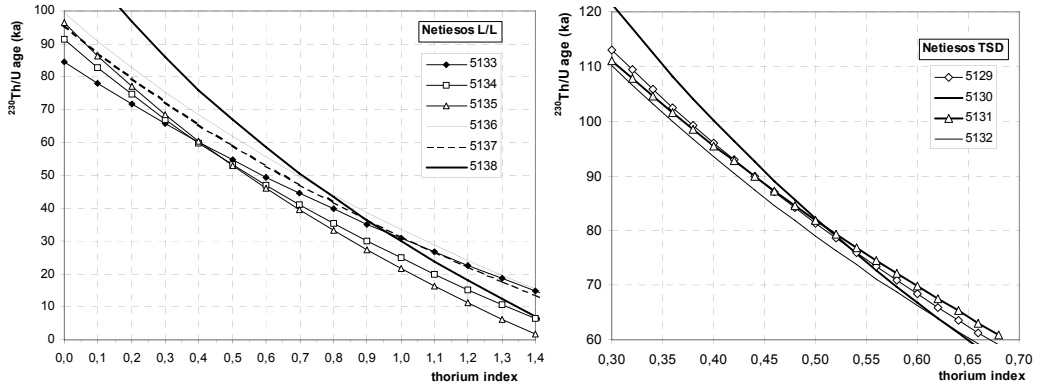


Fig. 10: Change of the detritus-corrected $^{230}\text{Th}/\text{U}$ ages with the iteratively increased f . Five lines of the L/L data and all lines of the TSD data have common intersection points at 61.6 ka and 76.4 ka, respectively. The bold line of the sample L-6 does not.

Abb. 10: Änderung der detritus-korrigierten $^{230}\text{Th}/\text{U}$ -Alter bei iterativ erhöhtem f -Wert. Fünf Linien der L/L-Daten und alle der TSD-Daten haben nahe zusammen liegende Schnittpunkte bei 61.6 ka und 76.4 ka. Die dicke Linie des L/L-Datensatzes der Probe L-6 liefert davon entfernte Schnittpunkte.

7 Minimum $^{230}\text{Th}/\text{U}$ age of samples

The limited precision of uranium and thorium isotope activity measurements means that there is an upper limit of the $^{230}\text{Th}/\text{U}$ age that can be determined. This limit is the minimum $^{230}\text{Th}/\text{U}$ age of a sample, corresponding to the maximum $^{230}\text{Th}/\text{U}$ age that can be determined with the $^{230}\text{Th}/\text{U}$ dating method. This minimum $^{230}\text{Th}/\text{U}$ age is expressed, for example, as >350 ka. Surprisingly, minimum ages are usually not estimated.

The isotope-ratio evolution plot of KAUFMAN & BROECKER (1965) can be used to develop a method for calculating the minimum $^{230}\text{Th}/\text{U}$ age (GEYH & MÜLLER 2005). X in Equation 1 is replaced by $^{230}\text{Th}/^{238}\text{U}$ AR and Y by the decay-corrected initial $^{234}\text{U}/^{238}\text{U}$ AR. Thus,

$$(4) \quad X = \frac{\lambda_{230}}{\lambda_{230} - \lambda_{234}} (Y - 1) \cdot (1 - e^{-(\lambda_{230} - \lambda_{234}) \cdot t}) + (1 - e^{-\lambda_{230} \cdot t})$$

The decay-corrected initial $^{234}\text{U}/^{238}\text{U}$ (Y) is given by

$$(5) \quad Y = (Y_o - 1) \cdot e^{-\lambda_{234} \cdot t} + 1$$

The uranium and thorium ARs of an infinitely old sample are given by a straight line in the isotope-ratio evolution plot. The equation for this isochron is derived from Equation 4 assuming an age $t = \infty$:

$$(6) \quad X = \frac{\lambda_{230}}{\lambda_{230} - \lambda_{234}} \cdot Y - \frac{\lambda_{234}}{\lambda_{230} - \lambda_{234}} + 1$$

The ellipse defined by the X and Y random uncertainties of any data point should touch but never cut the infinite-age isochron. When the infinite-age isochron is tangent to the ellipse of the X and Y random uncertainties, the minimum $^{230}\text{Th}/\text{U}$ age t_{\min} of the sample can be determined from the X_{\min} and Y_{\min} (Fig. 11). The equation of the ellipse with maximum axis lengths of $\pm 2\sigma X$ and $\pm 2\sigma Y$ around X_{\min} and Y_{\min} is given by

$$(7) \quad \left(\frac{X - X_{\min}}{2\sigma X} \right)^2 + \left(\frac{Y - Y_{\min}}{2\sigma Y} \right)^2 = 1$$

Equations 4-7 are solved iteratively by decreasing or increasing the age until the error ellipse of a date touches the infinite-age isochron. A detritus-corrected $^{230}\text{Th}/\text{U}$ date is considered reasonable if it is smaller than the corresponding minimum $^{230}\text{Th}/\text{U}$ sample age t_{\min} (GEYH &

MÜLLER 2005). Otherwise the result has to be expressed as the minimum $^{230}\text{Th}/\text{U}$ age.

8 Precision and accuracy of $^{230}\text{Th}/\text{U}$ dating of fen peat

The precision and accuracy of $^{230}\text{Th}/\text{U}$ dating of fen peat may be illustrated by three case studies. GAIGALAS et al. (2005) published L/L and TSD $^{230}\text{Th}/\text{U}$ ages of 108.8 ± 8.7 ka and 80.3 ± 5.9 ka, respectively, for fen peat deposits at the Netiesos site in Lithuania. The higher value from the L/L data was obtained after rejection of the data from the uppermost (L-1: 30 cm) and lowermost (L-6: 55 cm) peat samples. A rigorous examination of the sample data sets was not carried out. The decision to reject these two values was based neither on an elevated ^{238}U activity nor on ash content (Fig. 5).

A rigorous check of the reliability of the L/L data sets identified only sample L-6 as unsuitable for $^{230}\text{Th}/\text{U}$ dating. The L-6 data do not fit the mixing lines of the Rosholt-I and Osmond-I plots (Fig. 6), no minimum cluster size was found when the L-6 data was included in the evolution plot (Fig. 8), and in the iterative plot the L-6 line does not pass through the common intersection point of the L-1 to L-5 lines. Sample L-2 has a slightly elevated ash content (Fig. 5) and its AR values do not fit well the mixing lines of the Rosholt-I and Osmond-I plots (Fig. 7). Other indications that L-2 is unsuitable were not found. A rigorous check of the reliability of the TSD data sets does not identify any outliers. The $^{230}\text{Th}/\text{U}$ age was calculated with the Equations 1 and 2 and yielded 78.4 ± 4.3 ka and a detrital correction factor of 0.519 ± 0.161 . The χ^2 test applied to the four data sets yields $\chi^2 = 0.4$. Iterative evaluation yielded an age of 76.4 ± 1.3 ka with a detrital correction factor of 0.534 ± 0.010 .

A $^{230}\text{Th}/\text{U}$ age of 72.6 ± 10.0 ka and a detrital correction factor of 0.268 ± 0.365 and a $\chi^2 = 0.8$ were calculated using Equations 1 and 2, discarding the data from samples L-2 and L-6. Iterative evaluation yielded a detrital correction factor $f = 0.456 \pm 0.060$. An age of 77.0 ± 11.1 ka and a detrital correction factor $f = 0.199 \pm 0.381$ ($\chi^2 = 0.4$) were obtained when only the

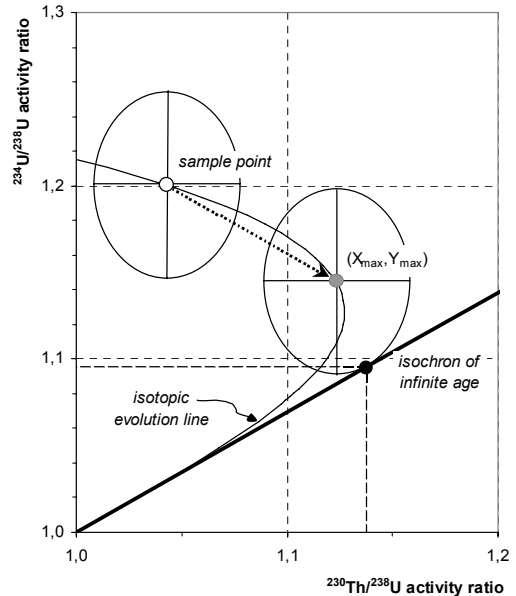


Fig. 11: Isotope-ratio evolution plot (KAUFMAN & BROECKER 1965) to determine the minimum $^{230}\text{Th}/\text{U}$ age t_{\min} of a sample. When the data point (open circle) moves on the evolution line to the right with increasing age (grey circle) until its ellipse of 2σ X and Y uncertainties touches the infinite-age isochron (grey circle), the corresponding $X_{\min} = ^{230}\text{Th}/^{238}\text{U}$ AR and $Y_{\min} = ^{234}\text{U}/^{238}\text{U}$ AR yield the minimum age t_{\min} .

Abb. 11: Isotopenentwicklungsdiagramm nach KAUFMAN & BROECKER (1965) zur Bestimmung des methodischen Minimum- $^{230}\text{Th}/\text{U}$ -Alters einer Probe. Der weiße Punkt bewegt sich mit wachsendem Alter auf der Entwicklungslinie bis zu dem grauen Punkt, an dem die Ellipse der 2σ X- und Y-Standardabweichungen die Isochrone für unendliche Alter trifft (schwarzer Punkt). Die entsprechenden $X_{\min} = ^{230}\text{Th}/^{238}\text{U}$ - und $Y_{\min} = ^{234}\text{U}/^{238}\text{U}$ -Aktivitätsverhältnisse liefern das Minimum- $^{230}\text{Th}/\text{U}$ -Alter der Probe.

data from sample L-6 was discarded and the iterative evaluation yielded $f = 0.419 \pm 0.080$. Disregarding sample L-6 shifts the calculated lines in the Rosholt-I and Osmond-I of the L/L data plots to the right and downwards, respectively. This yields an acceptable $^{230}\text{Th}/\text{U}$ age of 77.0 ± 11.1 ka. It is in agreement with the TSD $^{230}\text{Th}/\text{U}$ age of 78.4 ± 4.3 ka. This age falls within the time span of MIS 5a (Brørup interstadial) between 76 and 78 ka of the SPECMAP time scale (MARTINSON et al. 1987).

The second case study presented here was the first attempt to check whether it is possible to date fen peat and lignite by the ²³⁰Th/U dating method (GEYH 2001). Eemian peat deposits in Europe seemed to be the most suitable for this as they contain the only Quaternary interglacial material that can be palynologically firmly identified and related to the SPECMAP timescale (GEYH 2001). Unfortunately, no cores were available and all samples were collected from exposures with the associated risk of interfering processes.

Seven sites in Germany yielded a mean ²³⁰Th/U age of 111.9 ± 1.2 ka for the detritus-corrected ²³⁰Th/U ages and 114.4 ± 1.9 ka using Equations 1 and 2. The agreement is good but the ages are below what is expected from SPECMAP chronology. There are several possible reasons for this. The data sets were not subjected to a rigorous reliability test. The samples might not have completely fulfilled the two basic premises for ²³⁰Th/U dating due to the lack of optimum sampling conditions. In any case, the ²³⁰Th/U ages and their standard deviations for these seven sites may be considered as representative of the attainable accuracy of ²³⁰Th/U dating of fen peat. The precision, however, is good enough to firmly assign the dated Eemian interglacial deposits to MIS 5e but is not sufficient to assign a precise chronological age to sub-layers.

In the third case study, the ²³⁰Th/U ages for Holsteinian/Hoxnian Interglacial peat were obtained from drill cores and agree better with the SPECMAP chronology (GEYH & MÜLLER 2005). The Hoxnian Interglacial in England has been correlated with the Holsteinian Interglacial (TURNER 1970). A mean ²³⁰Th/U age of 317 ± 14 ka was obtained from the fen peat at Tottenham in the Nar Valley of NW Norfolk (ROWE et al. 1997). The ²³⁰Th/U ages from the Marks Tey site in the UK, which has been palynologically classified as Holsteinian, (ROWE et al. 1999), have a very low precision and could be interpreted only by using a probabilistic approach. The authors concluded that these deposits correlate with MIS 11 or some older stage with 87 % confidence. Using Equations 4 to 7, ²³⁰Th/U minimum ages of >165 to >245 ka

were obtained for their data (GEYH & MÜLLER 2005). This implies that the measured U and Th data reliably allow only the statement that the geological age of the Holsteinian samples is greater than MIS 7.

B

The Bossel site about 30 km west of Hamburg contains two organic layers: one above and one below the sediments of the Holsteinian Sea. This location became the reference site for the Holsteinian Interglacial in northern Germany as declared by the European Commission on the Stratigraphy of the Quaternary. The “isochron” plot of the AR of ten samples from both peat layers yielded a mean detritus-corrected ²³⁰Th/U age of 323 ± 5 ka with individual detritus-corrected ²³⁰Th/U ages between 298 and 347 ka. The lower peat layer yielded a mean detritus-corrected ²³⁰Th/U age of 312 ± 3 ka with individual detritus-corrected ²³⁰Th/U ages between 293 and 369 ka. The four ²³⁰Th/U ages of the upper peat layer yielded a detritus-corrected mean age of $327+50/-37$ ka.

These case studies provide evidence that ²³⁰Th/U dating of fen peat and lignite does not yield high-precision ages. The accuracy may be around 10,000 a. But this is adequate to reliably relate any interglacial peat deposit to only one of the documented warm periods of the SPECMAP chronology (MARTINSON et al. 1987).

9 Sampling, analysis and isotope ratio determination

To fulfil the requirements for isochron²³⁰Th/U dating the samples must be coeval from the same interglacial or interstadial. For this it is sufficient to take samples from anywhere within the centre of the fen peat deposit. Rim layers of at least 10 cm thickness have to be excluded from the analysis. It is recommended to take the samples from a monolith or a drill core. At least four samples with a wide range of concentration of the detrital contamination, but even better more should be dated.

The sample preparation is more or less the same for both measurement methods for determining the ARs and consists of several steps. Ultrapur

acids which are free of even traces of uranium and thorium have to be used. The chemical preparation should be done in a clean-air laboratory in order to lower the risk of any detrital contamination. This is especially important if small samples are treated for measurement in a TIMS (thermal-ion mass spectrometer) or MC-ICP-MS (multi-collector inductively coupled plasma mass spectrometer). In radiometric laboratories repeated and comprehensive contamination tests have to be carried out.

Preferentially, peat should be treated by "total sample dissolution" (TSD) (BISCHOFF & FITZPATRICK 1991; LUO & KU 1991). This method includes both lattice-bound and adsorbed thorium. Empirically it has been found that $^{230}\text{Th}/\text{U}$ ages obtained by the "leachate/leachate" method (SCHWARCZ & LATHAM 1989; KAUFMAN 1993) often deviate from the palynologically expected age and do not fit the SPECMAP timescale. Selective leaching of peat samples does not adequately separate the radiogenic and detrital ^{230}Th components. In addition, the radiogenic ^{230}Th may become readsorbed on detrital material during incomplete dissolution. The following steps are taken in the "total sample dissolution" method:

- The samples of 3-5 g of fresh or 0.3-0.5 g of dry peat are taken from visibly undisturbed micro ranges within monoliths or cores.
- The surface of each sample is removed. The peat is then broken into pieces, dried and ignited in a quartz tube at a maximum temperature of about 800 °C in a stream of oxygen to burn all organic material. Insoluble glass beads do not form below this temperature and, therefore, loss of uranium and thorium is avoided.
- The weighed peat ash is treated with NaOH in order to remove traces of humic acids. The residue is completely dissolved in a con. HF/HNO₃/HCL mixture to completely dissolve uranium and thorium. If the L/L technique is applied, the sample is leached with a HNO₃/HCL mixture for at least 6 h. Next, 0.5 mL of a ^{229}Th spike and 0.5 mL of a $^{233}\text{U}/^{236}\text{U}$ double spike are added as a check of thermal fractionation in the ion source.

The solution is heated with an infrared lamp for up to 20 h in order to ensure perfect mixing of sample and spike.

- The leachate is separated from the residue by centrifuging.
- Uranium and thorium are co-precipitated with Fe(OH)₃.
- The precipitate is dissolved in con. HNO₃.
- Uranium and thorium are separated from the mixture by standard ion-exchange chemistry using an actinide-specific resin (DOWEX 1×8 100-200 mesh). Thorium is eluted from the column by dilute HCl, uranium is then removed with HBr.

$^{230}\text{Th}/\text{U}$ age determinations are preferentially done now by TIMS and MC-ICP-MS. Details are given by SCHOLZ & HOFFMANN (2008). Radiometric measurements of the ARs require at least ten times more material and are less precise by one order of magnitude. The extracted uranium and thorium are electroplated on stainless steel discs. The latter are put into an alpha spectrometer and the emitted alpha particles are counted. Due to the long ^{230}Th half-life of 75,690 ka, only one out of 6×10^6 atoms of ^{230}Th decays during the common measurement time of one week. Hence, especially the counting statistics limits the precision of radiometric $^{230}\text{Th}/\text{U}$ ages.

10 Conclusions

Interglacial fen peat and lignite with ages of up to 350-500 ka can be dated with the $^{230}\text{Th}/\text{U}$ dating method. The main prerequisite is that the samples are collected from the parts of a deposit that behaved as closed system with respect to uranium during ageing and have no more than one detrital component. In the case of fen peat, the bottom and top rim layers of about 10 cm have to be discarded. Drill cores yield better samples than monoliths collected from exposed outcrops. Small samples of a few cm³ (about 1 g of dry peat) are sufficient for TIMS and MC-ICP-MS $^{230}\text{Th}/\text{U}$ dating. Radiometric $^{230}\text{Th}/\text{U}$ dating requires at least ten times more material. The coeval samples should have a wide range in the amount of detrital contamination. A

single uranium and thorium isotope analysis of peat does not yield a reliable $^{230}\text{Th}/\text{U}$ age. The TSD analytical method seems to be superior to the L/L method. Reliable $^{230}\text{Th}/\text{U}$ ages, however, always require a comprehensive and rigorous reliability check of the measured data in order to identify samples that were unsuitable for $^{230}\text{Th}/\text{U}$ dating. The accuracy of $^{230}\text{Th}/\text{U}$ ages of fen peat and lignite is around 10 ka, which is sufficient for reliable correlation with the warm periods of the global marine $\delta^{18}\text{O}$ chronology.

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Luminescence dating: basics, methods and applications

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Abstract: Luminescence dating is a tool frequently used for age determination of Quaternary materials such as archaeological artefacts, volcanic deposits and a variety of sediments from different environmental settings. The present paper gives an overview of the physical basics of luminescence dating, the necessary procedures from sampling to age calculation, potential problems that may interfere with correct age calculation as well as procedures to identify and resolve those problems. Finally, a brief summary of the most common fields of application is given ranging from artefacts to the variety of different sediments suitable for luminescence dating.

[Lumineszenzdatierung: Grundlagen, Methoden und Anwendungen]

Kurzfassung: Lumineszenzdatierung ist eine häufig angewendete Methode zur Altersbestimmung quartärer Materialien, wie z.B. archäologischer Artefakte, vulkanischer Ablagerungen oder von Sedimenten unterschiedlicher Ablagerungsräumen. Das vorliegende Manuskript gibt einen Überblick über die physikalischen Grundlagen der Lumineszenzdatierung, erläutert die notwendigen Prozeduren von der Probenahme bis hin zur Altersberechnung, diskutiert potenzielle Probleme die eine korrekte Altersberechnung beeinträchtigen können und stellt Verfahren vor, mit denen diese Probleme erkannt und beseitigt werden können. Abschließend wird ein kurzer Überblick über die gängigsten Anwendungsgebiete gegeben, von Artefakten bis hin zu verschiedenen Sedimenten, die für Lumineszenzdatierung geeignet sind.

Keywords: Luminescence, physical dating methods, archaeology, geosciences, Quaternary

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1 Introduction

Luminescence techniques enable evaluation of the time that has elapsed since mineral grains crystallised, were last exposed to daylight or heated to a few hundred degrees Celsius. The method uses an optically and thermally sensitive light or *luminescence* signal in minerals such as quartz and feldspar. During exposure to light or heat the luminescence signal within the grains is erased (optically bleached or thermally annealed) until it is completely removed (zeroed) (Fig. 1). Once the grains are sealed from daylight and remain at normal environmental temperatures, the luminescence signal accumulates again, being induced by naturally occurring radioactivity. For dating, the amount of absorbed energy per mass of mineral ($1 \text{ J kg}^{-1} = 1 \text{ Gy}$ (Gray)) due to natural radiation exposure since zeroing - known as the palaeodose - is determined by comparing the natural luminescence signal of a sample with that induced by artificial irradiation. Several laboratory techniques have been developed to accomplish the necessary stimulation and recording of the weak but measurable luminescence emitted from minerals. The time elapsed since the last daylight exposure or heating is calculated by dividing the palaeodose by the dose rate, the latter representing the amount of energy deposited per mass of mineral due to radiation exposure acting on the sample over a certain time (Gy a^{-1}). This relation is represented by the following simple equation:

$$\text{Luminescence age (a)} = \frac{\text{Palaeodose (Gy)}}{\text{Dose rate (Gy a}^{-1}\text{)}} \quad (1)$$

Luminescence dating has been applied to a wide range of topics within Quaternary research such as landscape evolution, palaeoclimate, geohazards and (geo)-archaeology, and has undergone several important methodological refinements since its early days. One of the first publications suggesting the use of thermoluminescence (TL) as a research tool was by DANIELS et al. (1953) and a few years later TL was used to date ceramics (GRÖGLER et al. 1958; AITKEN et

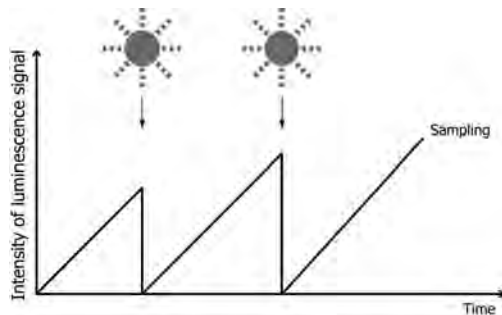


Fig. 1: The basic principle of luminescence dating: zeroing of the signal during daylight exposure (or by heating) and subsequent accumulation of the signal with time, when the material is sealed from daylight and not exposed to heat.

Abb. 1: Die Grundlage der Lumineszenzdatierung: Nullstellung des Signals während Tageslichtexposition (oder durch Erhitzen) und anschließende Akkumulierung des Signals während der Zeit, in der das Material vor Sonnenlicht geschützt bzw. keiner Erhitzung ausgesetzt ist.

al. 1964, 1968). An important benchmark was the application of TL to the dating sediments (WINTLE & HUNTLEY 1979, 1980; WINTLE 1980). Another breakthrough came with the introduction of optical stimulation by both visible (HUNTLEY et al. 1985) and infrared light (HÜTT et al. 1988). More recent advances concern the development of measurement procedures. Of these, notable developments are the use of a single aliquot (DULLER 1991; MURRAY & WINTLE 2000), as well as single grain dating techniques (MURRAY & ROBERTS 1997; DULLER et al. 2000), new analytical tools such as linearly modulated luminescence (BULUR 1996), radiofluorescence (KRBETSCHKE et al. 2000; ERFURT et al. 2003) and spatially-resolved luminescence (GREILICH et al. 2002; GREILICH & WAGNER 2006).

The aim of the present paper is to give an overview of the method as a whole with particular emphasis towards non-luminescence specialist researchers interested in the application of luminescence dating in Quaternary research. It is neither intended to give a full literature review nor to explain every technical aspect in too much detail but rather to give the reader

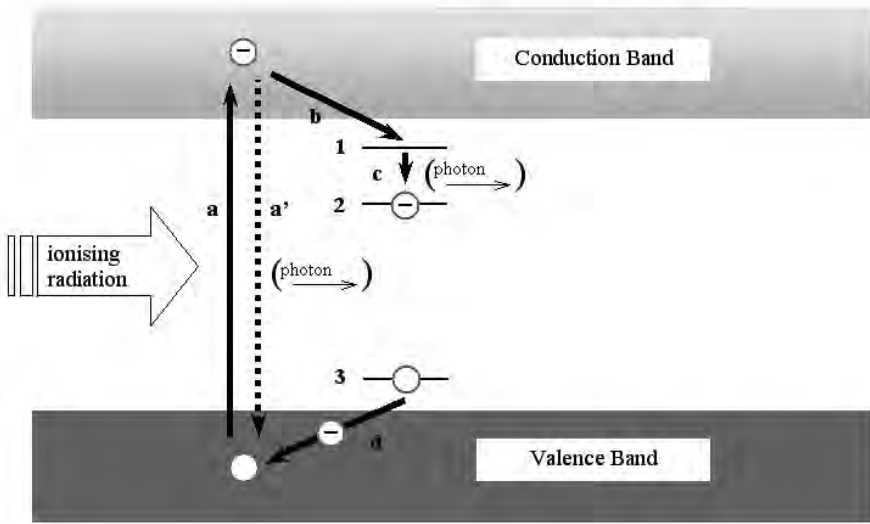


Fig.2.1: Basic processes leading to a latent luminescence signal (schematic):

- a – A valence electron is excited by ionising radiation, and has sufficient energy to reach the conduction band, leaving a hole in the valence band.
- a' – Most of the excited electrons dissipate their energy by recombining immediately with a hole in the valence band; the transition is sometimes accompanied by photon emission.
- b, c – Prompt transition of a few excited electrons into localised energy states below the band edge resulting in electron trapping; light is potentially emitted during these processes..
- d – A hole in the valence band may be filled by electrons from localised levels above the valence band edge; hence the hole transfers from the valence band to the localised level.

Abb. 2.1: Grundlegende Prozesse, die zu einem latenten Lumineszenzsignal führen (schematisch):

- a – Ein Elektron aus dem Valenzband wird durch ionisierende Strahlung angeregt, es besitzt ausreichend Energie um das Leitungsband zu erreichen und hinterlässt ein Loch im Valenzband.
- a' – Die Mehrheit der angeregten Elektronen verliert ihre Energie durch unverzügliche Rekombination mit einem Loch im Valenzband; dieser Übergang ist mitunter mit der Emission von Photonen verbunden.
- b, c – Spontane Übergänge einiger angeregter Elektronen in lokalisierte Energiezustände unterhalb der Bandkante („Fallen“) führen zu stabilem Elektroneneinfang; zum Teil wird bei diesen Prozessen Licht emittiert.
- d – Ein Loch im Valenzband kann durch Elektronen aus lokalisierten Niveaus oberhalb der Valenzbandkante aufgefüllt werden; damit geht das Loch vom Valenzband in den lokalisierten Zustand über.

a firm grounding for the potential, limitations and modern approaches of quality control in luminescence dating of Quaternary materials.

2 Physical background

2.1 Origin of the luminescence signal

The process behind the phenomenon of luminescence is best described by the energy-level

representation of insulating solids (Figs. 2.1 and 2.2). The basic mechanism is that ionising radiation causes the excitation of atoms within the crystal lattice, leading to activated electrons at higher energy states. The vast majority of the activated electrons leave the activated states instantaneously, but some charge is captured at electronic levels below the edge of the conduction band (Fig. 2.1 a-c). These levels are called *electron traps*. The charge deficit creates a hole

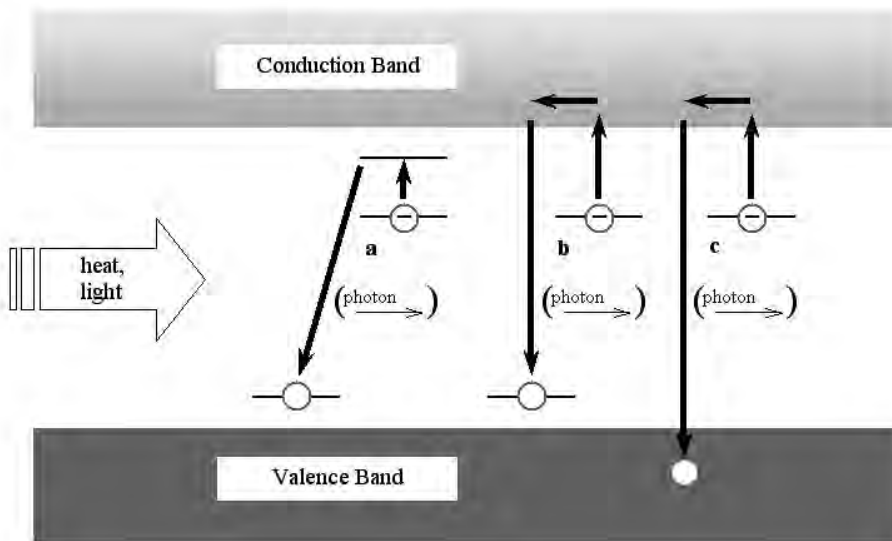


Fig. 2.2: Stimulation of luminescence by heat and light. Possible processes leading to the emptying of trap levels after excitation by light or heat (schematic):

- a, b – Recombination with localised holes above the valence band edge; a) the electron is first transferred to an unstable level below the conduction band; b) the recombines via the conduction band.
- c – Recombination with a hole in the valence band.
The energy loss may be released in the form of light (= luminescence).

Abb. 2.2: Lumineszenzanregung durch Wärme oder Licht. Mögliche Prozesse, die zum Entleeren der Fallen-niveaus nach Anregung durch Licht oder Hitze führen (schematisch):

- a, b – Rekombination mit lokalisierten Löchern oberhalb der Valenzbandkante ; im Fall a wird das Elektron zuerst in einen instabilen Zustand unterhalb des Leitungsbandes transferiert, in Fall b rekombiniert es über das Leitungsband.
- c – Rekombination mit einem Loch im Valenzband.
Der Energieverlust kann als Licht freigesetzt werden (= Lumineszenz).

in the valence band, which may move to form *recombination centres* within the band gap (Fig. 2.1 d). Both electron traps and recombination centres are linked to lattice defects such as oxygen vacancies or foreign atoms (e.g., Al^{3+} instead of Si^{4+} in the quartz lattice). It is important to note that various types of defects exist in the different minerals used in luminescence dating and that the energy representation shown in Fig. 2 very much simplifies the complex situation found in natural minerals. In reality, several kinds of traps and recombination centres exist (cf., CHEN & MCKEEVER 1997; KRBETSCHKE et al. 1997; MCKEEVER & CHEN 1997; MCKEEVER 2001). A basic characterisa-

tion of an electron trap is its energetic depth below the conduction band. The trap depth represents the energy necessary to lift the electron from the trap back to the conduction band. Only electrons captured at traps of a certain depth (in energy terms ~ 1.6 eV or more) remain captured for several million years, which is the prerequisite for dating Quaternary materials since the possibility of charge leakage is less likely for longer storage times (AITKEN 1998). With time, more and more electrons will be captured at the traps within a crystal and so the latent luminescence signal within the mineral will increase. Since the number of traps is limited the latent luminescence cannot increase indefinitely but

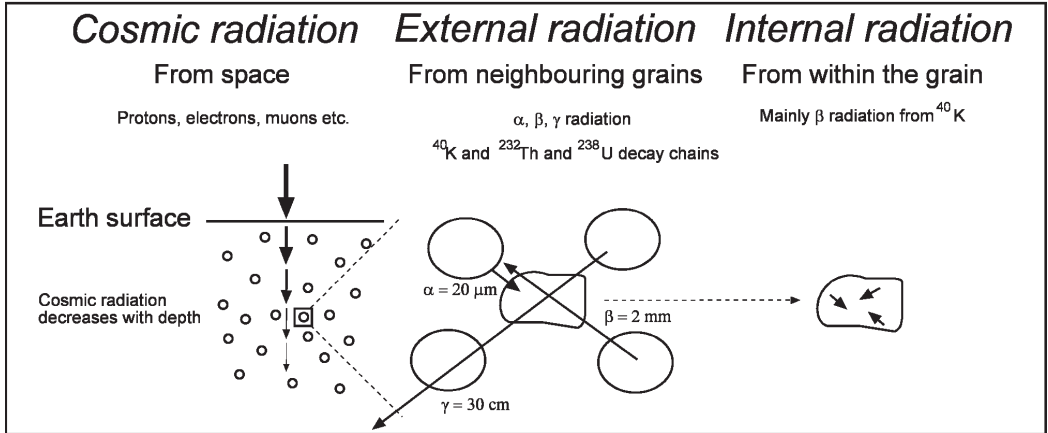


Fig. 3: Different components relevant for the calculation of dose rate for luminescence dating.

Abb. 3: Verschiedene Komponenten, die für die Berechnung der Dosisleistung relevant sind.

will reach a saturation value (saturation dose). Electrons are released from the traps once the mineral is exposed to a particular stimulation energy (Fig. 2.2). In this context it is necessary to consider that the depth of a trap defines the kind of stimulation the captured electron is sensitive to. The deeper the trap, the higher is the required stimulation energy. Once the electron has been lifted to the appropriate recombination level within or below the conduction band, there is a certain probability that it will recombine at an electron hole (recombination centre) and in doing so, release some of the stored energy in the form of light. It is this light released during the recombination process that is referred to as luminescence, and is dependent in various ways on whether TL, optically stimulated luminescence (OSL), etc., is used as the stimulation method. The intensity of light emitted by a sample during stimulation is proportional to the number of recombining electrons and, hence, proportional to the amount of electrons trapped prior to stimulation. The latter is a function of both time and the number of electrons trapped per time interval, which in turn is a function of the dose rate. As a consequence, the intensity of emitted luminescence is linked to the total energy imparted to the mineral due to radioactive exposure since the signal was last zeroed via sufficient stimulation energy.

2.2 Environmental dose rate

Natural ionising radiation occurs in the form of alpha, beta and gamma radiation and cosmic rays (Fig. 3). Alpha radiation consists of Helium nuclei (He^{2+}) that have a penetration depth in sediment of about 20 μm and hence penetrate only the outer part of sand-sized mineral grains. Beta particles (electrons) and gamma rays (photons) have a much higher penetration depth in sediments, of the order of a few mm or dm, respectively, and thus pervade the whole mineral. Because of their small size compared to the penetration depth of alphas, silt and clay-sized particles are completely penetrated by all three types of radiation. In addition to their low penetrating power, a further important factor in the context of dosimetry is that alpha particles induce less luminescence signal, with respect to the absorbed energy, being usually only about 5–20 % compared to beta and gamma radiation (AITKEN 1998).

With regard to dating, the environmental dose rate comprises internal, external and cosmic dose rate components. Internal dose rate originates from radioactive elements that may be present within the lattice of the luminescent mineral. Most important is the contribution from ^{40}K when using alkali feldspars as natural dosimeters. In quartz, the contribution of inter-

nal dose rate to the total dose rate is usually considered to be negligible. External dose rate comprises all radiation acting on the luminescent grain from the surrounding sediment, and is derived from ^{40}K , abundant in some feldspars, micas and clay minerals, and the isotopes of the $^{238}\text{U}/^{235}\text{U}$ and ^{232}Th decay chains. Uranium and Thorium are found at relatively high concentrations in zircon and some exotic minerals, but are ubiquitously present in low concentrations in a variety of common minerals such as carbonates (uranium) and clay minerals. A minor contribution to the total dose rate comes from the presence of ^{87}Rb (typically < 1 %). The contribution from other radioactive elements is so small that it is usually neglected. For calculating external dose rate the moisture content of the sediments is of great importance, as the attenuation of ionising radiation is much greater if the pores in the sediment are filled with water rather than air. Cosmic radiation consists of a variety of different particles such as neutrons, muons, electrons and photons that constantly penetrate the Earth's surface. Due to the nature of the Earth's magnetic field and absorption by the atmosphere, the intensity of cosmic radiation increases pole-wards and with altitude; overburden by both sediment and water (e.g., in lake or marine environments) will shield the sample and reduce the strength of cosmic radiation.

3 Luminescent minerals and their properties

Although a variety of minerals show the phenomenon of luminescence, the application of luminescence for dating purposes is, so far, mainly limited to quartz and feldspar. This is due to their abundance in sediments at most geological settings, as well as their ability to fulfil the requirements of sensitivity to radiation dose and behavioural characteristics. They are also sufficiently resistant to weathering compared to, for example, carbonates, which are also known to carry a luminescence signal (e.g., WIESER et al. 1993; CARMICHAEL et al. 1994). Furthermore, the luminescence signal in both feldspar and quartz is suffi-

ciently bleachable by daylight, is stable over long periods of time, and signal growth can be readily described using mathematical functions for the physical behavior. The dating potential of other minerals such as zircon (TEMPLER & SMITH 1988; SMITH et al. 1991; VAN ES et al. 2000) and halite (BAILEY et al. 2000) was initially tested but little systematic work has been carried out so far to investigate the potential of these minerals in more detail. The following briefly describes the key properties of quartz and feldspar and discusses their advantages and disadvantages for dating.

3.1 Quartz

Quartz is presently the mineral favoured for dating by many luminescence specialists. Beside its common occurrence in sediments, resistance to weathering and relatively well investigated luminescence properties, this is in particular due to the fact that it is not effected by the phenomenon of anomalous fading, as is at least some feldspar (see section 6.3). Due to these advantages, most research during the past decade has concentrated on developing quartz luminescence dating with special attention paid to the development of procedures to determine the palaeodose (e.g., WINTLE & MURRAY 2006). However, there are a few circumstances when quartz may not be the mineral of choice and it appears that many problems related to quartz have only more recently been identified. First of all, there are some geological settings, although relatively rare, where quartz does not occur due to the alkaline composition of the host rock. In other areas, luminescence from feldspar inclusions within the quartz may complicate the detection of the luminescence originating from the quartz itself. Although procedures have been suggested to deal with feldspar inclusions (WALLINGA et al. 2002) these are not straightforward. It has also been shown that quartz of volcanic origin may display anomalous fading (BONDE et al. 2001; TSUKAMOTO et al. 2007) and this problem needs to be addressed in future research. A further problem, possibly related to a young sedimentary history of the grain, is

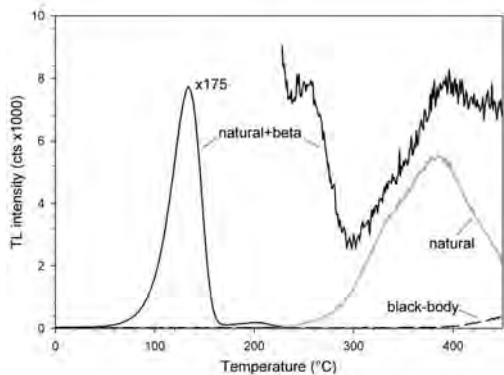


Fig. 4: Natural and artificial TL glow curve and black body radiation of a quartz sample from Nigeria (GUMNIOR & PREUSSER 2007).

Abb. 4: Natürliche und künstlich induzierte TL-Glühkurve und Schwarzkörperstrahlung einer Quarz-Probe aus Nigeria (GUMNIOR & PREUSSER 2007).

that quartz from some areas, shows rather low, if any, luminescence (e.g., LUKAS et al. 2007) and changes in sensitivity that are not able to be corrected for (PREUSSER et al. 2006). Thermal transfer of charge from light-insensitive to light-sensitive traps, that may cause palaeodose overestimation, has also been reported in this context (RHODES & BAILEY 1997; RHODES 2000). A considerable disadvantage of quartz is that the saturation dose, when all traps are captured by electrons, and, as a consequence, its potential dating range, is usually up to one order of magnitude lower than that of feldspar.

3.2 Feldspars

Potassium-rich feldspars are commonly used in luminescence dating studies. The disadvantages of using Na-rich feldspars for dating are discussed in KRBETSCHKEK et al. (1997) and only a few studies have focussed on this mineral (e.g., KRAUSE et al. 1997). The major advantages of feldspar are that it typically has bright luminescence signals and a higher saturation dose, which allows the potential to date much older deposits than with quartz. Another advantage is that a substantial proportion of the dose rate comes from ^{40}K within the feldspar,

which lowers the uncertainty from external dose rate, in particular due to variations in past sediment moisture. A disadvantage that has often been attributed to feldspar is that its optical signal is less light sensitive compared to quartz (GODFREY-SMITH et al. 1988; WALLINGA 2002a), although this does not appear to be supported by other experimental evidence which shows a similar resetting behaviour for both minerals (PREUSSER 1999a; KLASSEN et al. 2006).

The major problem in feldspar dating is the loss of part of the signal with time, a phenomenon referred to as anomalous fading (WINTLE 1973), which affects feldspars of at least some geological origins and causes an underestimation of the luminescence age if this is not corrected for. This will be discussed in more detail below (section 6.3).

4 Luminescence methods

Luminescence dating comprises a range of different but related phenomena. The fact that a strict nomenclature is not always adhered to and that some techniques are not often used may cause some confusion to the non-specialist. The following nomenclature refers to the most-widely used terminology and the techniques used in luminescence dating are described (Table 1).

4.1 Thermoluminescence (TL)

The term thermoluminescence (TL) describes the light emitted by a mineral, other than incandescence or black body radiation, when heated. The light originates from captured electrons being freed due to heat stimulation and subsequent recombination. Thus, it is also (and more correctly) referred to as thermally-stimulated luminescence (TSL) although this term is not often used. In addition to sensitivity to heat, a number of the electrons are also sensitive to optical activation, and this has also been utilised in the dating of sediments by TL. In practice, the sample is typically heated from ambient temperature to 450°C while the emitted photons are detected by a photomulti-

Table 1: Summary of the different methods used in luminescence dating.

Tab. 1: Zusammenfassung der verschiedenen Methoden der Lumineszenzdatierung.

Abbrev.	Method	Main application	Primary Reference
TL	Thermoluminescence	Dating heated materials	AITKEN et al. (1964)
ITL	Isothermal TL	Experimental (quartz)	JAIN et al. (2005)
OSL	Optically stimulated lum.	Dating sediments (quartz)	HUNTLEY et al. (1985)
IRSL	Infrared stimulated lum.	Dating sediments (feldspar)	HÜTT et al. (1988)
IR-RF	Infrared radiofluorescence	Dating sediments (feldspar)	TRAUTMANN et al. (1999a)
LM-OSL	Linearly modulated OSL	Analytical tool (quartz)	BULUR (1996)
HR-OSL	Spatially resolved lum.	Dating rock surfaces	GREILICH et al. (2002)
TT-OSL	Thermally transferred OSL	Experimental (quartz)	WANG et al. (2006 a)

plier tube and subsequently counted. The first measurement of TL empties the electron traps after which a second heated measurement is undertaken to record the unwanted light signal due to black body radiation or incandescence. This starts to grow around 400°C when using blue-violet detection filters, and the sum of this is subtracted from the first signal (Fig. 4). The different TL peaks of the glow curve represent different trap populations, with electrons from “shallow” traps recombining at lower stimulation temperatures than those from “deep” traps at higher temperatures, and a comparison of naturally and artificially induced TL reveals information about the stability of different signal components. This characteristic is utilised to identify the appropriate thermal pre-treatment for reliable measurement, and to investigate the extent to which a sample was zeroed by the natural heating/bleaching process.

TL was the only method used in retrospective dosimetry and luminescence dating until the mid-1980s, and was originally developed for the dating of ceramics (AITKEN et al. 1964), although it was also used later to date volcanic rocks (e.g., HWANG 1970; MAY 1979; RAYNAL et al. 1982), heated artefacts and sediments (HUXTABLE et al. 1978) as well as aeolian deposits (e.g., WINTLE & HUNTLEY 1979, 1980; WINTLE 1981; SINGHVI et al. 1982). However, of the applications mentioned above it is only the TL dating of ceramics and heated artefacts that remains in regular use today, while the dating

of sediments using TL was largely abandoned in the mid-1990s as it presents a significant disadvantage compared to optical dating. This arises because part of the thermally stimulated luminescence signal is not sensitive to natural daylight, on which sediments rely on for zeroing or bleaching. Thermal stimulation releases trapped charges associated with both light-sensitive and light-insensitive components and so can lead to potentially severe age overestimates. Under optimum experimental settings, i.e. direct sunlight exposure of min-

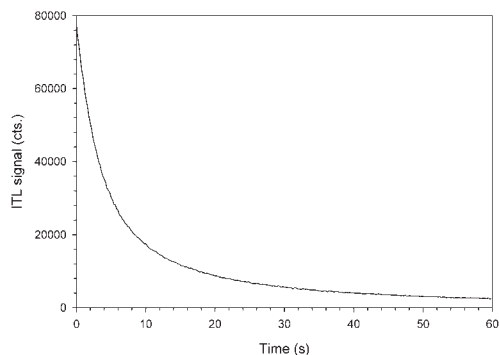


Fig. 5: Natural isothermal decay curve of a quartz sample from Nigeria (GUMNIOR & PREUSSER 2007), preheated at 230°C for 10 s and measured at 325°C.

Abb. 5: Natürliches isothermales Zerfallssignal einer Quarz Probe aus Nigeria (GUMNIOR & PREUSSER 2007), die bei 230°C für 10 s vorgeheizt und anschliessend bei 325°C gemessen wurde.

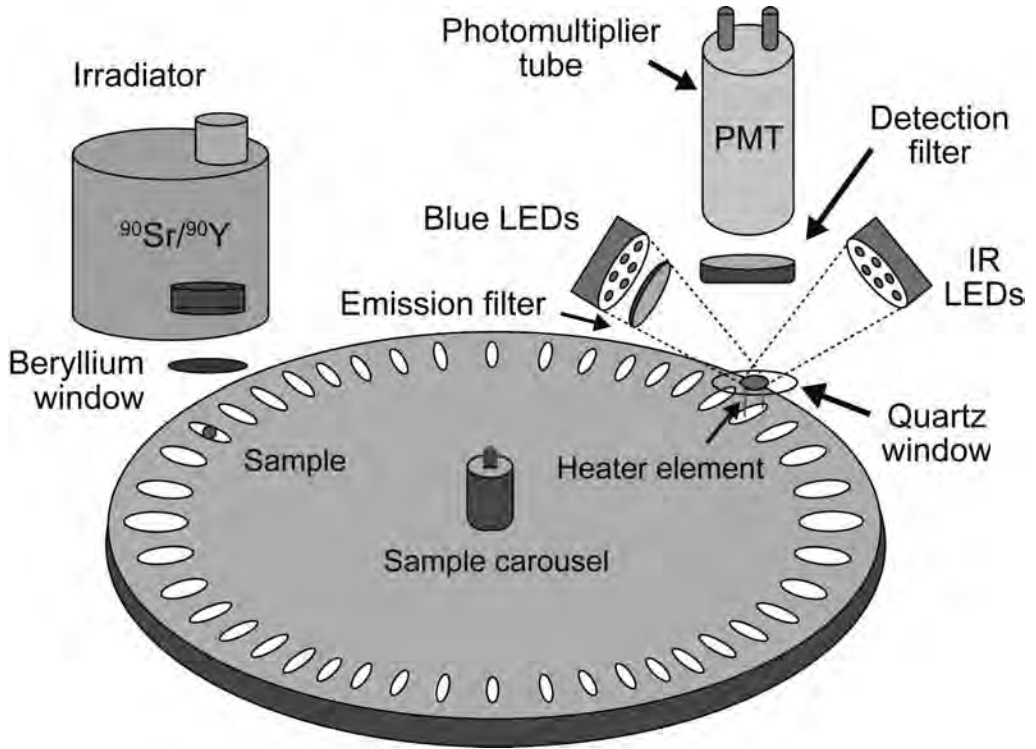


Fig. 6: Sketch showing the basic features of a TL/OSL reader (redrawn after Risø National Laboratory).

Abb. 6: Die Skizze zeigt den grundlegenden Aufbau eines TL/OSL Gerätes (umgezeichnet nach Risø National Laboratory).

eral grains, it will take several hours for the TL signal to be reduced to a small residual signal while the optical signal is almost fully reset within a few minutes (GODFREY-SMITH et al. 1988).

A modification of the classical TL approach is the isothermal TL (ITL) method, in which TL is recorded while the sample is held at a constant or isothermal temperature (JAIN et al. 2005; HUOT et al. 2006) (Fig. 5). The advantage of this method is that a specific trap population is emptied while deeper traps are not stimulated. In contrast to TL it is thus possible to focus on a trap population with certain physical properties. A first test study indicated that the potential of the method lies in the prospect of increasing the age range towards older samples (JAIN et al. 2005; CHOI et al. 2006a), although, while some tests on quartz

showed quite promising results, other test studies identified several problems and age overestimates in comparison to optical dating (BUYLAERT et al. 2006). Nevertheless, ITL appears to be an interesting approach with some potential, especially for older sedimentary samples and heated objects.

4.2 Optical stimulation

The most important breakthrough in recent years in luminescence dating has been the use of optical stimulation (optical dating). This area of luminescence dating mainly comprises stimulation by visible light as introduced by HUNTLEY et al. (1985), and by infrared (IR) as first described by HÜTT et al. (1988), may be collectively referred to as photoluminescence or photon-stimulated luminescence (PSL). The

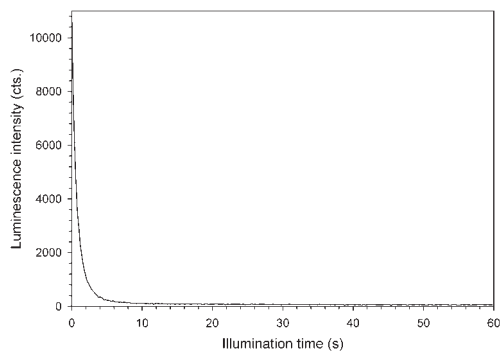


Fig. 7: OSL decay curve of a quartz sample from Nigeria (GUMNIOR & PREUSSER 2007) resulting from blue diode stimulation. Sample preheated at 230°C for 10 s and measured at 125°C.

Abb. 7: OSL-Zerfallskurve einer Quarz-Probe aus Nigeria (GUMNIOR & PREUSSER 2007), stimuliert mit blauen Dioden. Die Probe wurde bei 230° vorgeheizt und bei 125°C gemessen.

advantage of any optical method is that only the light-sensitive part of the luminescence signal is stimulated, which for sedimentary deposits considerably minimises the problem of incomplete bleaching of the luminescence signal prior to deposition. Furthermore, more reliable and precise methods for determining the palaeodose have been developed since the mid-1990s and have revolutionised luminescence dating. These methods will be described below. Regardless of what kind of photon-stimulation is used, optical filters are placed between the sample and the light-sensitive surface of the photomultiplier tube to minimise crosstalk between light from the stimulation source and light emitted from the sample (Fig. 6). When optical excitation begins the latent luminescence in the mineral will start to decay, resulting in a so-called decay curve recorded by the photomultiplier (Fig. 7).

4.2.1 Optically stimulated luminescence (OSL)

The term optically stimulated luminescence (OSL) is used here only with reference to stimulation by visible light, although in some

literature OSL is used as an umbrella term for all light stimulation including IR wavelengths. Stimulation by visible light is conducted by exposing the sample to a laser beam, the light of a filtered halogen lamp or to high-power light emitting diodes (LEDs), the latter being the latest and most versatile development, and hence used in the majority of currently manufactured luminescence readers (Fig. 6). The width of the excitation waveband differs for different stimulation sources but all lie in the green to blue part of the spectrum, i.e. between 420-550 nm. Both quartz and feldspar respond to stimulation within this waveband although for dating applications it is principally used for measurements of quartz. Light of longer wavelengths becomes increasingly inefficient at stimulating OSL in quartz, whereas wavelengths in the near infrared excite luminescence in feldspars due to one or more excitation resonances. Light emissions for OSL from quartz are detected in the ultraviolet range.

4.2.2 Infrared stimulated luminescence (IRSL)

In contrast to visible light, low-energy photons from the IR band will stimulate luminescence in feldspars but not in quartz (HÜTT & JAEK 1989). The most common term for this phenomenon is infrared stimulated luminescence (IRSL) although some researchers refer to it as IR-OSL or IR-PSL. Stimulation uses either an IR laser or IR diodes, the latter now being the most commonly used. The fact that quartz does not respond to IR stimulation is regularly used to check quartz-separates for feldspar contamination. One advantage of feldspar stimulation with IR is that it allows the detection of different recombination processes in feldspars with different physical properties that are reflected by distinct light emission in the UV, blue, yellow and red emission bands (KRBETSCHKEK et al. 1997). The potential of comparing different emission bands, in particular in the context of detecting and overcoming fading of feldspar IRSL (see below), is not yet fully utilised as only a few studies have so far compared the

various sample emissions (e.g., KRAUSE et al. 1997; PREUSSER 1999b; PREUSSER et al. 2003). Recent research has focused on IRSL red emissions and it is expected that the apparent stability of the IRSL signal in this part of the spectrum may overcome problems related to signal stability, i.e. regarding fading and resulting age underestimation (FATTAHI & STOKES 2003 a, b; STOKES & FATTAHI 2003). However, the detection of red IRSL from feldspars using conventional luminescence readers presents some problems, and the use of more sophisticated instrumentation has shed doubt on its stability (BARIL & HUNTLEY, 2003). Spectral measurements in this study identified a very low stability of the red (about 700 nm) IRSL emission of feldspars.

4.2.3 Infrared radiofluorescence (IR-RF)

Infrared radiofluorescence (IR-RF) dating was first introduced by TRAUTMANN et al. (1999a) and was referred to as radioluminescence (RL) dating. Because the underlying physical process of this type of luminescence is quite different from that associated with TL, OSL and IRSL dating, the change in nomenclature was useful to physically distinguish this more recently developed dating method (ERFURT & KRBETSCHKEK 2003b). In IR-RF dating the palaeodose is determined by a (IR emitting) fluorescence process, i.e. a prompt radiative charge transition. An emission at 865 nm during excitation by ionising radiation is typical for potassium feldspars (orthoclase, microcline) only. The physical basis of this phenomenon and methodological aspects of its application in dating were investigated to develop a reliable new technique for age determination of sediments (SCHILLES et al. 1999; TRAUTMANN et al. 1999a, b; KRBETSCHKEK et al. 2000; SCHILLES & HABERMANN 2000; TRAUTMANN et al. 2000; TRAUTMANN 2000; ERFURT 2003, ERFURT & KRBETSCHKEK 2003a). The IR emission can be interpreted as the transition of electrons from the conduction band into a particular kind of electron trap. During ionising irradiation a number of electrons reach the conduction

band from which they promptly recombine with luminescence centres and emit visible (VIS) fluorescence or transfer to that electron trap by emission of near-IR fluorescence (cf., process c in Fig. 2.1). The trap is probably the same as stimulated by IRSL (process a in Fig. 2.2). Compared to OSL and IRSL, the IR-RF signal bleaches slightly more slowly under light exposure although much faster than TL. As the RF-dose characteristics in quartz are far more complex and variable than in K-rich feldspars (KRBETSCHKEK & TRAUTMANN 2000), the majority of studies have so far concentrated on feldspars.

Palaeodose determination based on the phenomenon of IR-RF of potassium feldspars has different advantages compared to TL, OSL and IRSL. Firstly it is a direct measure of the electron density of a (well defined) trap, which does not rely on the conventional luminescence centres (which, as shown, can fade). This also explains why IR-RF dose response curves decrease with increasing dose (Fig. 8) – if the electron trap is empty (zero or low dose) the number of transitions into the traps are high (large signal), if the trap is filling up, the signal decreases due to the decreasing number of free traps. Another advantage of this method is that luminescence stimulation and dose accumulation are applied at the same time. This allows continuous measurement and the recording of a very high number of dose points (Fig. 8) and follows a strict stretched single-exponential decay curve for physically defined reasons. Furthermore, single aliquot regeneration procedures (see Section 5.3) are used for dose determination (ERFURT & KRBETSCHKEK 2003b) and also contribute to high precision in palaeodose estimates using the IR-RF method.

The measurement of an IR luminescence emission itself however, together with its low dynamic range and the special procedure of dose application and measurement is not a simple task and requires special instrumentation (ERFURT et al. 2003), which is not commercially available, and explains why this method is broadly accepted (BØTTER-JENSEN et al. 2003a; GEYH 2005) but only carried out at

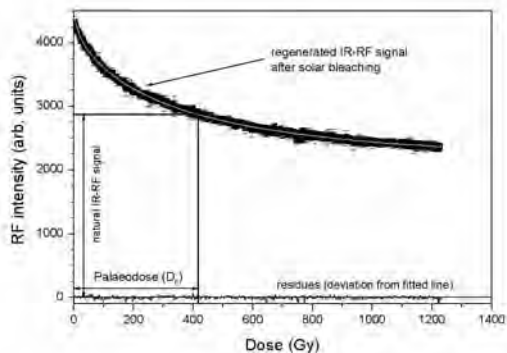


Fig. 8: IR-RF single aliquot regeneration dose response curve of a fluvial sample from central Germany (open cast mine Delitzsch) used to determine the palaeodose (IRSAR-protocol by ERFURT & KRBECSHEK 2003b). Note the high number of dose points and how the bottom curve (residual: deviations from the fitted line) shows good agreement to the fitted stretched-single-exponential decay curve. The low dynamic range of the IR-RF signal can also be seen.

Fig. 8: IR-RF – Dosisfunktion eines Einzelpräparates einer fluviatilen Probe aus Mitteldeutschland (Tagebau Delitzsch), verwendet zur Bestimmung der Paläodosis (IRSAR-Protokoll nach ERFURT & KRBECSHEK 2003b). Man beachte die große Zahl an Dosispunkten und die durch die untere Linie (Residuen: Abweichung von der angepassten Kurve) gezeigte gute Übereinstimmung mit der Anpassung an eine gestreckt einfach-exponentiell absinkende Kurve. Der geringe dynamische Umfang des IR-RF-Signals ist ebenfalls sichtbar.

one luminescence dating laboratory (Freiberg, Germany). Its dating-range spans 20 ka to about 350 ka in most cases and as far back as 500 ka with low dose rates. The IR-RF signal stability has been proven through different physical experiments, and also shows a good agreement with independent age control up to 250-300 ka (ERFURT et al. 2003; DEGERING & KRBECSHEK 2007). It has so far been applied to sediment dating where the signal is optically reset but can also be used to date thermally reset events. DEGERING & KRBECSHEK (2007) and KRBECSHEK et al. (2008) have published data of limnic, fluvial and other water-lain sediments back to about 350 ka. Developments

in instrumentation including the application of single grain dating, for which the first successful experiments have been made, will improve this method with regard to its precision and the potential to extend the age range.

4.2.4 Linearly modulated OSL (LM-OSL)

In contrast to “conventional” optical stimulation where the energy of the stimulation source is kept constant (continuous wave, CW-OSL), in linearly modulated OSL (LM-OSL) the stimulation power from the LEDs is ramped slowly (typically over 10^3 to 10^4 s) in a linear fashion from zero to some preset value (BULUR 1996; BULUR et al. 2000). The result of this slow increase of stimulation intensity is that in quartz the trapped charge is released first from shallow, and subsequently to deeper traps. This procedure results in overlapping peaks of luminescence emission related to different traps (in some sense similar to TL peaks). Due to the overlap of different components it is necessary to apply mathematical deconvolution to the LM-OSL data to discriminate between different components (cf., CHOI et al. 2006b). The result of this fitting gives information on the presence of different OSL components in a particular sample (Fig. 9), which are grouped according to when they occur during LM-OSL. Fast, medium and slow components have been identified, with the latter consisting of up to five individual sub-components (JAIN et al. 2003; SINGARAYER & BAILEY 2003), and all of which have different light sensitivities (BAILEY et al. 1997). The first of these is the fastest emission and it follows that this is the easiest to optically bleach in the natural sedimentary environment. Whether or not these different components originate from the same or different optical charge traps is still not fully understood. It is important to note that not all components are present in quartz of different geological origin (Fig. 9) and that the relation between sample properties and different trap populations remains vague. Furthermore, due to uncertainties related to mathematical deconvolution, LM-OSL has rarely been used for direct dating purposes. However, LM-OSL

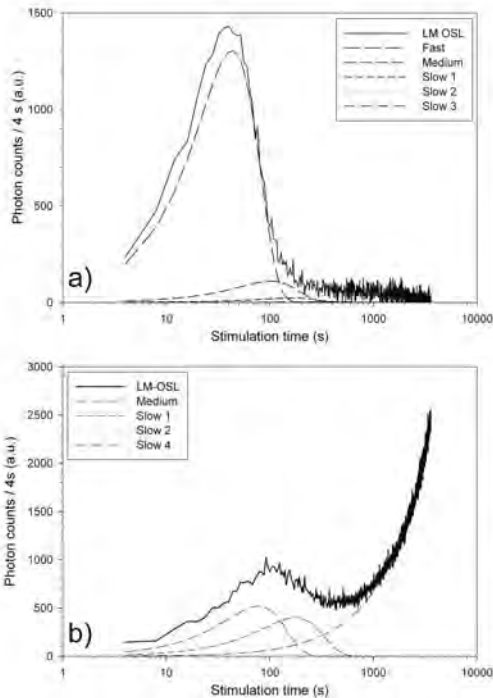


Fig. 9: Deconvoluted LM-OSL curve for (a) well-behaved Australian quartz and (b) quartz sample from Bavaria. The fast component that dominates the signal in sample (a) is not present in sample (b), which is dominated by an unstable medium component.

Abb. 9: Entfaltete LM-OSL Kurve für (a) einen australischen Quarz mit guten und (b) einen Quarz aus Bayern mit schlechten Lumineszenzeigenschaften. Die schnelle Komponente die das Signal in Probe (a) dominiert ist in Probe (b) nicht vorhanden; diese wird durch eine instabile mittlere Komponente dominiert.

is an extremely useful tool for characterising the properties of individual samples and can be used to detect the potential for poor behaviour in quartz, in an early phase of the dating process, although most important is the use of the fast component in circumventing problems of partial bleaching.

4.2.5 Spatially-resolved luminescence (HR-OSL)

A novel approach in luminescence is high resolution spatially-resolved luminescence (HR-

OSL) dating of stone surfaces (GREILICH 2004; GREILICH et al. 2002, 2005; GREILICH & WAGNER 2006). Similar to sediment dating, the aim is to determine the time when a stone surface was last exposed to daylight. The upper and lower surface of a granitic block, for example, will be shielded from daylight after erecting the wall of a historic monument. Dating the last daylight exposure of mineral grains within construction material hence reflects the age of a building. Besides archaeology, the approach also has great potential for geological applications, as sediments often contain pebbles and boulders. Optical dating of stone surfaces requires new approaches to sample preparation, palaeodose measurement and dose rate determination. In contrast to conventional luminescence dating, stone surfaces are kept intact to ensure only the grains exposed to light during the construction process are analysed. Because samples remain intact, this method has the great advantage that all information on the micro-dosimetric field is preserved; a detail which is destroyed during the traditional sample preparation. Aliquots are prepared by retrieving small drill cores (diameter ~10 mm) from a stone surface, cutting a thin slice (~2 mm thick) from the surface end of the core and mounting this slice in a specially designed metal holder after which, the luminescence measurements basically follow the single-aliquot regenerative-dose (SAR) protocol (see Section 5.2). However, the conventional way of measuring bulk luminescence signals using a photomultiplier tube is not appropriate for a stone surface with varying minerals and grain-size. In contrast, detection of the luminescence signal is achieved by means of a charged-coupled device (CCD) chip, which permits spatial resolution down to $25 \mu\text{m} \times 25 \mu\text{m}$. This produces a large amount of data (several thousand data points per individual measurement) and data management and analysis require a special software program (GREILICH et al. 2006). This software allows production of palaeodose values for each of the many picture elements, filtering of data and statistical analyses that can be spatially resolved for each pixel and then displayed in histograms.

The same spatial resolution for palaeodose calculation is needed for dose rate determination. This is achieved by using an energy dispersive X-ray analysis detector mounted on a scanning electron microscope (SEM-EDX) for the determination of the potassium content of feldspar grains. A special variant of fission-track analysis using tracks induced by fast neutrons is used to determine dose rates from uranium and thorium (WAGNER et al. 2005). From the spatially-resolved palaeodose and dose-rate data, luminescence ages are calculated for each of the analysed grains from a drill-core surface-slice. The last daylight exposure of a stone surface is calculated by combining data of several individually drilled samples.

4.2.6 Thermally transferred OSL

A new approach in the optical dating of quartz is the thermally transferred OSL (TT-OSL) technique (WANG et al. 2006 a). This method makes use of the two components of the thermally transferred OSL signal, which can be divided into the recuperated OSL signal and the basic transferred signal (AITKEN 1998). The recuperated OSL signal is generated by electrons being transferred via optical stimulation from the OSL traps into thermally unstable traps (refuge traps) (WANG et al. 2006a). After the OSL traps are emptied (through light exposure), the transfer from the refuge traps into the OSL traps is generated by thermal treatment. The subsequent light exposure results in a recuperated OSL signal, which is less sensitive compared to the OSL signal (WANG et al. 2006b), and is believed to have the potential to date samples beyond the level of saturation of the fast component of the OSL signal used in a common SAR-procedure (WANG et al. 2007). In contrast, the basic transfer signal derives from electrons trapped in light-insensitive traps and is released into light-sensitive traps by preheating and therefore inapplicable for dating. WANG et al. (2007) presented a SAR procedure using the recuperated OSL signal for palaeodose determination and showed agreement with OSL results.

5 Luminescence procedures

Several values are required to calculate a luminescence age and are either taken from empirical observations or are derived from mathematical expressions. These values are either associated with the determination of palaeodose and the reliability of this measurement or are used for the calculation of a dose rate. The following section gives a brief overview of how these values are determined, how they are taken into account in the age model and which values give information on the reliability of the applied dating procedures.

5.1 Sampling

A first and important point in any dating process is correct sampling. It is mandatory to collect detailed information on the geological context (including photographs, detailed field notes and so forth), particularly concerning the environment of sedimentary deposition and sediment overburden (sample depth below surface, indication for post-sedimentary disturbance, hiatuses, sedimentation cycles). Present and past hydrological conditions (sediment moisture) of the sample need to be assessed as far as possible. To ensure a uniform radiation field, the sample should be taken from a homogenous layer with a thickness of at least 50 cm. If no such layer is available, on-site measurement of gamma radiation (using a portable gamma spectrometer or a gamma counter) should be utilised to minimise the impact of inhomogeneity on dose rate calculation. Samples used for the determination of palaeodose have to be taken without exposing the material to daylight, and is usually achieved by forcing opaque metal or plastic tubes into a freshly cleaned exposure. Material from both ends of the tubes, which is likely to have had some daylight exposure, is discarded for palaeodose measurements but can be used for dose rate determination. Some laboratories require additional material for dose rate determination, particularly when high-resolution gamma spectrometry measurements are performed, but this

material need not be protected from daylight exposure. It is strongly recommended that researchers having little experience in sampling for luminescence dating should contact an expert prior to sample collection to get information on specific requirements.

5.2 Sample preparation

Prior to any luminescence measurements, a preparation procedure is carried out to retrieve quartz and K-feldspar separates. All preparation work for the determination of the palaeodose is carried out under subdued laboratory illumination to avoid any loss of luminescence. Most luminescence facilities have either red or orange low intensity lights installed. The first steps include treatment by HCl and H₂O₂ to remove carbonates and organic material, respectively, always followed by washing with deionised water. A particular grain size is then isolated by

either sieving (sand-size grains) or settling (silt). For sand sized grains, quartz and K-feldspar separates are isolated using heavy liquids with densities of 2.58 g cm⁻³, 2.62 g cm⁻³ and 2.70 g cm⁻³ (MEJDAHL 1985). Treating the quartz fraction with concentrated HF acid etches the surface of the quartz by about 10 µm to minimise the alpha-luminescence and also to minimise feldspar minerals by dissolution (plagioclase has a similar density as quartz), followed by treatment with HCl to remove any fluorides that may have formed during etching and dissolution. Etching with 10 % HF is often applied to remove the outer part of K-feldspar grains and etching with either HF (at various concentrations) or H₂SiF₆ is used to isolate fine-grain quartz separates (cf., MAUZ & LANG 2004). Alternatively, one can also use polymineral fine-grain samples for palaeodose determination. In such samples the IRSL signal will usually be dominated by feldspar emissions as quartz is not sensitive to

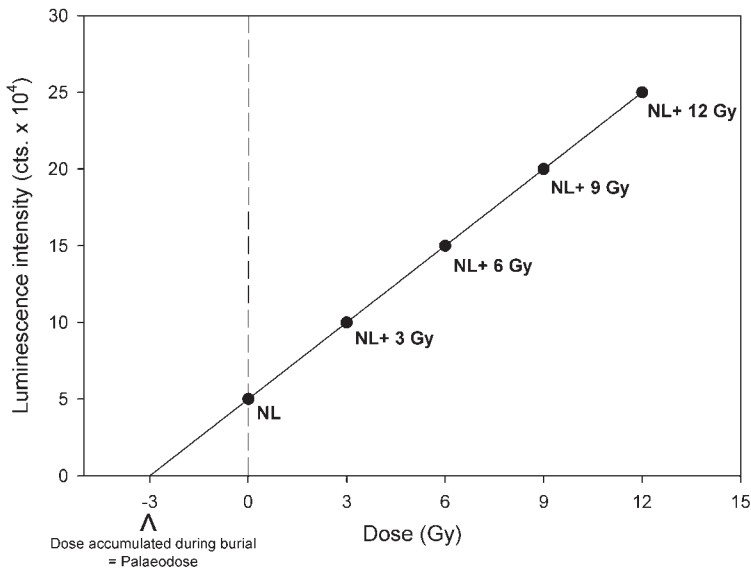


Fig. 10: Additive-dose response curve. Different laboratory doses are added to the natural signal (NL) to characterise the increase of luminescence intensity with dose. The best fitting function is then extrapolated to calculate the dose absorbed during burial (palaeodose).

Abb. 10: Dosisfunktion nach der additiven Methode. Verschiedene Dosiswerte werden im Labor zum natürlichen Signal (NL) hinzugefügt um das Anwachsen der Lumineszenz mit zunehmender Dosis zu charakterisieren. Die beste Kurvenanpassung an die Messwerte wird dann extrapoliert, um die Dosis zu bestimmen, die während der Lagerungszeit absorbiert wurde (Paläodosis).

IR stimulation. In all approaches the grains are mounted on ~10 mm diameter stainless steel or aluminium discs. Fine-grained silts are left to slowly settle (usually through a small vial of acetone) onto the discs; a silicon spray is used to mount sand-sized grains. With sand-sized grains, circular templates are commonly used to determine the quantity of mounted grains.

5.3 Determination of the palaeodose

Measurements are carried out on specialised luminescence equipment and further information regarding technical details about the most commonly used device manufactured by the Risø National Laboratory is found in BØTTER-JENSEN et al. (2003b). The machine consists of a measurement chamber equipped with a sample changer, a sample heating device, stimulation sources (laser or LEDs), a detection unit (photomultiplier tube and optical filters) and, usually in all laboratories, an integrated irradiation source ($^{90}\text{Sr}/^{90}\text{Y}$ beta source) (Fig. 6). In multiple aliquot dating (see below) irradiation is often carried out using external radioactive sources.

In principle there are two different approaches to determine the palaeodose: additive dose and regenerative dose. For both approaches, a so-called dose response curve is calculated reflecting the increase of latent luminescence in the mineral grains with increasing dose. In the additive dose approach an artificial laboratory-induced signal is added to the natural signal (Fig. 10), while in the regenerative dose method the latent luminescence signal is first removed and the sample is then irradiated and a laboratory signal regenerated (Fig. 11). The advantage of the regenerative method is that no extrapolation of the dose response curve is necessary. This fitting of the curve can cause significant uncertainties in the additive dose approach, especially where data are scattered or for older samples where the growth curve reaches saturation. Small fitting errors can produce large errors in palaeodose determination. The disadvantage of the regenerative method is that the process of zeroing the signal in the grains has been proven to often change the luminescence

properties of a sample. If such changes in luminescence sensitivity are not corrected for, the palaeodose will be systematically under- or overestimated (WINTLE 1997).

Regardless of which approach is being used, the measurement of palaeodose comprises three different steps: irradiation, preheating and measurement of luminescence. Irradiation is carried out to induce a latent luminescence signal in the grains and is performed by using either beta sources (external or integrated in the luminescence reader) or external gamma sources. During artificial irradiation all traps, including those unstable over longer time periods, capture electrons. To allow comparison with the palaeodose absorbed over the burial time, the unstable component of the luminescence signal has to be removed, and is achieved by heating the sample (between 200°C and 300°C for usually 10 s, or several hours at temperatures of about 150°C) causing eviction of the electrons caught at shallow traps. Once this preheating of the sample has been performed, the luminescence is measured by stimulating the sample with either heat or light.

5.3.1 Multiple aliquot techniques

Up to the late 1990s, most luminescence dating studies used multiple aliquot approaches. With this method, determination of the palaeodose is carried out using several (usually five) subsamples (aliquots) for the different irradiation doses. The average of these individual dose groups is then used for the construction of a dose response curve. The major disadvantage of a multiple aliquot approach is that each aliquot often behaves differently (due to e.g., variable sensitivity changes, incomplete and variable bleaching, etc.) giving rise to scatter in the data. For this reason, a relatively large uncertainty is often associated with mathematical fitting of dose response curves constructed by multiple aliquot approaches. Furthermore, it has been shown that samples are often affected by changes in luminescence sensitivity when using regenerated doses and this approach should not be used if this change cannot be

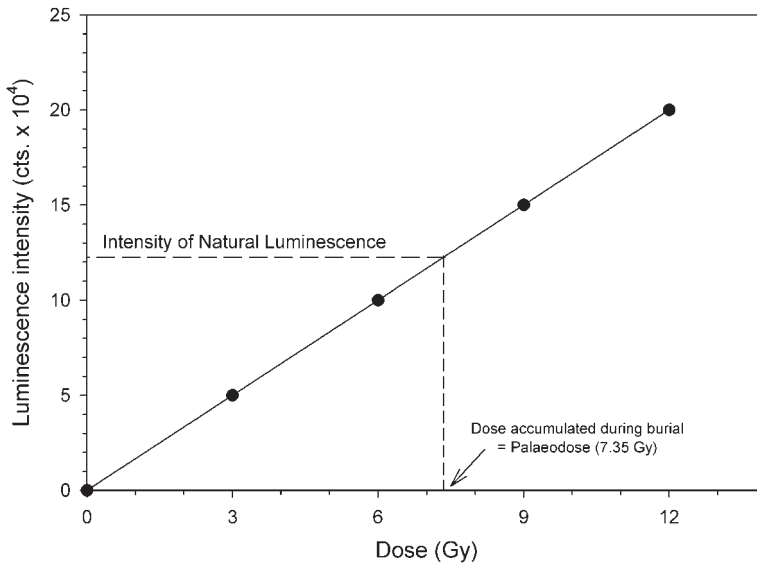


Fig. 11: Regenerative-dose response curve describing the increase of luminescence with dose. Once the natural luminescence signal has been erased by either light or heat exposure, different laboratory doses are administered to the aliquots, and the luminescence response to these used to construct a curve of best fit. The intensity of the natural luminescence signal (dotted line) is then projected onto this to determine the dose absorbed during burial (palaeodose) using interpolation.

Abb. 11: Dosisfunktion nach der regenerativen Methode. Mit verschiedenen Dosiswerten werden im Labor Einzelpräparate bestrahlt, deren Lumineszenzsignal zuvor durch Licht oder Wärme gelöscht wurde. Damit soll das Anwachsen des Lumineszenzsignals mit der Dosis bestimmt werden. Die Intensität des natürlichen Lumineszenzsignals (gestrichelte Linie) wird dann auf die beste Kurvenanpassung projiziert, um die Dosis durch Interpolation zu bestimmen, die während der Lagerungszeit akkumuliert wurde (Paläodosis).

corrected (DULLER 1994; WINTLE 1997). The major disadvantage with the additive dose approach as mentioned above is the uncertainty of extrapolation with particular regard to older samples.

5.3.2 Single aliquot techniques

When HUNTLEY et al. (1985) published their benchmark paper on optical dating, they already mentioned the possibility of using single aliquot approaches. The main aspect of any such approach is that all measurements are carried out on a single portion or aliquot of the same grains, which circumvents the problem of individual luminescence properties of grains from different aliquots. Several approaches were tested in the 1990s, most of which con-

tributed to the improvement of methodology but included methodological problems that threw doubt on the use of these approaches in routine dating applications (e.g., DULLER 1991; MEJDAHL & BØTTER-JENSEN 1994; ZANDER et al. 2000). A major breakthrough was made with the development of the single-aliquot regenerative-dose (SAR) technique that was mainly developed and further refined by MURRAY & WINTLE (2000, 2003). As this method is now used in most dating applications by the majority of laboratories world-wide, the principles and procedures will be described in some detail below. A more detailed review focussing on technical details has been presented by WINTLE & MURRAY (2006).

The basic idea behind the SAR technique is to utilise a regenerative dose approach and correct

for any sensitivity change that may occur in the course of the measurement procedure. To carry out this sensitivity correction, a so-called test dose, which remains constant during the whole protocol, is administered after measuring each regenerative (including natural and zero) dose (Fig. 12). The luminescence intensity resulting from each regenerative dose (L_x) is normalised by the luminescence intensity of the following test dose (T_x) and the ratio L_x/T_x , which represents the sensitivity-corrected luminescence signal, is used to construct a dose response curve (Fig. 13). For suitable curve fitting, construction of the latter requires at least three different regenerative dose points (plus a zero dose point). Flexibility in the SAR protocol permits an increase in the number of dose points if required, and can be used to improve the mathematical fitting of the dose response curve when a sample is approaching saturation.

The performance of each individual SAR cycle is verified by two internal tests: recuperation and recycling. The first test considers the signal from an aliquot given zero regenerative dose and then the same test dose as every other SAR cycle, for which L_0/T_0 should be close to 0.00. Values above zero indicate that an unwanted signal was induced by preheating the sample prior to measurement (recuperation), and MURRAY & WINTLE (2000) suggest that recuperation should not exceed 5 % of the natural sensitivity corrected signal intensity (L_N/T_N). If recuperation exceeds this value, it is necessary to modify the protocol by either changing the preheat procedure or by adding a so-called “hot-bleach” (illuminating the sample at elevated temperature) to avoid any such effect (MURRAY & WINTLE 2003). The second test (recycling ratio) confirms whether correction for sensitivity change was successful. This test is carried out by measuring the luminescence response to the same regenerative dose at the beginning, and then again at the end of the SAR cycle (Fig. 13). If the sensitivity correction was successful, the ratio of the two measurements will be unity. MURRAY & WINTLE (2000) suggest discarding any measurements where the

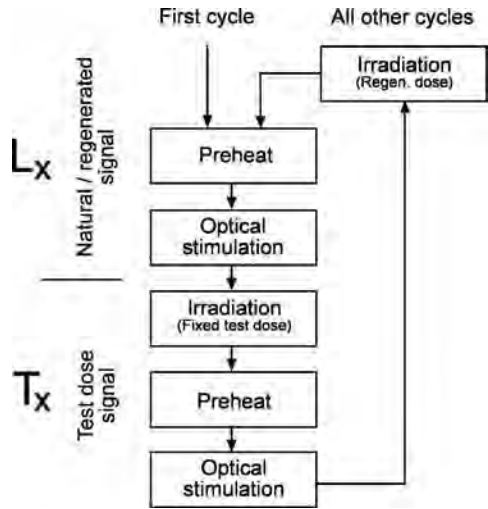


Fig. 12: Overview of the single-aliquot regenerative-dose (SAR) protocol (MURRAY & WINTLE 2000). L_x describes the luminescence signal of the natural sample (L_N) or different regenerative doses (e.g., $L_{10\text{ Gy}}$). T_x is the luminescence response to a constant test dose (e.g., 2 Gy) that is measured after the natural and each regenerative dose.

Abb. 12: Überblick über das Einzelproben-Protokoll mit regenerierter Dosis (SAR) (MURRAY & WINTLE 2000). L_x ist das Lumineszenzsignal der natürlichen Probe (L_N) bzw. verschiedener regenerierter Dosispunkte (z.B., $L_{10\text{ Gy}}$). T_x ist das Lumineszenzsignal, das aus der Bestrahlung mit einer konstanten Testdosis (z.B. 2 Gy) gewonnen wird. Dieses Signal wird nach der natürlichen Lumineszenz und nach jedem regenerierten Dosispunkt gemessen.

recycling ratio is $>10\%$ from unity (i.e., $0.90 < \text{recycling ratio} < 1.10$).

In addition to these quality controls performed during every SAR measurement, a series of further tests have been developed to ensure the reliability of the measurement procedure. If these tests do not provide acceptable results it is necessary to modify the measurement procedure. In this context it is important to recognise that, due to the nature of the signal and the variety of sediment sources, luminescence properties of minerals are different in different geological settings. Hence, it is mandatory to carry out a rigorous testing program for all dating projects (e.g., BLAIR et al. 2005; KLASSEN et al. 2006).

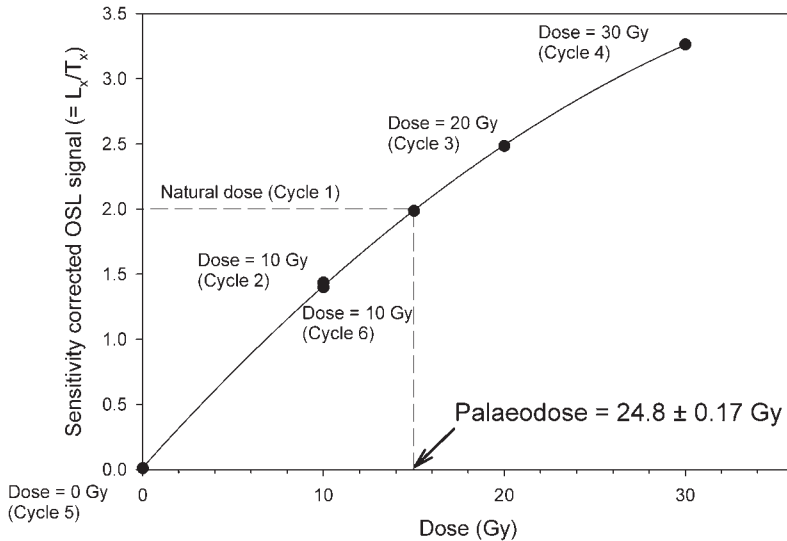


Fig. 13: A typical SAR-generated dose response curve. The ratio of L_x / T_x (e.g., L_{10} / T_{10}) is plotted versus applied regenerative dose. L_N / T_N is then projected onto this curve to determine the palaeodose through interpolation. The lowest regenerative dose is measured twice, at the beginning and end of each individual SAR cycle, to ensure that sensitivity changes have been sufficiently corrected for. The ratio of these two measurements is referred to as the *recycling ratio* and should be within 10 % of unity.

Abb. 13: Eine typische Dosisfunktion, die mit dem SAR-Protokoll gewonnen wurde. Der Quotient L_x / T_x (z. B., L_{10} / T_{10}) wird gegen die regenerierte Dosis aufgetragen. L_N / T_N wird dann auf diese Kurve projiziert um die Paläodosis durch Interpolation zu bestimmen. Das Signal der niedrigsten regenerierten Dosis wird zweimal, am Anfang und am Ende jedes einzelnen SAR-Zyklus, gemessen, um zu überprüfen, ob Sensitivitätsänderungen hinreichend korrigiert wurden. Das Verhältnis dieser wiederholten Messungen wird Wiederfindungsverhältnis (*recycling ratio*) genannt und sollte um weniger als 10% von Eins abweichen.

A basic requirement that proves the suitability of the SAR protocol is that a known dose that is administered in the laboratory can be retrieved by the measurement procedure (ROBERTS et al. 1999; WALLINGA et al. 2000). For testing this, a so-called dose recovery test is carried out during which the natural signal is first optically erased and then a known laboratory dose is given to the sample. This known dose is then treated as the natural dose in the SAR protocol and the ratio of measured/given dose should be unity if the procedure works correctly, and repeating this test for a sample gives information on the best achievable precision. This very much depends on the luminescence properties of the sample, especially luminescence intensity. Reported values range between relative standard deviations of a few percent, e.g. 4 % for Dutch

aeolian sands (VANDENBERGE et al. 2003), to between 10-15 % found for glacial deposits from Switzerland (PREUSSER et al. 2007).

A major problem that can, at least in some areas, interfere with correct determination of the palaeodose and is not directly detectable in standard SAR measurements is basic thermal transfer (RHODES & POWNALL 1994; RHODES & BAILEY 1997; RHODES 2000). The phenomenon is similar to the effect of recuperation (cf., AITKEN 1998) and has a major effect on the natural sample but not on subsequent regenerative dose measurements (RHODES 2000). A simple test to identify if thermal transfer affects a sample is carried out by erasing the latent luminescence and then measuring the apparent palaeodose (Fig. 14). Where the apparent palaeodose is significantly > 0 Gy this is due to a transfer of

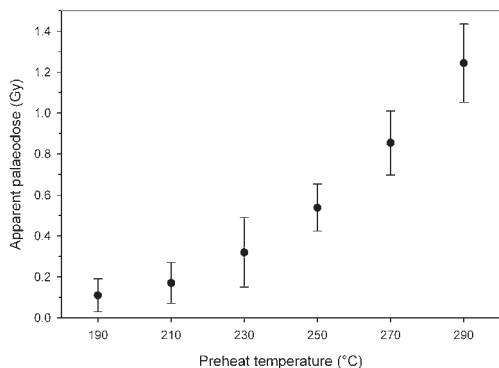


Fig. 14: Plot of palaeodose versus preheat temperature for a sample in which the OSL signal was zeroed by previous optical stimulation (thermal transfer test). The increase of apparent palaeodose results from an OSL signal that is induced by thermal transfer of electrons from light-insensitive to light-sensitive traps.

Abb. 14: Abhängigkeit der ermittelten Paläodosis von der Vorheiztemperatur für eine Probe, deren OSL-Signal durch vorherige optische Anregung auf Null gesetzt wurde (Test des thermischen Transfers). Das Anwachsen der scheinbaren Paläodosis resultiert aus einem OSL Signal, welches durch thermischen Transfer von Elektronen aus lichtinsensitiven in lichtsensitiven Fallen hervorgerufen wird.

charge from hard-to-bleach to easy-to-bleach traps. Most studies that have reported significant thermal transfer are related to quartz with a young sedimentary history, i.e. proglacial deposits (RHODES & POWNALL 1994; RHODES & BAILEY 1997; RHODES 2000; PREUSSER et al. 2006), although other studies have shown that thermal transfer may be negligible for some proglacial sediments (e.g., KLASSEN et al. 2006; PREUSSER et al. 2007). The test described above is a quick and effective way to exclude the presence of thermal transfer.

A further test exists to check whether the applied preheating procedure is sufficient to remove unstable signal components in the artificially induced signal (AITKEN 1985). For this, the palaeodose is determined for a series of different preheat temperatures (Fig. 15). If all the unstable charge is removed, the calculated palaeodose should not change with increasing

preheat temperature. However, it is possible that the phenomenon of thermal transfer is also present and in this case a compromise between a preheat procedure suitable to remove all unstable charge but without causing significant thermal transfer is necessary.

For feldspar samples, storage tests are carried out to monitor the short-term stability of the artificially-induced luminescence signal. This is carried out because feldspars can sometimes be affected by a phenomenon referred to as anomalous fading that describes a loss of signal with time (WINTLE 1973) (see section 6.3).

5.4 Determination of the dose rate

After obtaining the palaeodose, the next step in age determination for a sample requires assessment of the rate at which the radiation dose was delivered to the mineral grains. Similar to the palaeodose this assessment is made over the burial period, being the time since the sample last saw daylight during transport and deposition or since it was last exposed to heat. In most cases, the accumulated dose per unit time, the dose rate, is constant and the age t of a sample is simply calculated from the measured palaeodose D according to Equation 1. A minor component of the dose rate is produced by ionising cosmic radiation and can be estimated from geographic position and burial depth of the sampled material (PRESCOTT & HUTTON 1994) (Fig. 16). The larger part originates from the natural radionuclide ^{40}K and from the uranium and thorium decay series.

In principle, there are three ways to determine this contribution by either measuring the dose rate (a) directly in the sediment using dosimeters, (b) by measuring alpha, beta and gamma dose rates using radiation counting devices, or (c) by analysing the activity and/or concentration of the dose rate relevant nuclides followed by determination of the dose rate using well-established conversion factors. In practice, most laboratories use the last approach. Within the calculation some general assumptions are made for convenience (AITKEN 1985):

- The material is isotropic and homogenous

with respect to the distribution of the sources of ionising radiation.

- The investigated system is indefinitely expanded.
- All radionuclide concentrations are constant in time.

Infinite system dose rates are easily calculated from the specific activities of the radionuclides using conversion factors tabulated in the literature (cf., ADAMIEC & AITKEN 1998). Moisture corrections as described in AITKEN (1985) are necessary to consider the radiation attenuation by pore water. Inhomogeneity due to grain size, pore volume and moisture distributions, as well as of the mineral composition are removed in multi-grain samples by averaging over a large number of grains. An inhomogeneity remains between the radionuclide content of the investigated grain itself (causing the internal dose) and the averaged irradiation from all sources outside the grain (the external dose). This effect is dealt

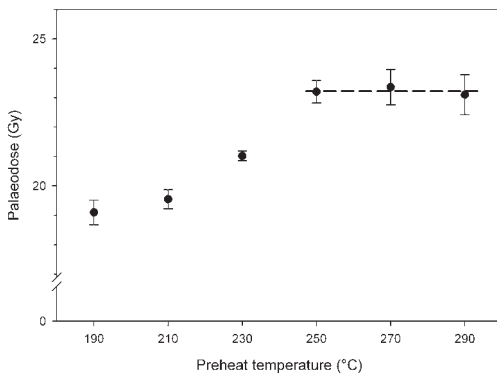


Fig. 15: Preheat tests are performed to determine the thermal pre-treatment that erases all thermally unstable luminescence components in a sample. In this example, palaeodose remains constant for preheat temperatures of 250°C and above (preheat plateau).

Abb. 15: Vorheiztests werden durchgeführt, um diejenige thermische Vorbehandlung zu ermitteln, mit der alle thermisch instabilen Lumineszenzkomponenten in der Probe entfernt werden. In diesem Beispiel bleibt die Paläodosis konstant für Vorheiztemperaturen ab 250°C (Vorheizplateau).

with using a correction factor, which denotes the ratio of the actual absorbed dose to the theoretical one of an infinite system. Furthermore, the influence of HF etching of grain surfaces can be taken into account by such a factor. Calculated correction values were originally reported by MEJDAHL (1979) and BELL (1979, 1980) and more recently determined by Monte Carlo simulations (BRENNAN et al. 1991, BRENNAN 2003).

There is a variety of methods that can be utilised for radionuclide analysis in this context (cf., SINGHVI & KRBETSCHKEK 1996). Most commonly used are inductively-coupled-plasma mass-spectrometry (ICP-MS), high-resolution gamma spectrometry, neutron activation analysis (NAA) and flame photometry (for K only). To check the performance of the procedures certified reference materials are available for all methods. ICP-MS has three main advantages: (a) small sample size (< 1 g required), (b) low detection limit and (c) high precision. The disadvantages of ICP-MS are: (a) small sample size can be a problem if the sediment exposure is inhomogeneous, (b) total dissolution of the sample, especially if zircon is present, is not straightforward, (c) measuring K by ICP-MS is rather problematic (cf., PREUSSER & KASPER 2001) and (d) this method does not give any information concerning whether radioactive disequilibrium is present in the uranium and thorium decay chains. High-resolution gamma spectrometry is rather time-intensive (each measurement takes at least a day) and requires usually at least 100 g of sample material. Larger sample sizes do, however, reduce uncertainty due to inhomogeneity. The most important advantage of gamma spectrometry is related to the assessment of possible radioactive disequilibrium in the thorium and uranium decay chains (see section 6.4).

The internal dose rate contribution is usually considered to be negligible for quartz grains. In K-feldspars it is often estimated from the stoichiometry of the mineral. DÜTSCH & KRBETSCHKEK (1997) proposed a method for the direct determination of the internal potassium concentration of feldspar grains by studying the peak position of the red radiophosphorescence at about 720 nm. This method was successfully

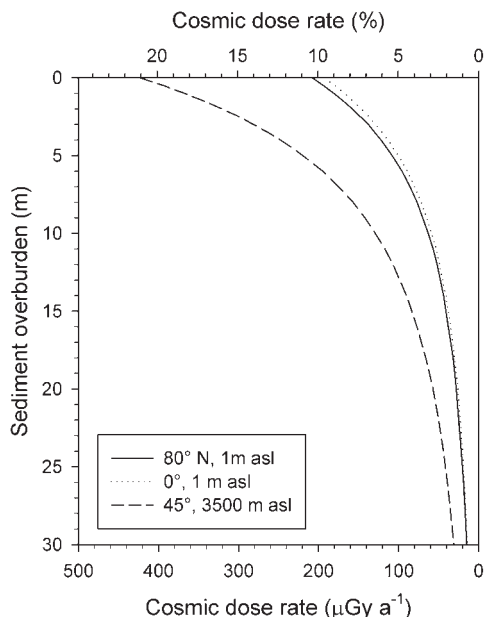


Fig. 16: Dependency of cosmic dose rate on geographic location and sediment overburden, with examples illustrated for sea-level at both high latitude and an equatorial position, as well as a mid latitude position at high altitude. Longitude has a negligible effect on cosmic dose rate. The lower x-axis gives absolute values while the upper x-axis indicates the relative contribution from cosmic rays for a generic sample reflecting a typical sediment with a mean dose rate of 2 mGy a^{-1} .

Abb. 16: Abhängigkeit der kosmischen Dosisleistung von der geographischen Position und der Sedimentüberdeckung. Dargestellt sind Beispiele für hohe Breiten bzw. am Äquator auf Meeressniveau sowie eine Position in den mittleren Breiten in großer Höhe. Der Längengrad hat einen vernachlässigbaren Effekt auf die kosmische Dosisleistung. Die untere x-Achse enthält Absolutwerte, während die obere x-Achse den relativen Beitrag der kosmischen Strahlung für eine Probe mit einer für viele Sedimente typischen mittleren Dosisleistung von 2 mGy a^{-1} anzeigt.

applied to sediments with non-stoichiometric K contents (DEGERING & KRIBETSCHKE 2007).

5.5 Age calculation

Once both the palaeodose and all dose-rate relevant information are available the lumi-

nescence age of a sample together with its uncertainty can be calculated (cf., Aitken 1985). In single aliquot dating, the technique now used by the majority of luminescence dating laboratories, the palaeodose is assessed from many replicated single aliquots. Several statistical methods have been developed to analyse luminescence data and determine the palaeodose. For those samples shown to be completely bleached prior to deposition, some form of central tendency approach is used such as (weighted) arithmetic mean, median or a central age model (cf., Galbraith et al. 1999; Bailey & Arnold 2006). For the palaeodose values determined using any of these approaches standard deviation and standard error are then determined (cf., Geyh 2008). For incompletely bleached samples determination of the palaeodose is more complex (see sections 6.1 and 6.2). For dose rate calculation, the results of measuring dose rate relevant elements (K, Th, U) are combined with the contribution from cosmic radiation and several sample specific factors such as grain size and moisture content have also to be considered (see sections 5.4 and 6.4). These quite complex calculations are usually carried out using special computer programs such as AGE (by R. Grün, Canberra) or ADELE (G. Kuhlig, Freiberg).

6 Problems hindering accurate age determination

6.1 Incomplete bleaching

One of the major problems in luminescence dating is incomplete resetting or bleaching of the latent signal. For sediments, this can occur when the grains are exposed to adequate daylight conditions for too little time and/or if the grains have been transported and deposited by water. Incomplete bleaching is a particular problem for sediments from glacial environments as well as for some fluvial and hill-slope deposits. For all such deposits it is mandatory to investigate if the sample has been completely bleached prior to deposition or not and several approaches exist for doing so.

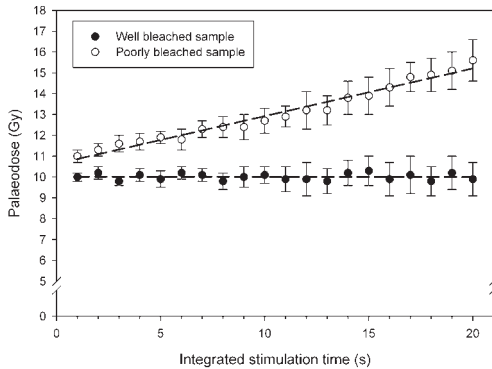


Fig. 17: Shine plateau tests for two samples indicating completely (plateau) and incompletely (increasing palaeodose values) bleached samples. The presence of a shine plateau does not guarantee that a sample has been completely bleached prior to deposition, where they may be potential interference from an unstable signal component.

Abb. 17: Ausleuchtplateautests für zwei Proben, die auf eine vollständig (flaches Plateau) bzw. unvollständig (ansteigende Paläodosisiswerte) gebleichte Probe hinweisen. Das Vorhandensein eines Ausleuchtplateaus belegt jedoch nicht zweifelsfrei, dass eine Probe vor der Ablagerung vollständig gebleicht wurde, da mögliche Störungen durch instabile Signalkomponenten vorliegen können.

One approach is to plot palaeodose as a function of optical stimulation time (in the case of OSL and IRSL) or temperature (in the case of TL). The basic idea behind this approach is that the signal during initial stimulation and at lower temperature, respectively, is more light-sensitive than the subsequent following signal (LI 1991, SINGHVI & LANG 1998, BAILEY 2003a, b). As a consequence, one would expect an increase of palaeodose with stimulation time (temperature) for incompletely bleached samples and a flat shine-plateau-plot for samples that were completely reset at deposition (Fig. 17). Unfortunately, this approach has not been proven to ubiquitously identify incomplete bleaching (e.g., FIEBIG & PREUSSER 2007). Hence, a flat shine-plateau may only be interpreted as evidence for, but not proof of complete bleaching.

The alternative and recently more widely used approach is based on the assumption that sediment grains are not only incompletely, but differentially bleached prior to deposition. This means that the luminescence signal may be completely bleached in some of the grains while the remaining may carry varying amounts of residual signal. When measuring several individual grains from a sample, the lower values in a distribution of doses will most likely represent the grains that were zeroed while the upper values in the distribution will reflect grains that had residual luminescence at deposition (MURRAY et al. 1995). Typically, a completely bleached sample is expected to reveal a Gaussian distribution while incompletely bleached samples should show a positively skewed distribution of palaeodose estimates (Fig. 18). Although many studies have demonstrated Gaussian distributions for samples with the same radiation dose, recent work indicates that in some cases a Gaussian distribution may not be a sufficient criterion for identifying completely bleached samples (FUCHS et al. 2007).

If a sample has been identified as being incompletely bleached the age estimate based on the mean palaeodose for all aliquots has to be considered as the maximum deposition age. To determine the true burial palaeodose the dose distribution must be investigated further, and for this, it is necessary to measure at least a few dozen individual palaeodose estimates to ensure a substantial statistical basis. The different statistical approaches to extract the true burial palaeodose from the distribution of differentially bleached samples will not be discussed in detail here, we would refer the reader to the overview by BAILEY & ARNOLD (2006). For the time being we have to consider that none of the approaches has proven to ubiquitously provide the correct result when compared to independent age control. Dating results for samples taken from certain environments, in particular proglacial settings, should be considered carefully until more sophisticated methods are available. As each sample has its own sedimentary history, it is advisable to date several

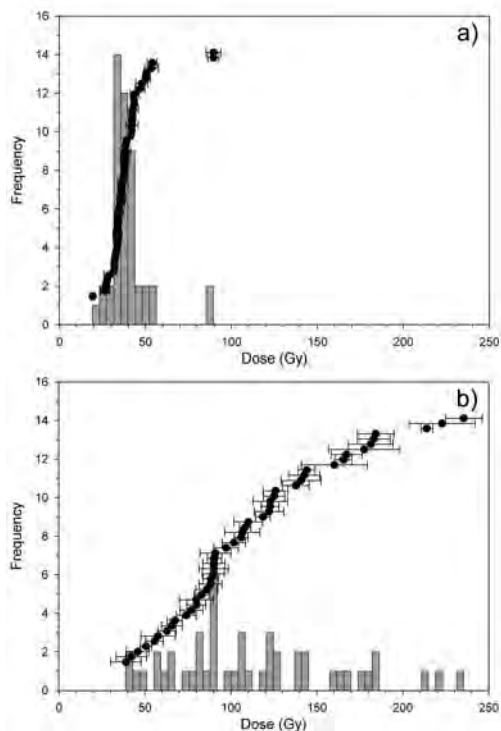


Fig. 18: Scatter of repeated palaeodose measurements for (a) a relatively well-bleached and (b) very poorly bleached sample. While sample (a) displays a symmetric distribution with only two aliquots having significantly higher doses, aliquots of sample (b) show a large scatter in palaeodose values. Both samples are from last glacial (Würmian, ca. 24 ka old) proglacial sediments from Switzerland (from PREUSSER et al. 2007).

Abb. 18: Streuung wiederholter Paläodosismessungen für (a) eine relative gut gebleichte und (b) eine sehr schlecht gebleichte Probe. Während Probe (a) eine symmetrische Verteilung mit nur zwei Aliquots bei signifikant höheren Dosiswerten zeigt, weisen die Aliquots der Probe (b) ein deutlich höheres Streuen der Paläodosismessungen auf. Beide Proben sind aus proglazialen Sedimenten der letzten Vergletscherung (Würmeiszeit, ca. 24 ka alt) aus der Schweiz entnommen worden (aus PREUSSER et al. 2007).

samples from the same sedimentary layer and cross-check the results, and will in most cases help to prove if the age can be considered reliable or needs to be interpreted with caution. Earlier studies also explored the lower light sensitivity of TL compared to optical signals.

For example, WINTLE et al. (1993) compared TL and IRSL of colluvial samples and found that TL ages are up to 10 ka higher than IRSL ages, although IRSL ages also overestimated the expected age and demonstrated that the approach can only be used for sediments where incomplete bleaching is of minor importance such as loess.

Different grain size fractions may also show different degrees of bleaching, with generally two different fractions used in luminescence dating: fine grains (4–11 μm) and coarse grains (90–200 μm). For alluvial sediments, several case studies have shown that the coarser grains appear to be better bleached than finer fractions (e.g., OLLEY et al. 1998; TRUELSSEN & WALLINGA 2003) although relatively little systematic work has been carried out on this issue.

6.2 Scatter of single aliquot and single grain palaeodose values

Multiple replicates of palaeodose measurements on sub-samples from the same sediment sample always show a certain variation among the individual estimates, the causes of which are not all fully understood or quantified at present. One major demand in luminescence studies is to assess the extent of the scatter in palaeodose distributions and determine whether the extent of such scatter represents the natural variation of the dose absorbed during burial. If such a baseline variation is known, additional sources of scatter between palaeodose estimates, in particular caused by incomplete resetting of the luminescence signal prior to burial or pedogenesis (see below), can be identified and discriminated outside of this natural baseline variation (GALBRAITH et al. 2005). Only if these requirements are fulfilled can the true palaeodose accumulated during burial be extracted from complex dose-distributions. One important issue in this context is that the spread in palaeodose is very much dependent on the number of grains on the measured aliquot (WALLINGA 2002b). With regard to aliquot size, it is also important to recognise that only a small proportion of all grains show

a measurable optical signal (cf., DULLER 2004), hence, the luminescence signal from small aliquots most likely originates from a few or even single grains only. For large aliquots, containing several hundred or thousand of grains, the variation of natural palaeodose will be relatively small, whereas for small aliquots the variation will increase, and for single grains will increase still further. For example, the variation in palaeodose values observed for some fine-grain aliquots, which contain hundreds of thousands of grains, can be less than 1 %. On the other hand, a scatter of 20-30 % for small aliquots (less than 50 grains) of bright Australian aeolian quartz has been reported (LOMAX et al. 2003, 2007), although the OSL characteristics of this material are considered to be very suitable. Poorly behaved dim quartz grains, for example from the foreland of the Swiss Alps, show an even higher variation (PREUSSER et al. 2007).

Four different major sources may cause broad and complex palaeodose distributions and these are: (a) laboratory reproducibility of the measurement; (b) partial bleaching; (c) post-depositional sediment mixing caused by bioturbation or similar phenomena; and (d) variation in the distribution of radioactivity within the sediment (microdosimetry).

Reproducibility of the laboratory measurements can easily be determined for each individual sample with a dose recovery test (section 5.3.2). Depending on the particular properties of the minerals investigated and on aliquot size, this typically varies from about 5 % to 15 % (cf., PREUSSER et al. 2007). This is much less than the spread of palaeodose values observed in many natural samples. Incomplete bleaching of the luminescence signal, as discussed above, can result in a substantial scatter of palaeodose values resulting in overestimation of OSL ages. Additionally, pedo-/bioturbation can have an important effect on dose distributions, in particular for sandy deposits such as aeolian dunes (BATEMAN et al. 2003). Pedoturbation is common in former drylands that have later experienced increased humidity resulting in high floral and faunal occupation (BATEMAN et

al. 2007). The most common effect of pedoturbation will be contamination of older sediment by younger surficial material. This will cause a negatively skewed dose distribution where the lower palaeodose values are not representative of the variation in true burial dose. In contrast, partial bleaching will cause a positively skewed dose distribution where the true burial dose lies in the lower dose region, while the upper part of the distribution represents grains that were incompletely zeroed at deposition.

A source that may cause variation of palaeodose is inhomogeneity of exposure to beta irradiation from grain-to-grain within sediments (microdosimetry). This occurs due to the fact that radioactive elements are sufficiently abundant in only a few minerals such as K-feldspar (^{40}K) and zircon (^{238}U and ^{232}Th), so-called “hotspots” of radioactivity. As beta particles penetrate to only a few mm within typical sediment, grains proximal to hotspots will be exposed to a much higher dose rate than other grains and hence absorb more dose during burial. Although the majority of causes of natural variation of palaeodose are difficult to constrain, it is likely that the effect of microdosimetry may be inversely related to the number of hotspots within the sediment matrix. MAYYA et al. (2006) calculated that inhomogeneous microdosimetry will cause positively skewed palaeodose distributions and that the effect on the standard deviation for repeated single grain measurements may be between 18 % and 46 %, being inversely dependent on the K-content of the sediment.

6.3 Anomalous fading

The phenomenon of anomalous fading was first observed by WINTLE (1973) in a study of TL dating of sanidine. Fading is a loss of luminescence signal with time; this implies that some of the electrons captured at traps are unstable and do not possess the trap lifetime exhibited by the majority of electrons in the trap. The effect in a dating application will be that the laboratory induced signal from a particular trap will originate from both stable and unstable charge,

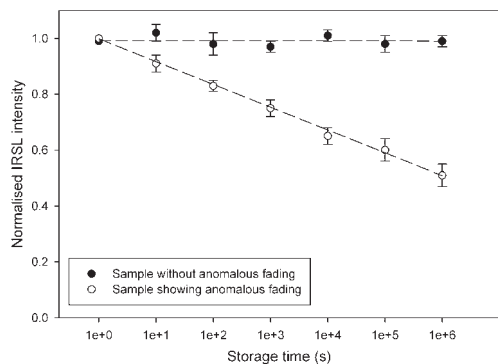


Fig. 19: In the presence of anomalous fading laboratory irradiation will produce a signal that decays (fades) over time. This same unstable signal is not present in the natural sample and, as a consequence, the absorbed palaeodose and hence the age of the sample will be underestimated.

Abb. 19: Bei vorhandenem anormalem Ausheilen (Fading) produziert die Laborbestrahlung ein Signal, welches mit der Zeit zerfällt. Dieses instabile Signal ist in der natürlichen Probe nicht vorhanden. Daraus resultiert, dass die absorbierte Paläodosis und somit das Alter der Probe unterschätzt werden.

whereas the natural signal from the same trap will derive only from the stable charge. The same artificial radiation dose will produce a much higher signal than that resulting from natural irradiation and, as a consequence, the palaeodose will be underestimated as will the age of the sample (Fig. 19).

Preheating the samples prior to measurement (section 5.3) will remove most of the unstable charge, but several studies have shown that this unwanted charge may remain even after applying rigorous thermal pre-treatments. One possible fading mechanism that has been suggested is attributed to the tunnelling of electrons (e.g., VISOCEKAS 1985), whereby charge tunnels between traps and luminescence centres rather than recombining via the conduction band. While the phenomenon is normally only attributed to feldspars, recent publications imply that it may also exist in volcanic quartz (BONDE et al. 2001; TSUKAMOTO et al. 2007). With regard to feldspar, there is much debate over whether fading affects this mineral ubiqui-

tously (e.g., HUNTLEY & LAMOTHE 2001) or, if it is related to feldspar of specific geological origin and, at least partly, inappropriate laboratory procedures. With regard to the latter, two issues are of major relevance. Firstly, the thermal pre-treatment must be long enough, and the temperature high enough to remove thermally unstable components. Secondly, KRBETSCHKEK et al. (1997) have shown that unstable components in feldspars are mainly related to emissions in the UV band, and recommend that these emissions should be avoided, suggesting either the use of short-wave blocking optical filters (e.g., a Schott GG400) or interference filters that only allow transmission of emissions in a very narrow band. While studies implementing these recommendations often produce ages consistent with independent age control (e.g., CLARKE & RENDELL 2003; PREUSSER 2003), those studies incorporating the UV emissions have reported significant underestimation of luminescence ages (e.g., HUNTLEY & LAMOTHE 2001).

Nevertheless, further studies utilising a restricted detection window have still observed fading of feldspars in their samples (e.g., WALLINGA et al. 2007), and this may be related to the measurement of feldspars of volcanic origin, which have been shown to be predominantly affected by fading, and may be due to the disordered crystal lattice in volcanic minerals resulting from rapid cooling and crystallisation (SPOONER 1994). This may also explain the fading observed in volcanic quartz.

Storage tests performed at either room or elevated temperature indicate whether a sample is affected by fading (AITKEN 1985; SPOONER 1992), and are a suitable approach as it is expected that fading components decay on a logarithmic scale (VISOCEKAS 1985, 2002; SPOONER 1994). Where fading is observed in samples it is possible to calculate the rate of fading per decade and use it to correct the age (HUNTLEY & LAMOTHE 2001; AUCLAIR et al. 2003; LAMOTHE et al. 2003). LAMOTHE et al. (2003) undertook fading correction for samples both on the linear part of the dose response curve and for geologically old sediments in luminescence field saturation. However, a recent study by WALLINGA et al. (2007) dem-

onstrated that the applied fading correction was not sufficient to account for the offset between feldspar and quartz ages. More research in this area is necessary to fully utilise the potential of feldspars in dating; in particular the higher saturation dose and hence increased dating range.

6.4 Accurate dose rate determination

Besides the problems associated with the determination of the dose absorbed during burial (palaeodose) there are also potential errors related to the correct calculation of dose rate, some of the most important of which are discussed in the following.

6.4.1 Alpha efficiency

Alpha particles produce much less luminescence signal compared to beta and gamma rays, and so the effective dose from alpha irradiation is therefore multiplied by a factor that reflects this lower ionisation efficiency; this factor, referred to as the a -value, is usually < 0.1 (AITKEN & BOWMAN 1975).

For sand-size minerals, for which most research has concentrated during the last decade, a -values are of minor importance as alpha particles penetrate only the outer ca. $10 \mu\text{m}$ of the grain, and the outer rim of such grains is usually removed by HF etching during sample preparation. When measuring fine-silt grains, for example when dating loess, lacustrine sediments or fine silicate inclusions from ceramics, the a -value is of major importance. For such materials it is recommended that the a -value is determined for each individual sample, which involves comparison of laboratory-induced luminescence due to both alpha and beta radiation (e.g. AITKEN 1985).

6.4.2 Internal K-content

With regard to the internal dose rate of feldspar, there is the potential to underestimate the K-concentration where chemical analyses (e.g. AAS) or beta counting are used, and is due to the fact that potassium content lower than the

stoichiometric maximum can be caused by contamination of the sample by, for example, quartz that is often found in the feldspar fraction, but is not relevant for palaeodose determination. An extended chemical analysis (e.g. K, Na, Ca, Al, Si concentration), the use of micro-probe equipment and the calculation of a theoretical feldspar composition may overcome this problem to some extent (DÜTSCH & KRBETSCHKE 1997).

6.4.3 Sediment moisture

Another major uncertainty is associated with the moisture content of a sample during burial. Water in the sediment pores absorbs much more radiation than air and so the effective dose acting on a grain is higher in sediments with low moisture content compared to a similar saturated sediment (Fig. 20). In age calculation, an average value for moisture over the whole burial time is usually assumed, but the lack of information concerning the extent of moisture variation throughout the burial period means that this is often the greatest source of uncertainty in a luminescence age. The present moisture content of the sample is often used as a guideline for age calculation but it is crucial that potential changes in the hydrological conditions are carefully considered during sampling. It is often also

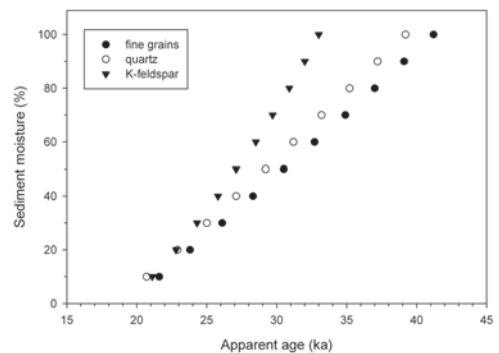


Fig. 20: Impact of sediment moisture on calculated luminescence age.

Abb. 20: Einfluss der Sedimentfeuchte auf das berechnete Lumineszenzalter.

appropriate to measure water up-take capability of samples to give an estimate of maximum moisture content (cf., AITKEN 1998). For example, fluvial sediments are usually water saturated directly after burial but the height of the water table may change when rivers incise or terraces are tectonically up-lifted. In such cases the sediment may be above the water table for a significant part of the burial period and pores may be only partially filled with water. In lacustrine settings, pore space will decrease with on-going consolidation of the sediment as discussed in the context of luminescence dating by JUSCHUS et al. (2007). Uncertainties related to sediment moisture are most important for water-lain sediments but can in principle also be significant in certain other sedimentary settings such as coastal deposits or sediments that have been affected by permafrost in the past.

6.4.4 Cosmic dose rate

The contribution of cosmic radiation to the total dose rate is usually calculated according to the sampling depth and geographical position of the sampling site following PRESCOTT & HUTTON (1994). The dynamic character of sedimentary systems with repeated events of erosion and accumulation results in variation of the thickness of the overburden above a buried sediment sample. Therefore, the contribution of cosmic rays to the total dose rate of the sample is not constant over the entire period of burial time, but may vary significantly. Such fluctuation in the overburden thickness is less significant in sedimentary environments where the contribution of cosmic rays to the total dose is generally low. Frequently the proportion measures less than 10 %. However in quartz-rich aeolian dune sand system the dose rate tends to be low and the cosmic ray contribution to the annual dose can be a major component of the total dose rate. In such cases a substantial error in total dose rate calculation and, hence, luminescence age can be introduced if the cosmic dose contribution is calculated using present sampling depth. To provide a more ac-

curate dose rate assessment the burial history of a sample should be taken into account, as far as this can be reconstructed (MUNYIKWA 2000).

6.4.5 Radioactive disequilibria

Special attention must be directed to sediments exhibiting radioactive disequilibria since the prerequisite of constant radionuclide concentrations does not hold (cf., KRIBETSCHKEK et al. 1994). The radioactive elements of uranium and thorium are the beginning members or 'parents' of three decay series starting with the isotopes ^{238}U , ^{235}U and ^{232}Th . Their half-lives are of the order of 10^9 to 10^{10} years and thus they are still present in nature. These parent radionuclides and their stable end 'daughter' products of ^{206}Pb , ^{207}Pb and ^{208}Pb , respectively, are linked together by decay chains including several daughter isotopes of various elements but with half-life times much shorter than 10^9 years. Table 2 gives the physical and chemical properties of the most significant members of ^{238}U and ^{232}Th decay series in dose-rate determination.

In an undisturbed geological situation the activities within each decay series are in 'radioactive equilibrium', characterised by equal activities of all involved nuclides. Physical and chemical differentiation processes lead to a loss of this balance and to radioactive disequilibria. In closed systems the balance is restored on a time scale of the order of ca. five half-lives of the radionuclide concerned. Open systems can reach stationary states if geochemical properties do not change with time. As a consequence of the time dependent change in activities in the case of radioactive disequilibria, the external dose rate also becomes a function that varies with time. Equation (1) must then be replaced by an iteration technique which searches for the point in time t , at which equation (2) holds:

$$\int_{t_0}^{t_0+t} \dot{D}(\tau) d\tau = D \quad (2)$$

where t_0 is the moment of sample bleaching. In the case of radioactive disequilibria one needs: (a) preferably an analysis of all long-lived ra-

Table 2: Physical and chemical properties of the dosimetrically most significant isotopes of ^{238}U and ^{232}Th decay series.Tab. 2: Physikalische und chemische Eigenschaften der dosimetrisch wichtigsten Isotope der ^{238}U und ^{232}Th Zerfallsreihen.

Decay series	Nuclide	Half life	Significant Properties
^{238}U series	^{238}U	$4.5 \cdot 10^9$ a	Actinide, heavy metal, forms organic complexes, valence states U(IV) (low solubility) and U(VI) (soluble as uranyl ion (UO_2) ²⁺)
	^{234}U	$2.5 \cdot 10^5$ a	
	^{230}Th	$7.5 \cdot 10^4$ a	Actinide, tetravalent, low solubility
	^{226}Ra	$1.6 \cdot 10^3$ a	Earth alkali metal, soluble
	^{222}Rn	3.8 d	Inert gas
	^{210}Pb	22 a	Heavy metal, forms organic complexes
^{232}Th series	^{232}Th	$1.4 \cdot 10^{10}$ a	cf. ^{230}Th
	^{228}Ra	5.8 a	cf. ^{226}Ra
	^{228}Th	1.9 a	cf. ^{230}Th

dionuclides; and (b) a model of the possible activity evolutions in the past in agreement with both the geochemical parameters of the system and with the modern radionuclide contents.

Low-level high-resolution gamma spectrometry is suitable for multi-nuclide analyses, mostly fulfilling criterium (a). Generally, this method is suitable for determination of the following long-lived nuclides (in some cases with the use of short-lived daughter nuclides): ^{238}U , (^{230}Th), ^{226}Ra , ^{210}Pb from ^{238}U series, ^{228}Ra , ^{228}Th from ^{232}Th series and ^{40}K .

The time evolution of radioactive disequilibria in closed systems and in special types of open systems can be analytically solved. Mathematically, the solution for each activity term is a superposition of exponential decays with coefficients containing the initial activities, the decay constants and exchange parameters in open systems (DEGERING & KRBETSCHKE 2007).

Modelling the disequilibrium in the past is based upon assumptions concerning the value of the prevailing parameters and their variability with time. In most cases this results in a more or less broad range of possible time evolutions, leading to the current radionuclide concentrations. In comparison to ages ignoring

disequilibria, the uncertainty in age increases but so too does reliability.

In recent years several samples exhibiting radioactive disequilibria have been accurately dated when compared to independent age control from radiocarbon dating. From these investigations some indications of “suspicious” systems and their treatment can be derived:

- Organic substances tend to accumulate complex-forming elements like uranium or lead. Since the solubility of U and Th are very different, organic sediments like peat often show strong U/Th imbalance. The most important is $^{234}\text{U}/^{230}\text{Th}$ disequilibrium and is definitely predictable for closed systems (DEGERING & KRBETSCHKE 2007).
- Changes in the moisture content or in the groundwater level may lead to variable redox conditions and hence changes between open and closed system characteristics. Especially for fine grain dating, the dosimetric consequence of variable moisture is a variable attenuation of the external irradiation with time. The resulting enhanced uncertainty in the dating result can only be reduced by applying a model considering this variation (PREUSSER & DEGERING 2007).

- Calcareous material deposits containing, for example, mollusc shells or corals are able to exchange radionuclides with their environment. Reasonable explanations of the modern radionuclide contents partly require the assumption of an open system, mainly connected with uranium uptake after deposition. The clear reconstruction of the evolution of disequilibrium in the past is hardly practical here. A careful investigation should include the calculation of borderline cases to give an impression of the likely span of reliable ages (ZANDER et al. 2007).

7 Suitable materials

Up until the 1980s most research in luminescence dating had focused on fundamental problems and the application of TL for dating heated objects and volcanic deposits as well as some initial work on TL dating of sediments (cf., AITKEN 1985). Since the introduction of optical dating (using IR and visible light) most research has focused on the dating of sediments, in particular in the context of palaeoclimate change as well as archaeology (Table 3, 4). General overviews on early work are provided by PRESCOTT & ROBERTSON (1997), AITKEN (1998) or STOKES (1999) and the following sections will concentrate on benchmark publications and the most recent developments.

7.1 Archaeological materials

A wealth of archaeological materials can be dated with luminescence methods. Apart from unheated sediments (see section 7.3) containing archaeological remains, two main categories can be distinguished: (1) heated sediment e.g. ceramics, pottery, bricks, fire places, fire pits, hearths, kilns; and (2) heated rocks e.g. flint, chert, quartzite, quartz, silcrete, sandstone and other rock materials. An additional category includes man made materials such as slag and walls constructed from rock materials.

The association of dated material and human occupation is always a key question when applying chronometric dating methods in es-

tablishing the age of archaeological sites or, in other words, the relationship between the event dated and the question asked has to be clearly shown. However, it is often only through luminescence on heated materials that an age of an archaeological event can directly be provided (cf., RICHTER 2007; ALPERSON-AFIL et al. 2007 for discussion of natural fires).

Table 3 lists the commonly used materials/samples in luminescence dating, together with the appropriate method and the event dated, which is sometimes not evident at first sight. In some cases it is important to take the life time of an artefact or feature into consideration, e.g. a kiln could have been used for an extended period of time, but in general only its last use can be dated and not the time of construction. Or in the case of dating rock surfaces, the time since the last exposure to light is dated, which can be the time of construction or destruction of a monument. Ceramics could be much younger than an associated construction or much older, e.g. when they are discarded in a pit or trench from older deposits and are therefore out of context. The depositional age of sediment infill provides age information concerning abandonment rather than construction, e.g. of a trench or pit. Anthropogenic sediments or sediments altered by human occupation are difficult to date with luminescence methods. It is therefore often advisable to select more suitable sedimentological units bracketing the archaeological layer of interest and thus OSL dating can provide *termini post quem* and *ante quem* age estimates.

Heated rocks are usually dated by TL but if the material is sufficiently sensitive OSL methods can be applied as well. In such cases, bleaching during excavation might be a major concern (e.g., TRIBOLO 2003), although flint, for example, has been shown to be insensitive to optical stimulation (POOLTON et al. 1995). Conversely, TL of flint is very hard to bleach by light for most samples from a highly diverse material group (RICHTER et al. 1999).

The first luminescence ages on archaeological material were obtained using TL on ceramics by GRÖGLER et al. (1958). TL dating of ceram-

Table 3: Overview of different dating applications for archaeological materials by luminescence methods.

Tab. 3: Überblick über die verschiedenen Anwendungsbereiche der Lumineszenzmethoden zur Datierung archäologischer Materialien.

Material	Material details	Method	Dated event	Interpreted as time of	Archaeological example
Brick	Quartz, feldspar	TL/OSL	Last firing	Manufacture	BAILIFF & HOLLAND (2000)
Brick surface	Quartz, feldspar	OSL	Last exposure	Construction; repair; destruction	BAILIFF & HOLLAND (2000)
Ceramic, pottery, tile	Quartz, feldspar	TL/OSL	Last firing	Manufacture; authenticity	BARNETT (2000)
Daub (burnt)	Quartz, feldspar	TL/OSL	Last heating	Destruction	QUICKERT et al. (2003)
fFigurine	Quartz, feldspar	TL/OSL	Last heating	Manufacture; authenticity	ZINK & PORTO (2005)
Hearth stone	Sandstone, limestone, granite (quartz, feldspar)	TL/OSL	Last heating	Last use	ICHIKAWA & NAGATOMO (1978)
Kiln	Quartz, feldspar	TL	Last firing	Last use	HONG et al. (2001)
Lithic artefact (heated)	Flint, chert, quartzite, quartz, silerete	TL	Last heating	Discard	RICHTER et al. (2007)
Limestone (heated)	Calcite, quartz	TL	Last heating	Last use	ROOUE et al. (2001)
Mortar	Quartz, feldspar	TL/OSL	Last light exposure	Building	GOEDICKE (2003)
Oven	Quartz, feldspar	TL	Last firing	Last use	ROOUE et al. (2002)
Pit/trench infill	Quartz, feldspar	OSL	Last light exposure	Abandonment; infilling	LANG & WAGNER (1996)
Rock surface	Granite, marble, limestone (quartz, feldspar, calcite)	OSL/TL	Last light exposure	Construction; destruction	GREILICH et al. (2005, 2006); LIRITZIS & VAFIADOU (2005)
Sediment (burnt)	Quartz, feldspar	TL/OSL	Last heating	Last use	GODFREY-SMITH & SHALEV (2002)
Sediment, aeolian	Quartz, feldspar	OSL	Last light exposure	Deposition	JACOBS et al. (2003 a,b)
Sediment, colluvial	Quartz, feldspar	OSL	Last light exposure	Deposition	LANG & HÖNSCHIEDT (1999)
Sediment, fluvial	Quartz, feldspar	OSL	Last light exposure	Deposition	FOLZ et al. (2001)
Slag	Quartz	TL	Last firing	Last use	HAUSTEIN et al. (2003)
Wasp nest	Quartz, feldspar	OSL	Last light exposure	Building	YOSHIDA et al. (2003)

ics and pottery soon became the backbone for establishing many archaeological chronologies in the world, but with the increased precision of archaeological dating (including seriation etc.) and the possibility of providing calendar ages by calibrating radiocarbon data, the application of TL dating of ceramics and pottery became less important from the 1980s onward. However, BARNETT (2000) used TL to verify the typo-chronologies for the British Late Bronze and Iron Age, using both shards that were diagnostic by their shape and/or decoration, as well as those that were undiagnostic (fabric based chronology), and it was shown that the established chronology for diagnostic pottery was supported by the TL results. As the conditions for luminescence dating were not different for the diagnostic samples, it was concluded on the other hand that the accuracy of chronologies based on fabric only required refinement (BARNETT 2000).

Establishing the timing of construction, repair and destruction of parts of the medieval citadel of Termez in Usbekistan was attempted by VIELLEVIGNE et al. (2006), who showed the feasibility of a combined OSL and TL approach on different bricks, that could be related to the reuse of older bricks, as was also done by BAILIFF & HOLLAND (2000).

The correlation of features considered to be fire places, hearths or fire pits, which lie close to an archaeological settlement site but lack remains like hearth stones, is often difficult to establish. The heat-reddened rim and infill of a pit from an Early Bronze Age context at Ashkelon Marina in Israel was dated with TL additive methods and OSL by GODFREY-SMITH & SHALEV (2002). While the TL age for the heated sediment agrees well with radiocarbon data, the TL result for the pit infill is greatly overestimated and clearly shows that this method is inappropriate for unheated sediment. On the other hand an OSL age for the pit infill agrees well with the TL age of the heat-reddened rim, suggesting the pit was rapidly filled after disuse and confirming the likely function of the fire pits for early copper smelting technology.

TL-dating of domestic ovens for the Neolithic

site of Dikili Tash in Greece (ROQUE et al. 2002) provided confidence in the often assumed association of radiocarbon dating samples with occupational levels and strengthens the Neolithic chronological framework for that region. It is possible to establish the authenticity of figurines and ceramics using TL and OSL, although an accurate date cannot usually be given due to the lack of knowledge of the precise radiation field of each individual object. ZINK & PORTO (2005) investigated a large collection of Tanagra figurines and used probability statistics to distinguish fake from authentic ones.

Quartz pebbles from the site of Pedra Furada in Brazil were dated with TL techniques by VALLADAS et al. (2003). These pebbles are assumed to have been used as hearthstones and/or for heating liquids. The site of Pedra Furada is controversial because of the claim that it has a much greater antiquity than the time generally recognised for the arrival of humans in the Americas. While the TL age estimates prove the antiquity of the heating, the question of the anthropogenic use of the pebbles cannot be solved solely by establishing the time when fire occurred (VALLADAS et al. 2003). Sandstones are the most common material used to establish the age of distinct hearths but limestone was more frequently used to construct fireplaces in the Palaeolithic. The feasibility of using TL for dating heated limestone is reported by ROQUE et al. (2001) from the Upper Palaeolithic site of Combe Saunière in France.

TL dating of heated flint plays a major role in establishing the relationships in time between tool assemblages, technocomplexes and hominid species, under the assumption that the time difference between archaeological material surrounding the intrusive burial and skeletal material is negligible. Heated flints from layers containing Middle Palaeolithic artefacts in several caves in Israel which are associated with Neanderthal remains in some and with modern humans in others were dated (VALLADAS et al. 1987; 1988, 1999; MERCIER et al. 1993). These dates provide evidence for an extended overlap of several thousand years of the two species within a relatively small region. In general, TL

Table 4: Overview of different applications of luminescence methods for dating sediments (n.a. = not available).

Tab. 4: Überblick über verschiedene Anwendungsgebiete der Lumineszenz zur Datierung von Sedimenten.

Sediment type	Severity	Most common problem	Pioneering study	Review
Loess	Minor		TL: WINTLE (1981)	SINGHVI et al. (2001)
Aeolian sand	Minor		TL: SINGHVI et al. (1982)	SINGHVI et al. (2001)
Fluvial deposits	Average	Partial bleaching	TL: FORMAN et al. (1988) OSL: PERKINS & RHODES (1994)	WALLINGA (2002 a)
Hillslope sediments	Average	Partial bleaching	IRSL: WINTLE et al. (1993)	FUCHS & LANG (2008)
Lacustrine sediments	Average	Sediment moisture	TL: BERGER (1990)	n.a.
Proglacial deposits	Challenging	Partial bleaching	TL: GEMMELL (1985) IRSL: HÜTT & JUNGNER (1992)	n.a.
Coastal deposits	Average	Radioactive disequilibria	TL: BAILESCU et al. (1991)	n.a.
Deep sea sediments	Average	Radioactive disequilibria	TL: WINTLE & HUNTLEY (1979)	n.a.

methods are used to establish the age of individual archaeological layers and thus help to establish the chronologies of technocomplexes beyond the range of radiocarbon dating, e.g. for the middle Palaeolithic chronology of Western Europe (RICHTER et al. 2007). Recently, a SAR protocol employing red TL was developed, which allows the dating of small samples of heated flint, thus increasing the number of Palaeolithic sites, which could be dated (RICHTER & KRBETSCHKE 2006). Mastering fire is regarded as one of the hallmarks in human evolution and certainly played an important role in dramatic changes in human behaviour connected with diet, defence and social interaction. Analysis of lithics suspected of having been heated using TL helped provide evidence for the controlled use of fire at the Acheulian site of Gesher Benot Ya'aqov in Israel, where distinct clustering of heated and non-heated materials reveal the positions of former fire places (ALPERSON-AFIL et al. 2007). TL dating of heated flint can also be used to verify the integrity of an archaeological site or layer. Results of heated flint from an assemblage dated by DEBENHAM (1994) provided

evidence of an otherwise unobserved severe mixing of a Palaeolithic site.

Slags are often the only remains from archaeometallurgical activities and the association of dates obtained from materials in the vicinity is often questioned. The application of a combination of the red TL with a SAR protocol on extracted quartz by HAUSTEIN et al. (2003) showed a fair agreement in comparison with independent age control.

Determining the age when a wall was built, a megalith was erected, repair work or destruction took place, or even when rocks were moved and discarded or reused is of great importance in archaeology. The recent development by GREILICH et al. (2005, 2006) on the OSL dating of surface exposure is a significant development. These authors were able to establish surface exposure ages of granitic surfaces from rocks used to construct some of the Nasca geoglyphs. These results were contrasted by the surface exposure age of rocks which were unmoved for several 10^3 years. But even standard TL and OSL methods are capable of producing reasonable age estimates of surface exposure of

Neolithic as well as Greek monuments (LIRITZIS & VAFIADOU 2005).

7.2 Volcanic deposits

The dating of volcanic deposits was developed in the early 1970s, soon after the successful application of TL to date archaeological materials. Despite the fact that anomalous fading appears to affect most volcanic feldspar (WINTLE 1973), and possibly volcanic quartz as well, (TSUKAMOTO et al. 2007) more than 50 studies have been published on the dating volcanic deposits. A comprehensive review on studies carried out in the 1970-90s is provided by FATTAHI & STOKES (2003b). TL dates have been used to establish chronological frameworks for volcanic activity in areas such as the Massif Central, France (VERNET et al. 1998) and China (LI & YIN 2001). While FATTAHI & STOKES (2003b) summarise good consistency of TL ages with independent age control for several dating studies, problems with anomalous fading occurred in many other studies. An important aspect is the use of the red emission from feldspar, which has proven to be more stable than other TL emissions (e.g. FATTAHI & STOKES 2000; MIALLIER et al. 2004; GANZAWA et al. 2005; BASSINET et al. 2006). However, relatively little research has been carried out and to date only a few research groups have focused on this topic.

7.3 Sediments

7.3.1 Loess

Loess was among the first sediments that were systematically dated by luminescence methods, in particular by TL (e.g., WINTLE 1981; WINTLE 1990). In most early dating studies, the optically insensitive TL signal was determined by laboratory bleaching using UV lamps (or by using daylight) and used to define the zero point of the dose response curve (WINTLE & HUNTLEY 1979). This total bleach approach assumes that all light-sensitive TL is removed from the grains during transportation and this

has been investigated by plotting palaeodose versus TL-temperature for the samples investigated (the so-called plateau test). It has been demonstrated that partial bleaching is of minor importance for loess that has been deposited by aeolian processes but can be relevant for re-worked loess.

A frequent problem has been a systematic underestimation of TL (e.g., ZHOU et al. 1995), the reason for which is not fully understood. This may be related in part to the use of UV filters for luminescence detection, which allow an unstable UV emission from feldspathic minerals to be transmitted. However, as many other studies have provided dates consistent with independent age control and geological time models it also became evident that the application of luminescence (both TL and optical stimulation) is apparently restricted to the last glacial cycle (i.e. the last 130 ka). A comprehensive review of the work carried out in the 1980-90s is provided by SINGHVI et al. (2001). More recently, single-aliquot methodology, in particular for quartz, has been applied to loess to improve both the precision and reliability of the chronology as well as to extend the datable age range. ZANDER et al. (2000) compared single-aliquot sand-size quartz OSL and multiple-aliquot silt-size polymineral IRSL and TL on samples from a site in Czech Republic. They found good consistency for the different methods but higher precision of the quartz ages. WATANUKI et al. (2005) dated a loess sequence in Japan applying the SAR methodology on quartz and polymineral fine-grain extracts. They produced reliable quartz ages as old as 500 ka with independent age control from tephra chronology. Polymineral fine-grain ages, which have been measured using the UV emission band, are underestimated by 10-20 % in this study. A similar approach has been tested by WATANUKI et al. (2003) and WANG et al. (2006a) on Chinese loess and these authors also identified a systematic underestimation of polymineral ages using UV emissions. A comprehensive summary of previous work on luminescence dating of Chinese loess is provided by STEVENS et al. (2007). These authors

also conclude, using high resolution sampling for OSL dating, that loess accumulation has been episodic and not continuous during the last glacial cycle, in contrast to many previous assumptions. BUYLAERT et al. (2007) concluded that the SAR procedures used in their studies do not allow dating beyond the Last Interglacial, and yielded a systematic underestimation of quartz OSL ages, for example, an OSL age of 112 ± 7 ka and fading corrected IRSL age of 301 ± 30 ka for loess from below the Brunhes/Matuyama (B/M) boundary.

Present work is focusing on extending the dating range in Chinese loess by developing new measuring techniques such as red TL from quartz (LAI & MURRAY 2006; LAI et al. 2006) and thermally-transferred OSL (WANG et al. 2007). The latter in particular looks rather promising, with WANG et al. (2006b) reporting a mean TT-OSL age of 771 ± 15 ka for loess from the B/M boundary.

7.3.2 Aeolian sand

Due to the aeolian transport processes of saltation, raptation (creep) and suspension the sediment grains are most likely exposed to sufficient light for optical re-setting of the luminescence signal prior to burial, and so poor bleaching is rarely a problem in aeolian materials (DULLER 2004). As aeolian environments provide ideal conditions for the application of luminescence dating, a vast number of dating studies on dune sands has been carried out during the past decades since SINGHVI et al. (1982) published the first study on aeolian sand dunes from Rajasthan, India. Since then, luminescence dating has been used to constrain the chronology of aeolian sands in a variety of drylands such as the Kalahari (STOKES et al. 1997a; TELFER & THOMAS 2007), Namib (BRISTOW et al. 2005, 2007), Sahara (LANCASTER et al. 2002; BUBENZER et al. 2007), Sahel (STOKES et al. 2004), Arabian (PREUSSER et al. 2002; STOKES & BRAY 2005), Taklamakan (YANG et al. 2006) and Australian (GARDNER et al. 1987; NANSON et al. 1992; TWIDALE et al. 2001; LOMAX et al. 2003; FITZSIMMONS et al. 2007;) deserts.

Furthermore, several studies have focussed on aeolian sand deposits in former periglacial regions such as those in the European sand belt (DIJKMANS et al. 1992; BATEMAN 1998; BATEMAN & VAN HUISSTEDEN 1999; HILGERS et al. 2001; VANDENBERGHE et al. 2004; HILGERS 2007; KASSE et al. 2007), the Pampa of Argentina (TRIPALDI & FORMAN 2007) and in North America (STOKES et al. 1997b; FEATHERS et al. 2006; FORMAN et al. 2006).

Compared to water-lain deposits luminescence dating of aeolian sand, especially considering the recent advances in methodology (e.g. SAR protocol), is relatively straightforward as partial bleaching is not usually a major concern for such sediments. However, aeolian deposits play a major role in understanding the complexity of dose distributions and hence in improving the accuracy and precision of luminescence dating (BATEMAN et al. 2003; VANDENBERGHE et al. 2003; LOMAX et al. 2007; see Section 6.4 for details). Aeolian deposits are also important in studies that have explored the possibilities of extending the minimum and maximum age limits. For example, BALLARINI et al. (2007) successfully explored the potential to date dune sand as young as 10 a. Some of the oldest luminescence ages (in this case TL) ever published have been determined for stranded beach dunes from SE Australia that yielded ages as old as 800 ka (HUNTLEY et al. 1993; HUNTLEY & PRESCOTT 2001). More recently, RHODES et al. (2006) provided OSL ages of about 500 ka for aeolian deposits from Morocco and a single age of 1 Ma, the latter being from inversely magnetised sediments attributed to the Matuyama chron. These latter two examples are, however, exceptional due to the low dose rate (< 1 mGy a^{-1}) of the investigated sediments, and saturation of the luminescence signal will be reached earlier at higher dose rates.

Dunes are complex and composite features formed during several events of deposition which may have been interrupted by episodes of erosion, and as such represent inherently discontinuous archives, which complicate the interpretation of OSL records in terms of their significance for palaeoclimate or pal-

aeo-environmental reconstructions. Thus, any interpretation of OSL data has to be done in the context of the complete environmental system, including the effective geomorphological processes (e.g., BUBENZER et al. 2007). Various approaches are taken to minimise this problem and to optimise the value of luminescence dating results. The necessity of an increase of samples analysed per dune as well as the investigation of several dunes within a dune field to obtain not only detailed but informative records of aeolian processes in a certain area have been demonstrated for example by TELFER & THOMAS (2007) and HILGERS (2007). The combination of ground-penetrating radar (GPR) and OSL dating provides a powerful tool to reveal the dynamic history of dunes. Based on the GPR profiles of the internal structure of the dunes sampling locations for OSL dating can be determined (e.g., BRISTOW et al. 2007).

7.3.3 Fluvial deposits

Most of the early studies using TL for dating fluvial and other water-lain sediments revealed age overestimations due to incomplete bleaching of the signal prior to deposition (e.g., FORMAN et al. 1988). With the advent of OSL methodology the dating of waterlain sediments was reconsidered, but took nearly a decade from the breakthrough publication of HUNTLEY et al. (1985) to the first systematic studies on dating waterlain deposits. For example, PERKINS & RHODES (1994) used multiple-aliquot methodology to date sandy River Thames sediments and achieved a good consistency with independent age control. Nevertheless, up to the late 1990s fluvial and in particular glaciofluvial sediments (see section 7.3.6) have been considered rather challenging and the reliability of optical dating of such deposits has been considered questionable at least.

However, it has been the dating of fluvial deposits that led to a major improvement in luminescence methodology with the development of single aliquot techniques (see section 5.3.2). One of the pioneering studies was that of MURRAY et al. (1995) who investigated sub-modern fluvial sediments from Australia using

a modified version of the so-called SARA protocol (MEJDAHL & BØTTER-JENSEN 1994), a technique that is only infrequently used nowadays. The study by MURRAY et al. (1995) was the first in which a large number (>100) of individual palaeodose values were calculated for individual samples. As already indicated by DULLER (1994), it was demonstrated that samples not fully zeroed consist of a mixture of grains having a different degree of bleaching, including grains that have been completely bleached at deposition. Hence, the scatter of several individual palaeodose values will be broad and positively skewed for incompletely bleached samples, and tight and normally distributed for well-bleached samples (cf., OLLEY et al. 1998, 1999). A comprehensive review on the problems and potential of optical dating of fluvial deposits has been provided by WALLINGA (2002a) and the following discussion will concentrate on a few aspects only.

As a major concern has always been the degree of resetting of the optical signal prior to deposition, several studies have investigated the resetting of modern analogues. For example, STOKES et al. (2001) observed a monotonic decrease of apparent palaeodose in modern deposits of the River Loire downstream from the source, with values close to zero for transport distances of more than 300 km. Consequently, STOKES et al. (2001) concluded that the resetting of the OSL signal in a large drainage basin is much more likely than considered from many earlier studies. This assertion was later confirmed by SINGARAYER et al. (2005) who identified different resetting for individual components of the OSL signal. FUCHS et al. (2005) investigated the degree of zeroing in deposits of the 2002 millennium flood in Saxony and compared quartz and feldspar signals. These authors observed residual ages of 0.14–0.45 ka for silt-size quartz and 1.12–4.00 ka for polymineral fine-grains for samples from the River Elbe. For the River Rote Weißeritz, where sand-size grains have been investigated, residual ages of 0.36–0.83 ka for quartz and 2.12–4.67 ka for feldspar have been calculated. Similar low residual quartz OSL ages are reported for mod-

ern deposits of River Ahr in western Germany (CHOI et al. 2007). On the other hand, FIEBIG & PREUSSER (2007) observed rather high residual ages in some of the young deposits of the River Danube, parts of which were deposited by the floods in 2002. For some of these samples residual ages are as high as several 10 ka. However, these exceptional values are probably the result of an artificial input of sediment to prevent riverbed erosion.

During the last decade OSL dating has been applied to the reconstruction of the fluvial history in several regions, both in large drainage systems such as River Rhine (e.g., WALLINGA et al. 2004, 2007; BUSSCHERS et al. 2005; SCHOKKER et al. 2005), Loire (Colls et al. 2001) and Mississippi (RITTENOUR et al. 2005) as well as small scale catchments, for example, in Lincolnshire, England (BRIANT et al. 2004) or the Klip River, South Africa (RODNIGHT et al. 2006). Besides work in the middle latitudes, luminescence dating has also frequently been applied to date fluvial dynamics in the semi-arid subtropics in regions such as the Chad Basin (GUMNIOR & PREUSSER 2007), Death Valley (SOHN et al. 2007) and, in particular, on the Indian Subcontinent (e.g., JUYAL et al. 2000; JAIN & TANDON 2003).

7.3.4 Hillslope deposits

The term hillslope deposits describes sediments that are eroded from and transported along hillslopes by running water or gravity and that usually form wedge-shaped deposits on the foot-slope. Such deposits are often referred to as colluvium although the term is inconsistently used by different researchers. First successful studies on dating colluvial samples were carried out by FORMAN et al. (1988) on colluvium from the US using TL. Later, WINTLE et al. (1993) and BOTHA et al. (1994) applied IRSL dating to sediments from South Africa. Detailed reviews on dating hillslope deposits are provided by LANG et al. (1999) and FUCHS & LANG (2008).

The major limitation in dating hillslope deposits is incomplete bleaching of the luminescence signal, due to the fact that several processes are

involved in the formation of hillslope deposits such as soil erosion (ploughing, sheet and rill erosion), mass movements (sliding, falling) and soil creep (e.g. solifluction). Many of these processes cause downslope movement of sediment as a rather compact block such as in the case of sliding. Hence, with respect to daylight bleaching, only a small fraction of the sediment in the colluviation process (i.e. only the outer layer of sediment grain on a rigid block) will be exposed to daylight. If material is transported by water (e.g., sheet erosion), the intensity and spectral composition of the daylight is subdued by the usually high suspended load (DITLEFSEN 1992). Another problem is related to coagulation of grains that hampers sufficient zeroing of the luminescence signal, because the inner grains of an aggregate are shielded from daylight exposure (LANG & WAGNER 1996). Furthermore, due to the usually rather small catchments of colluvial systems sediment transport distance tends to be relatively small, decreasing the probability of efficient bleaching as the duration of light exposure will be relatively short.

Despite these shortcomings, there are many examples of successful luminescence dating of colluvium, which might be explained by two controlling factors. Firstly, hillslope deposits are usually repeatedly reworked, which increases the probability of daylight exposure. Secondly, bioturbation and mechanical processes in the soil as well as cultivation ensure that mineral grains are frequently exposed to daylight before the sediment grains are eroded and transported. Nevertheless, investigating the degree of bleaching prior to deposition, as described in section 6.1, is a fundamental requirement in dating colluvial deposits.

Although luminescence dating of hillslope deposits is rather challenging, it has been applied to a variety of case studies worldwide. The first systematic study by WINTLE et al. (1993) and BOTHA et al. (1994) correlated sediments and soils with dryer or wetter climatic periods of the Late Quaternary in South Africa and describe in detail the complexity of slope evolution processes. Similar studies have been car-

ried out by, for example, CLARKE et al. (2003) in the UK and ERIKSSON et al. (2000) in Tanzania. Another application of dating hillslope deposits was in the context of palaeoseismicity research as vertical displacement along faults scarp leads to its erosional degradation. This potential was first explored by PORAT et al. (1996) on colluvial sediments associated with fault scarp activities for estimating seismic hazards. These authors established an earthquake chronology with four large earthquakes for the period 37 – 14 ka and five smaller earthquakes more recently. PORAT et al. (1997) confirmed the suitability of luminescence dating in palaeoseismic research but PORAT et al. (2001) found a slope-face dependency for the bleaching degree of colluvial sediments, with colluvial sediments from south-facing scarps being better bleached than colluvium derived from north-facing scarps. Studies in a similar context have been provided by LU et al. (2002) for China and FATTAHI et al. (2006) for Iran.

Increased sediment transport down-slope can also be induced by human activity, in particular during the Holocene. Widespread clearance of woodlands and associated soil erosion typically causes increased erosion along hillslopes, for example, when humans established permanent settlements and agriculture. Among the first studies was that by LANG (1994) who investigated colluvial sediments derived from a loess-covered landscape in southwest Germany, and this was followed by similar studies (e.g., KADEREIT et al. 2002). A frequency distribution of IRSL ages from the area revealed that increased colluviation coincides with phases of higher population density and show that the intensity of farming activities is the main trigger for soil erosion (LANG 2003). In a study in Greece, FUCHS et al. (2004) and FUCHS & WAGNER (2005) applied the SAR protocol to the quartz coarse grain fraction for reconstructing Holocene soil erosion and to elucidate the interaction of humans and environment since Neolithic times. Colluviation strongly fluctuated in the course of the Holocene, with a sharp increase during the Early Neolithic and the onset of agricultural activities. Further periods

of increased deposition of hillslope sediments are the Middle and Late Bronze Age, the Roman period and the period since the sixteenth century AD (FUCHS 2007). Similar studies have been carried out, for example, in Romania (KADEREIT et al. 2006) and in Belgium (ROMMENS et al. 2007)

7.3.5 Lacustrine sediments

Relatively few studies have been reported on attempts to date lacustrine sediments, although such deposits are important archives of past environmental change and hence relevant for reconstructing Quaternary climate history. Early work by KRONBORG (1983) and BERGER (1990) studied the amount of resetting of TL in modern sediments and on dating deposits of known age. Both studies have highlighted the potential as well as the limitations of TL. With the introduction of OSL new dimensions were added to the dating of water-lain sediment due to the higher bleachability of the optical signal (HUNTLEY et al. 1985). DITLEFSEN (1992) experimentally investigated the bleaching of sediment in suspension demonstrating that the optical signal is almost completely bleached in dilute suspensions within a few hours, little bleaching has been observed in dense suspensions. KRAUSE et al. (1997) used IRSL to date lake deposits from Antarctica and compared four different emission bands (280, 330, 410, 560 nm), with the inconsistent results of palaeodose (D_e) measurements being attributed to different bleaching characteristics of the different emission bands.

Late Glacial to Holocene sediments from Lake Holzmaar, Germany, gave IRSL ages consistent with varve chronology for samples with low concentrations of biogenic material. Inaccurate ages obtained for organic rich samples are explained by problems with dose rate determination (LANG & ZOLITSCHKA 2001). THOMAS et al. (2003) applied different approaches to date lacustrine deposits from Greece revealing an underestimation of polymineral fine grain IRSL and post-IR OSL UV emissions compared to quartz OSL ages and radiocarbon chronology.

It was demonstrated that the underestimation of IRSL ages was due to anomalous fading that was observed in storage tests. However, the presence of fading is probably explained by using IRSL UV emissions or by a specific geological setting at that particular site since other studies on dating lacustrine sediments using blue emissions have provided correct age estimates (DORAN et al. 1999; Wolfe et al. 2000; BERGER & DORAN 2001; BERGER et al. 2004). VANDERGOES et al. (2005) dated a 150 000 year old lake record from Okarito Pakihi, New Zealand and got IRSL and post-IR OSL ages in agreement with the results of radiocarbon dating, tephra chronology and the chronological time frame deduced from comparison with pollen records from ocean sediment. JUSCHUS et al. (2007) found SAR IRSL ages consistent with the age model proposed from several proxies tuned to regional insolation.

While all the examples given above are from humid regions in the middle to high latitudes, a few studies have used luminescence methodology to date ancient lake deposits from arid zones. The particular advantages of luminescence are that it circumvents contamination problems that often affect radiocarbon in arid environments and extends the dating range beyond the few 10 ka possible with radiocarbon. However, only very few studies have utilised this potential so far, in particular RADIES et al. (2005) who dated lake deposits of the Early Holocene humid period in Oman and ARMITAGE et al. (2007) who demonstrated that a lake occupied the Fazzan Basin, Libyan Sahara, during the Early Holocene, the Last Interglacial and probably during Marine Isotope Stage (MIS) 11.

7.3.6 Proglacial deposits

Some of the first attempts to date glaciofluvial deposits were carried out by GEMMELL (1985, 1994) who investigated the resetting of the luminescence signal in modern samples. MEJDAHL (1991) compared TL and radiocarbon ages of Late Glacial sediments applying different bleaching techniques. BERGER & EAST-

ERBROOK (1993) and BERGER & EYLES (1994) dated glaciogenic and water-lain sediments from the Western Washington, British Columbia and Toronto area. HÜTT & JUNGNER (1992) compared TL and IRSL dating of Late Glacial sediments but their results were overestimated due to partial bleaching. DULLER (1994) investigated the bleaching characteristics of glaciofluvial feldspars and identified two different types of partial bleaching: one type showing grains that were incompletely bleached to the same amount (type A) and the other type showing a mixture of grains containing a majority of completely bleached grains, together with some insufficiently bleached grains (type B). He concluded that successful IRSL dating of glaciogenic sediments that were deposited only a few kilometres in front of the ice sheet is unlikely (DULLER et al. 1995). Further investigations compared the resetting of TL, IRSL and OSL signals in modern and ancient fluvial and glaciofluvial sediments from Switzerland and Northern Germany (PREUSSER 1999c). Dating results reveal that the sediments from Switzerland were completely bleached while those from Northern Germany were not, for most samples investigated. These results were additionally supported by further investigations of PREUSSER (2003) and PREUSSER et al. (2003, 2005a). Dating glaciofluvial deposits from New Zealand showed problems due to low luminescence sensitivity of the quartz grains (PREUSSER et al. 2005b, 2006). Beside the problem of insufficient resetting prior to deposition, which leads to age overestimation, the influence of 'thermal transfer' as reported by RHODES & BAILEY (1997) causes an overestimation of the expected age on the investigated sediments. OSL dating of glaciogenic deposits from Northern Eurasia were presented by LARSEN et al. (1999) and SVENDSEN et al. (2004) as well as FORMAN (1999), FORMAN et al. (1999, 2002) and MANGERUD et al. (2001) implicating glacier advances during the Late Saalian, the Early Weichselian, the Middle Weichselian and the Late Weichselian. Optical dating of proglacial and glaciofluvial sediments from inneralpine valleys and the Alpine Foreland provided dif-

ferent problems with low quartz luminescence sensitivity and partial bleaching (KLASEN et al. 2006, 2007). While samples from the inneralpine valleys can be assigned to bleaching type B (DULLER 1994), incompletely bleached samples from the Alpine foreland consist of grains being reset to the same amount and are therefore assigned to type A (DULLER 1994). For these samples, calculating a mean palaeodose is challenging, and the methods of OLLEY et al. (1998) and GALBRAITH et al. (1999) which extract individual palaeodose values out of a positively skewed distribution, failed due to the fact that distributions are Gaussian. This is supported by investigations of FUCHS et al. (2007) who reported that partially bleached samples can also be represented by normal distributions. Therefore, frequency distributions should be regarded very carefully when used for the interpretation of bleaching characteristics. Additionally, quartz samples from the northern Alpine Foreland appear not to contain any 'fast component' within the OSL-signal (cf., BULUR et al. 2000). Applying SAR- methodology to these samples resulted in an underestimation of the age of the sediment as this protocol is designed for samples dominated by a 'fast component' (KLASEN 2007). DULLER (2006) investigated glaciogenic deposits from Chile and Scotland using single grain optical dating, and showed that grains were commonly affected by low luminescence sensitivity. Nevertheless, DULLER (2006) concluded that the single grain methodology is the method of choice to detect partial bleaching. Despite this, PREUSSER et al. (2007) determined OSL ages consistent with radiocarbon ages for proglacial sediments from Switzerland using small aliquots.

7.3.7 Coastal (water-lain) deposits

The pioneering study on dating coastal deposits was carried out by BALESCU et al. (1991) on raised interglacial beaches from the Channel Region (France, Belgium). These authors used TL from feldspars detected in the UV band and found residual ages of no more than a few ka in modern deposits. However, TL ages of older

deposits were underestimated by up to 40 %. BALESCU & LAMOTHE (1992) later demonstrated that ages consistent with stratigraphy are obtained when blue TL emissions are used for dating. The same authors calculated a minimum age of 271 ± 36 ka for the marine Herzelee Formation in northern France that is interpreted as being of Holsteinian age (BALESCU & LAMOTHE 1993). The first applications of IRSL to coastal sediments reveal a good consistency with TL and independent age control (BALESCU & LAMOTHE 1994; BALESCU et al. 1997). In another early study, MAUZ et al. (1997) used TL from quartz separates to date littoral deposits from southern Italy revealing that eustatic oscillations deduced from a deep ocean sediment proxy are not completely recorded within the inner shelf deposits. MAUZ & HASLER (2000) found evidence from both feldspar IRSL and quartz TL dating for relatively high sea-level (-15 m) during early MIS 3 (ca. 50 ka ago) in the Mediterranean although this is inconsistent with that from the Huon Peninsular of New Guinea.

While the studies summarised above used multiple-aliquot approaches, more recent studies have used single-aliquot methodology. RICHARDSON (2001) investigated the degree of resetting in modern intertidal deposits from England and Wales revealing substantial residual ages in many of the samples, especially for those deposited by turbid water suspension. The author also highlights the potential problem associated with estimating the correct average water content during burial for samples from such environments. HONG et al. (2003), however, found clear evidence for complete bleaching of sub-modern tidal-flat deposits from Korea and report ages between 40-120 a. Excellent consistency of OSL and ^{210}Pb as well as ^{137}Cs dating of sub-modern estuarine sediments from Denmark has been shown by MADSEN et al. (2005, 2007) with OSL ages ranging from a few years to about 1000 a. The age of near-surface samples in one core is 9 ± 3 a and 7 ± 4 a in another, indicating that partial bleaching does not apparently have any major effect in such environments. The applicability

of OSL for dating young tidal flat deposits has also been demonstrated for the Frisian Coast, Germany, where age control is available from radiocarbon dating of peat (MAUZ & BUNGENSTOCK 2007). ZANDER et al. (2007) showed that Holocene coastal sediments from the Persian Gulf that are affected by radioactive disequilibrium in the Uranium decay chain due to high amounts of calcareous material (shell debris etc.) can still be correctly dated using OSL. MURRAY & FUNDER (2003) dated several samples from an Eemian site in Denmark to test the accuracy of OSL dating. Although the mean age of the individual samples is 119 ± 6 ka including systematic uncertainties and, hence, in acceptable agreement with the expected age of the sediment (128–132 ka), there is a suggestion that OSL may systematically underestimate the true age by about 10 % percent. This question has been addressed by MURRAY et al. (2007) who dated an Eemian site in northern Russia consisting of foreshore marine deposits with an expected age of about 130 ka. The mean of 16 OSL ages determined for this deposit was 112 ± 7 ka (including systematic uncertainties) showing a similar underestimation to that of the site in Denmark. The authors discuss several possible explanations for the apparent underestimation of OSL ages, among which are changes in sensitivity of the natural signal at the beginning of the SAR procedure, which is not corrected for, contamination of the stable fast component of the OSL signal by less-stable components as well as open system radionuclide behaviour and inaccurate estimates of long-term water content.

7.3.8 Deep sea sediments

One of the first applications of TL dating on sediments was on deep ocean deposits (WINTLE & HUNTLEY 1979, 1980; BERGER et al. 1984). More recently, STOKES et al. (2003) used SAR methodology on silt-size quartz gathered from two cores from the Arabian Sea where age control is provided by radiocarbon, tephrochronology and a correlative marine-proxy age model. The investigated silt is interpreted as dust input

from the Arabian Peninsula transferred by aeolian processes into the ocean. In this study, particular attention was paid to radioactive disequilibria that are a major potential error source in dating deep ocean sediments. The nine OSL ages obtained by STOKES et al. (2003) are, with one exception, in good agreement with independent age control and demonstrate the potential of OSL to date deep ocean sediments. OLLEY et al. (2004) applied sand-size single-grain quartz for dating a core off the NW Australian coast, and these authors also found good consistency of OSL with radiocarbon ages but highlight that partial bleaching may be important, even for grains that underwent long distance aeolian transport. BERGER (2006) carried out a test study on a variety of cores from the Arctic Ocean and found varying levels of signal resetting in different regions. Using a new technique enabled correct dates for the Late Holocene deposits to be established. This study demonstrated the potential but also highlighted the problems associated with dating samples from the Arctic Ocean, which are probably related to sediment input through glacial transport (e.g., ice raft). KORTEKAAS et al. (2007) dated a core covering the last 15 ka from the Arkona Basin, Baltic Sea, using fine-sand quartz (63–100 μm). These authors observed a good agreement between OSL and radiocarbon age from bivalve mollusc shells, although radiocarbon ages on bulk sediment samples were between 1000 and 3000 a higher than OSL and bivalve ages. The incorporation of reworked material in the bulk samples is suggested as the cause for overestimation.

8 Summary and outlook

Luminescence dating allows the dating of mineral crystallisation, heating to a few 100°C or the last daylight exposure of sediment grains. It covers the time range from a few years or decades up to several hundred-thousand years. Luminescence dating represents a suite of related techniques, all of which provide an estimate of radiation history due to accumulation of trapped charges in mineral lattices. During the last de-

cade, several methods have been developed that have considerably improved the reliability and precision of luminescence dating. It is highly recommended that information with regard to tests of quality assurance is included when publishing the results of luminescence dating.

Recent research is focusing on improved and innovative procedures of both palaeodose and dose rate determination. For palaeodose determination, it is necessary to better understand the sources of variation observed for individual palaeodose estimates determined for the same sample, especially to quantify the effect of microdosimetry. Furthermore, it is expected that it may be possible to extend the upper dating limit using approaches such as thermally-transferred OSL in quartz or by using a more stable luminescence emission, such as the red emission band of feldspars. With regard to dose rate determination, improvements in the handling of radioactive disequilibria and variation in sediment moisture may help to further improve the precision and accuracy of luminescence dating.

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Electron spin resonance (ESR) dating of Quaternary materials

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Abstract: ESR dating has become an efficient tool in earth sciences for geochronological studies on different kinds of littoral deposits (coral reefs terraces, beach ridge systems, aeolianites) during the last ten years. Improvements in annual dose rate (D') estimation and the newly developed approach for equivalent dose (D_E) determination ($D_E - D_{max}$ plot procedure) increase the precision of ESR dating of Holocene and Pleistocene corals as well as marine and terrestrial mollusc shells. This is strongly supported by the comparison of ESR dating results with other numeric dating methods such as radiocarbon and TIMS Uranium series analysis (TIMS $^{230}\text{Th}/^{234}\text{U}$). The latter is the main focus of this paper. The uncertainties associated in ESR dating of Holocene corals coincide with the variability of ^{14}C ages caused by the marine reservoir effect. The dating of Pleistocene corals permits the differentiation between the main marine isotope stages (MIS) 5, 7, 9, 11 and 13 as well as between sub-stages $5e_{3/2}$ and $5e_1$, $5c$, and $5a_2$ and $5a_1$. The average error range when dating corals is between 5 to 8%. Furthermore, ESR dating of marine and terrestrial mollusc shells has yielded some promising results and permits the differentiation between the interglacial MIS 1, 5, 7 and 9 with an average dating error range of 10 to 15%.

ESR dating of quartz is another promising dating technique for Quaternary and even Neogene geological formations. The presence of quartz in volcanic rocks, tephra, fault gouge and sediments (heated or unheated) allows determining the last time of heating, fault movement or sunlight exposure. Although challenged by several experimental issues, ESR dating of quartz is often the only method able to produce numerical ages for older formations.

ESR has also been applied to a wide variety of other materials such as foraminifera, speleothems, travertines, calcretes and tooth enamel. The most common and reliable application is the ESR dating of mammal teeth, which becomes in conjunction with laser ablation U-series dating, an important method for determining the age of archaeological sites beyond the time range of the ^{14}C dating method back to about 200 to 300 ka.

[Elektronen Spin Resonanz (ESR)-Datierung quartärer Materialien]

Kurzfassung: ESR hat sich im letzten Jahrzehnt bei der Datierung verschiedenster littoraler Ablagerungen (Korallenriffterrassen, Strandwallsysteme, Dünen) als effizientes Datierungswerkzeug etabliert. Verbesserungen in der Bestimmung der jährlichen Dosisleistung (D') und ein neu entwickelter Ansatz zur Bestimmung der Äquivalenten Dosis ($D_E - D_{max}$ Verfahren) haben die Präzision der ESR-Datierung sowohl an holozänen und pleistozänen Korallen als auch an marinen und terrestrischen Molluskenschalen verbessert. Dies wurde durch den Vergleich mit anderen numerischen Datierungsverfahren wie Radiokohlenstoff und TIMS-Uranserien-Analyse (TIMS $^{230}\text{Th}/^{234}\text{U}$) unterstützt. Der Vergleich mit letzterer Methode steht im Fokus dieses Artikels. Die mit der ESR-Methode verbundenen Ungenauigkeiten bei der Datierung holozäner Korallen liegt in der Größenordnung der Variabilität von ^{14}C -Altern, die durch den marinen Reservoir-Effekt bedingt ist. Die Datierung pleistozäner Korallen erlaubt die Differenzierung der wichtigen marinen Isotopenstadien (MIS) 5,

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7, 9, 11 und 13 sowie der Untereinheiten $5e_{3/2}$ und $5e_1$, $5c$ und $5a_1$ und $5a_2$. Der durchschnittliche Fehler bei der Datierung von Korallen liegt zwischen 5 bis 8%. Weiterhin hat die Datierung mariner und terrestrischer Mollusken mittels ESR viel versprechende Resultate geliefert, die eine Differenzierung der Interglaziale MIS 1, 5, 7 und 9 ermöglichen, bei einem Fehler von 10-15%.

Die ESR Datierung von Quarz ist eine weitere viel versprechende Datierungstechnik für quartäre und sogar neogene geologische Formationen. Das Vorkommen von Quarz in vulkanischen Gesteinen, Tephren, Störungen und Sedimenten (thermisch beeinflusst und unbeeinflusst) ermöglicht die zeitliche Bestimmung des letzten Zeitpunkts vor der Erhitzung, Störung oder der Aussetzung von Sonnenlicht. Obwohl durch einige experimentelle Ergebnisse angezweifelt, ist die ESR Datierung von Quarz die einzige Möglichkeit Altersdaten älterer Ablagerungen zu liefern.

ESR wurde auch bei einer Vielzahl anderer Materialien angewendet, wie zum Beispiel Foraminiferen, Speleothemen, Travertinen, Kalkkrusten und Zahnschmelz. Die gebräuchlichste und zuverlässigste Anwendung ist die ESR-Datierung von Mammutstoßzähnen. Im Zusammenspiel mit der Laser-Ablation Uranserien Datierung ist ESR eine wichtige Methode zur Altersbestimmung archäologischer Fundstätten jenseits der Bestimmungsgrenzen der Radiokohlenstoffmethode bis in den Bereich von 200 bis 300 ka.

Keywords: Electron spin resonance, dating, geochronology, Quaternary, littoral

1 Introduction

Although IKEYA (1975) introduced Electron Spin Resonance Spectroscopy (ESR) for dating stalagmites more than 30 years ago, the full potential of this relatively young method is still not fully utilised. Since then, the quality of ESR-spectrometers and the understanding of the structure and behaviour of the ESR signal used for dating have been significantly improved. Detailed overviews about the ESR dating method have been provided by IKEYA (1993), GRÜN (1989a, 1989b, 2007), RADTKE (1989), JONAS (1997) and RINK (1997).

This method is used for a wide variety of materials with most reliable applications on corals, mollusc shells, quartz, foraminifera, speleothems and teeth. In this text, the potential and present restrictions of ESR dating of Quaternary coral, mollusc shells and quartz is illustrated for selected sites and a short review is presented about the relevance of ESR for dating other carbonates (foraminifera, speleothems) and tooth enamel. For further details about problems and further applications of ESR dating including more literature, see e.g. GRÜN (2007), BLACKWELL (2006) and RINK (1997).

Regardless of the recent methodological improvements in ESR and its frequent use for dating corals, mollusc shells, quartz and

teeth, there is still a huge potential for further development of the method for these and other materials (e.g. SKINNER 2000). Similar to all other methods of age determination, ESR dating is confronted by specific methodological problems that cannot be considered in error calculation. This means, as applies for most analytic methods, that high precision does not automatically guarantee the accuracy of an age estimate. The latter is influenced by different geological factors that cannot be quantified and are hence not part of the age and error calculation. In ESR dating, these are mainly diagenetic alterations of the dated material, incomplete resetting of the ESR signal and up-take or loss of radioactive elements (U, Th, K) (Fig. 1). With regard to these problems, proving the reliability of new dating approaches by independent age control is essential. In the context of the ESR method, this is possible using radiocarbon or $^{230}\text{Th}/^{234}\text{U}$ dating for corals and mollusc shells or luminescence, palaeomagnetism or $^{40}\text{Ar}/^{39}\text{Ar}$ for dating quartz.

2 Methodology

The ESR dating method is one of several radiation exposure methods based on radiation dosimetry such as thermoluminescence (TL), optically stimulated luminescence (OSL) and radioluminescence (RL). All these methods

use the phenomenon that common minerals act as natural dosimeters. The radiation causes charge (electrons, free radicals) to be trapped at defects in the crystal lattice of a wide range of minerals such as aragonite, calcite and quartz. The amount of trapped charge accumulation increases with time and can be quantified by the ESR measurement.

2.1 Nature of the ESR signal and its quantification

The process of trapping charge results from the interaction of naturally occurring alpha, beta and gamma, and, to some minor extent, cosmic radiation (D'_{cos} , cosmic dose rate) with matter. The first consists of internal radiation from

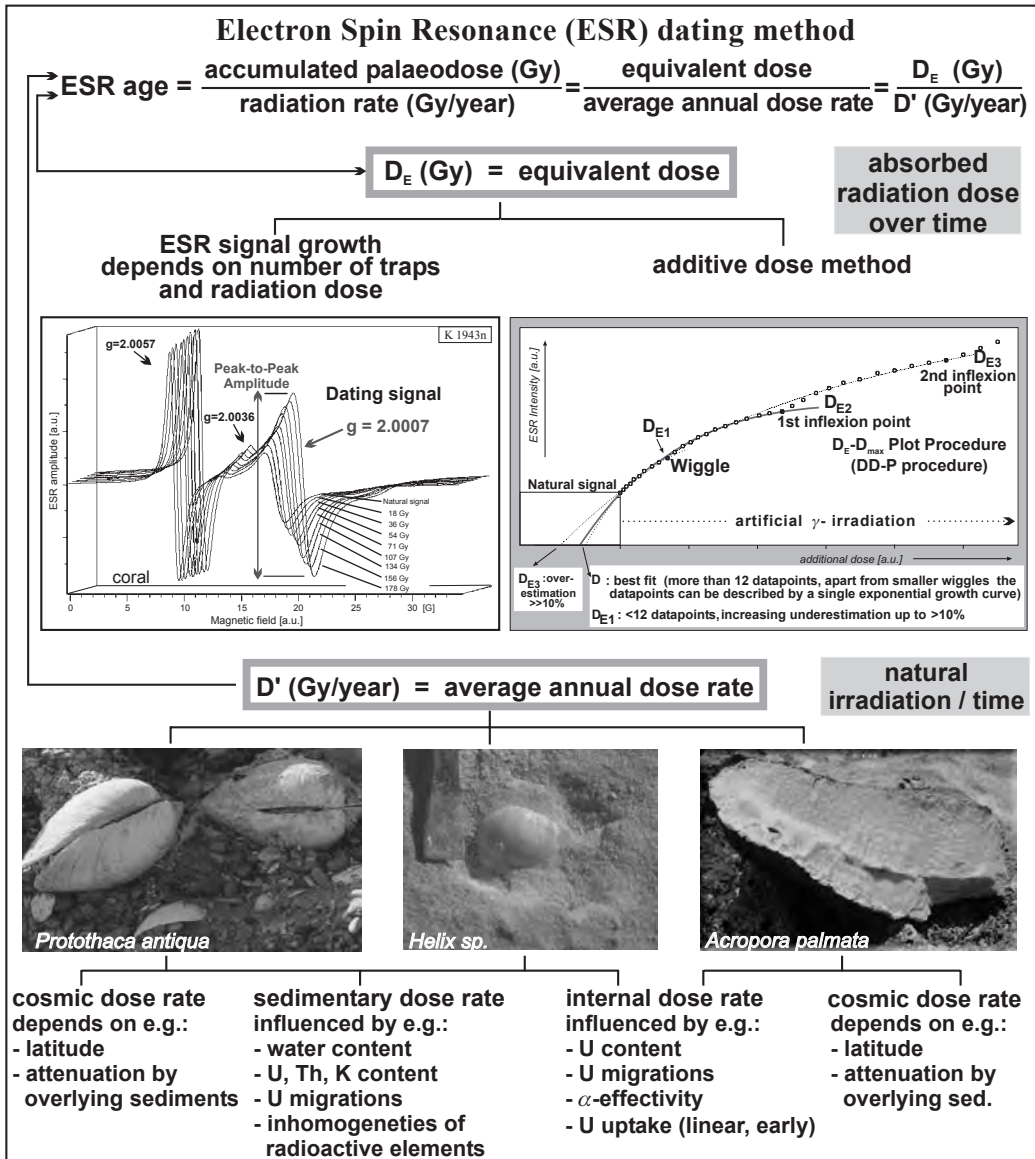
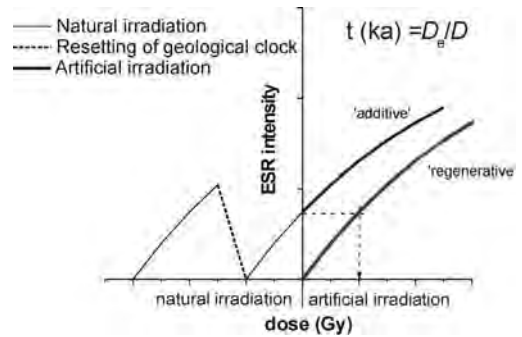


Fig. 1: Generalised principle of ESR dating of aragonitic coral and mollusc shells

Abb. 1: Generalisiertes Schema der ESR-Datierung von Korallen und Muschelschalen.

the mineral itself (D'_{int} , internal dose rate: i.e. Uranium and daughter isotopes, to some extent additionally Th and K in quartz) as well as radiation from the surrounding sediment (D'_{ext} , external dose rate mainly from Uranium and Thorium decay chains and Potassium). Such ionising radiation causes the activation of electrons to an excited energy level. Charge defects in the crystal lattice, so-called traps, capture part of the excited electrons in the band gap. A detailed description of the underlying physical processes is provided by e.g. GRÜN (1989a, 1989b, 2007), JONAS (1997) and RINK (1997). The amplitude of the ESR signal represents the amount of unpaired electrons at lattice defects (traps). Each material investigated has a characteristic ESR spectrum that may consist of one or several single signals, but not all the individual signals are suitable for dating. Suitable are only ESR signals that are both sensitive to radiation and thermally stable at the prevailing temperature that occurred during deposition and burial. The ESR signal at $g = 2.0007$ (Fig. 1) is most suitable for dating aragonitic mollusc shells and corals (e.g. RADTKE & GRÜN 1988, WALTHER et al. 1992, SCHELLMANN & RADTKE 1999, 2001). A basic principle in dating biogenic material is that the ESR signal starts to increase after the shell, tooth enamel or coral has been formed.

For ESR dating of quartz, a prerequisite is the existence of a resetting mechanism in nature to zero the ESR signal (and thus the geological clock). In order for the method to be reliable, any pre-existing ESR signals must be erased prior to the event to be dated (Fig. 2). The zeroing process responsible for resetting the geological clock is dependent on the geological context of the quartz mineral. The specific behaviour of ESR centres in quartz allows to determine the moment of the last heating (volcanic rocks, tephra, heated sediments), the moment of the last fault movement (quartz in fault gouges exposed to shearing) and the last exposure to sunlight (sedimentary quartz). The accuracy of the ESR age is dependent on the completeness of the zeroing process. Incomplete signal zeroing will inevitably lead to age



overestimates, unless suitable tests can reliably determine the degree of zeroing that may have occurred, through which this can be accounted for. Several ESR centres in quartz have shown to be potential dosimeters of ionising radiation

Abb. 2: Generalisiertes Prinzip der ESR-Datierung von Quarz. Die grundlegende Annahme der ESR-Datierung ist, dass das ESR-Signal vor dem zu datierenden Ereignis auf Null gesetzt wird (Zurückstellen der geologischen Uhr). Danach nimmt das ESR-Signal, aufgrund natürlicher Strahlung, bis zum Zeitpunkt der Probennahme wieder zu (linke Seite der ESR Intensitätsachse). Generell gibt es zwei Methode die Akkumulierte oder Äquivalent Dosis (D_E) zu bestimmen. Bei der Additiven Dosis Methode wird auf das natürliche Signal eine künstliche Dosis hinzu gegeben. Die Äquivalent Dosis wird dann durch Abtragen der Wachstumskurve auf der X-Achse bestimmt. Bei der Regenerativen Dosis Methode wird das Signal zuerst auf Null gesetzt und die natürliche Intensität wird auf die regenerative Wachstumskurve projiziert, um die Äquivalent Dosis zu erhalten.

overestimates, unless suitable tests can reliably determine the degree of zeroing that may have occurred, through which this can be accounted for.

Several ESR centres in quartz have shown to be potential dosimeters of ionising radiation

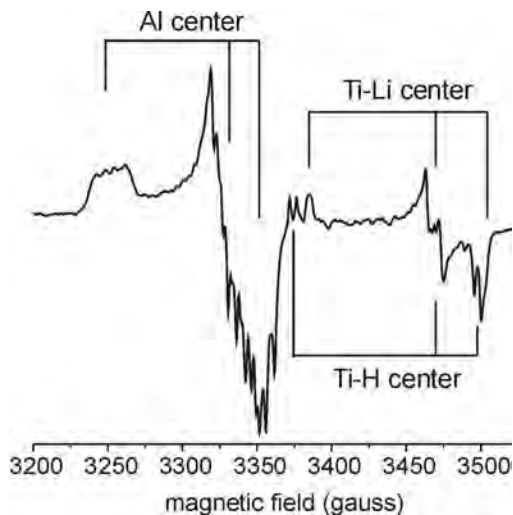


Fig. 3: ESR spectrum of crystalline quartz grains (100-200 μm , 300 mg) at 100 K (Australian sedimentary quartz). Microwave absorption patterns are indicated for the Al-center, Ti-Li-center and Ti-H-center, according to the 3 principal g-values (vertical lines).

Abb. 3: ESR-Spektrum kristalliner Quarzkörner (100-200 μm , 300 μm) bei 100 K (sedimentärer Quarz; Australien). Mikrowellenabsorptionsmuster sind für die Al-, Ti-Li- und Ti-H-Zentren angegeben, bezogen auf die drei grundlegenden g-Werte (vertikale Linien).

the equivalent of the palaeodose is determined by a so-called additive dose response curve. For this, the sample is irradiated using artificial β - or γ -sources and an individual dose response curve is constructed for each sample. By extrapolation on the x-axis, D_E is calculated (Fig. 1). The equivalent dose of quartz can also be determined using the regenerative dose method, owing to the regeneration characteristics of the paramagnetic defects in the mineral. Following thermal annealing (heating) or optical bleaching, the ESR signal is regenerated with an artificial radioactive source, and the dose is estimated by interpolation of the natural ESR intensity (Fig. 2).

2.2 Dose rate determination

The cosmic dose rate is related to the depth

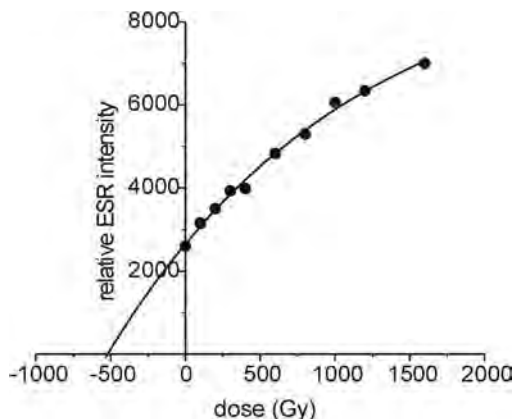


Fig. 4: ESR dose response curve of the Al-center from an aeolian sample (NWB1, Murray Basin, Australia; unpublished results). The data points are fitted to a rising exponential (GRÜN 1989). Positive values refer to the artificially added doses, while negative values refer to the past irradiation dose. Assuming a dose rate of about 1 Gy ka^{-1} , which is not uncommon in sedimentary contexts, the dose range covered in this example would equal more than 2 million years.

Abb. 4: ESR-Dosisaufbaukurve für das Al-Zentrum gemessen an einem äolischen Sediment (NWB1, Murray Basin, Australien, unveröffentlichte Daten). Die Datenpunkte sind an einen steigenden Exponenten angepasst (GRÜN 1989). Positive Werte beziehen sich auf die künstlich zugegebene Strahlung, wohingegen negative Werte die in der Vergangenheit akkumulierte Dosis repräsentieren. Unter Annahme einer Dosisleistung von ungefähr 1 Gy ka^{-1} , was für sedimentäre Ablagerungen nicht ungewöhnlich ist, deckt die akkumulierte Dosis in diesem Beispiel eine Spanne von mehr als 2 Millionen Jahren ab.

of the sample below surface, latitude and elevation. Further corrections to burial depth may be needed where nearby obstructions or drop-offs in the land surface occur: these are not accounted for by the tables of PRESCOTT & HUTTON (1994). The radioactivity of the surrounding material is either measured in the field using a portable gamma spectrometer or calculated via the determination of the concentration of Potassium, Thorium and

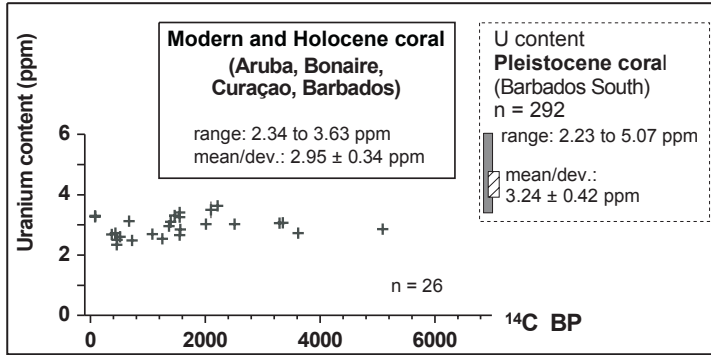


Fig. 5: Uranium content (ppm) of modern, Holocene and Pleistocene corals (slightly modified from SCHELLMANN & RADTKE 2003).

Abb. 5: Uran-Gehalte (ppm) modernen, holozänen und pleistozänen Korallen (leicht verändert nach SCHELLMANN & RADTKE 2003).

Uranium in the sample. For aragonitic mollusc shells and corals, the internal dose rate is caused almost completely by Uranium. If possible, the Uranium content of each sample should be verified by repeated measurements. In contrast, the internal alpha dose rate in quartz is usually very small in comparison with the external dose rate, allowing it to be neglected in the final age calculation. Most sub-modern, Holocene and Pleistocene corals have Uranium contents of about 3.0 to 3.2 ppm (Fig. 5). It is hence concluded that corals absorb such amounts of Uranium from seawater during their life-time or shortly after their death. Extremely high (up to 5 ppm) and low (below 2.5 ppm) Uranium concentrations are exceptional and at the moment, it can only be speculated about their origin.

For the calculation of ESR ages of corals, an early Uranium up-take and hence a relatively high radiation level from the very beginning can be assumed. This is of importance as ESR ages that are calculated based on an early Uranium up-take model will result in much lower age estimates compared to ages calculated based on the assumption of a linear Uranium uptake. In contrast to this, modern mollusc shells have extremely low Uranium contents of 0.1 to 0.2 ppm (max. 0.7 ppm). Only after an age of more than 2500 yr, do mollusc shells show significantly higher contents of more than 2 ppm Uranium (Fig. 6) and reach levels frequently observed in Pleistocene fossils. This implies that mollusc shells absorb most of the Uranium *post mortem*. However, this delayed up-take of Uranium is negligible for age calcu-

lation when dating Pleistocene mollusc shells. For Early to Mid Holocene mollusc shells, which have Uranium contents higher than 0.5 ppm, a delayed up-take of Uranium has to be considered when calculating the internal dose rate. As the exact *post-mortem* up-take of Uranium cannot be reconstructed, the true age will be between the ESR ages estimates calcu-

Table 1: Exemplarily calculation of the effect of different Uranium contents on the ESR age of a Holocene mollusc shell at constant external dose rate. Shown are the age differences that result from linear Uranium up-take and early Uranium up-take, respectively.

Tab. 1: Exemplarische Berechnung zur Verdeutlichung des Einflusses unterschiedlicher Uran-Gehalte auf die ESR-Alter holozäner Muschelschalen unter der Annahme einer konstanten externen Dosisleistung. Sichtbar werden die Altersunterschiede, die aus einer linearen bzw. einer früheren Uraufnahme resultieren.

U content (ppm)	linear U-uptake model (ESR age diff. to early U-uptake model)
<0.6	same age
0.7	+140 yrs.
1.0	+200 yrs.
1.5	+270 yrs.
2.0	+350 yrs.
2.5	+400 yrs.
3.0	+460 yrs.
3.5	+510 yrs.
4.0	+560 yrs.
4.5	+600 yrs.
5.0	+640 yrs.

lated for linear and early Uranium up-take. For Late Holocene mollusc shells with Uranium contents > 0.5 ppm, linear up-take represents probably the most likely scenario. A detailed discussion of Uranium up-take is provided by JONAS (1997) and RINK (1997). Depending on the contribution of internal dose (= Uranium content of mollusc shell) to the total dose rate, differences in age between the two models will be up to 1000 yr (and more) for samples having a high Uranium content (>5 ppm). For Uranium contents below 0.6 ppm, both models usually result in age estimates consistent within error. Table 1 demonstrates the effect of Uranium up-take on the ESR age of a mid-Holocene mollusc shell.

There are several other potential sources of error associated with dose rate determination

that may lead to incorrect ESR ages (Fig. 1). Among these is a change in past sediment water content that can hardly be quantified and may have caused variations in external dose rate. However, this can be accommodated by placing a very large error on the water content in the calculations, for example a value of up to 100% of the water content. Another potential problem can be radioactive disequilibria, i.e. the loss or gain of radioactive elements mainly from the Uranium decay chain (e.g. loss of soluble Uranium). Furthermore, calculation of dose rate for mollusc shells from heterogenous settings, i.e. poorly sorted sand and gravel, is much more insecure than for samples (e.g. land snail shells) from homogenous environments such as aeolianites. Only a few problems are associated with the dose rate determination of

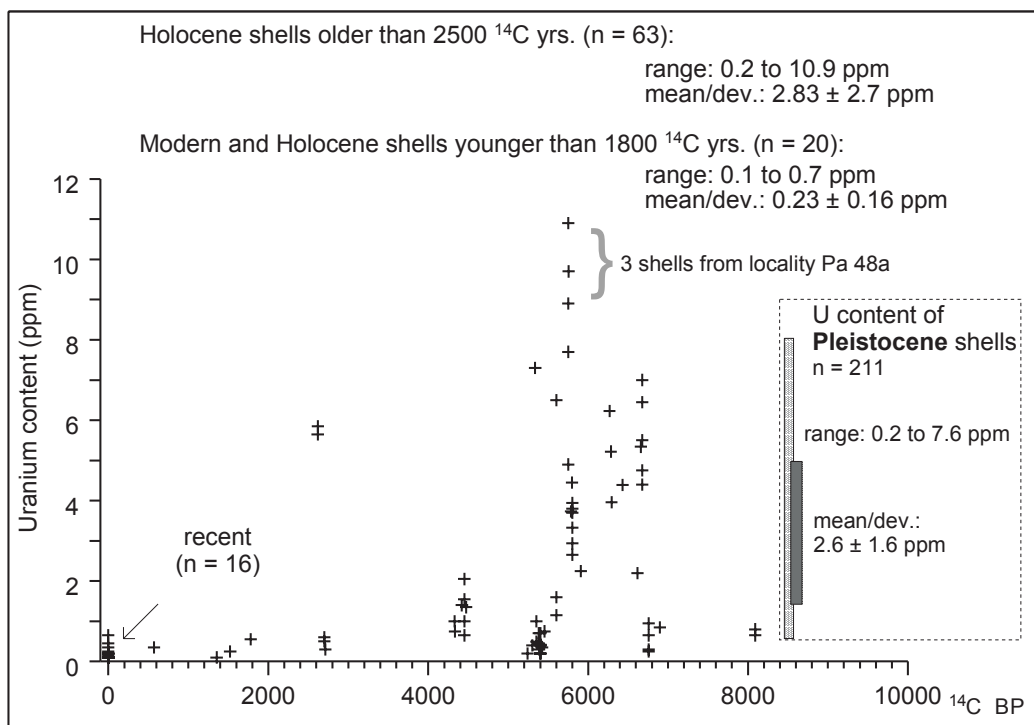


Fig. 6: Uranium content (ppm) of modern, Holocene and Pleistocene mollusc shells from SCHELLMANN & RADTKE (2007). In addition to the Uranium content of radiocarbon dated mollusc shells, values measured for *in situ* bivalve shells that were collected from the same horizons are included.

Abb. 6: Uran-Gehalte (ppm) modernen, holozänen und pleistozänen Muschelschalen (aus SCHELLMANN & RADTKE 2007). Zusätzlich zu den Uran-Gehalten der ^{14}C -datierten Muschelschalen, sind auch die Werte von den *in situ* im gleichen Horizont gefundenen bivalven Schalen angegeben.

corals since only cosmic radiation and internal Uranium are relevant for total dose rate (Fig. 1). The problems of changing water content in the surroundings of the sample and erosive or accumulative processes that may affect cosmic dose calculation have only minor effects.

Supplementary uncertainties in the dose rate for ESR dating of quartz are due to variations in water content and disequilibria in the decay chains of U and Th. Radioactive disequilibrium (i.e. a misbalance between parent and daughter nuclides) might occur if one (or more) of the Th- and/or U-chain members are lost or gained during burial, an effect that would be more pronounced in very permeable sediments. Especially, radionuclides of Rn (gas) and Ra (very leachable), which occur halfway in the U- and Th-chains, could induce disequilibrium. In the case of sediments, it is also possible that disequilibrium already existed at the time of deposition. In order to calculate an accurate dose rate, it is important to check whether any equilibrium exists and if so, whether this equilibrium has remained constant over time. However, in many dating studies, radioactive equilibrium is simply assumed.

A further error source in the calculation of ESR ages is the only poorly known so-called alpha-efficiency, which is also known as k-factor (see JONAS 1997 for details). This value describes the efficiency of α -particles to induce ESR signals compared to other kinds of radiation. GRÜN (1985) and GRÜN & KATZENBERGER (1994) experimentally determined by using a ^{241}Am α -source k-values between 0.07 and 0.10 for mollusc shells. For corals, RADTKE & GRÜN (1988), GRÜN et al. (1992) and MALMBERG & RADTKE (2000) determined k-values between 0.05 and 0.07. According to LYONS (1987, cit. in RADTKE & GRÜN 1988) the actual alpha-efficiency of particles from the Uranium decay chain is 20-30 % higher than the values determined using a monoenergetic artificial α -source. Following GRÜN (2007: 1509), the best k-values for molluscs are 0.07 ± 0.01 , for corals 0.06 ± 0.02 and for tooth enamel 0.13 ± 0.02 . The influence of uncertainties on the α -efficiency in quartz is re-

duced because the internal alpha dose rate can usually be neglected (cf. supra). Furthermore, the influence of external alpha rays (several 10 μm) is erased by sample preparation techniques using HF-solutions. As such, in many cases the total dose rate to quartz can be simplified to the sum of the external beta and gamma dose rate and the cosmic dose rate.

3 ESR dating of aragonitic coral and marine and terrestrial mollusc shells

Besides the problems in dose rate determination mentioned above, ESR dating of mollusc shells and corals is associated with another uncertainty that has yet not been quantified. This problem is related to the amplitude of the ESR signal of most mollusc shells, and also rarely for corals, which does not show simple exponential growth to saturation resulting from artificial gamma irradiation. Instead, so-called inflexion points (Fig. 1) have been observed at which signal growth increases suddenly. The physical nature of these inflexion points is yet only poorly understood. Most likely, it results from interference of the ESR dating signal with the so-called a-complex, which shows a relatively higher increase at higher doses and apparently individually disturbs the signal used for dating (KATZENBERGER & WILLEMS 1988, BARABAS et al. 1992). However, it is also possible that inflexion points are related to defects in the crystal lattice that are produced by gamma irradiation (GRÜN 1990). Attempts to eliminate inflexion points without alternating the ESR dating signal by changing the parameters of the ESR measurement or including thermal pre-treatments have not been successful so far (e.g. BRUMBY & YOSHIDA 1994a, HOFFMANN et al. 2001, MOLODKOV et al. 1998). Different preheat temperatures and durations can actually cause significant differences in D_E values and consequently different ESR ages (SCHELLMANN & RADTKE 2007). Discontinuous growth of the ESR signal can cause overestimation of calculated D_E of more than 10 % when too high artificial doses are used for construction of the dose response curve (Fig. 1). For determination

of D_E values, only the undisturbed, low-dose part of a dose response curve prior to reaching the first inflexion should be used. Only this part is dominated by the growth of the ESR dating signal and should reflect natural increase. This part of the growth curve can be described by a simple exponential saturation curve. The impact of inflexion points can be minimised by either using several dose points in the lower part of the dose response curve or by applying the standardised procedure of D_E - D_{max} -plots (DDP) (Fig. 1; SCHELLMANN & RADTKE 1999, 2001, 2003). Nevertheless, inflexion points are probably the main reason for age scatter observed in isochronal samples within mollusc bearing sediment layers. Additive dose response curves of corals, on the other hand, rarely show pronounced inflexion points, presumably the reason why ESR dating of isochronal corals scatter much less than molluscs (see below). The D_E values of all ESR ages mentioned here were determined using D_E - D_{max} plots. Prior to this, mollusc and coral samples were ground by hand and sieved to 125-250 μm . At least 20 aliquots with a weight of 0.2000 g were prepared and irradiated using a ^{60}Co -source (Centre of Nuclear Medicine, University of

Düsseldorf); dose rate between 0.8 and 2.5 Gy min^{-1}). The maximum irradiation dose was typically between two and three times D_E . Typical parameters on the ESR spectrometer were 10 or 25 mW microwave power, 0.5 or 1.0–1.2 G modulation amplitude, 41.9 s scan-time, 40–50 G scan width and 5 to 40 scans. All D_E values were determined using the programme „Fit-sim“ (version 1993) and ESR ages were calculated using the programmes „Data IV“ (version 1990) and „Data V.6“ (version 1999), written by Rainer Grün.

3.1 Comparing ESR, TIMS Th/U and radiocarbon dating results ages of mollusc shells from Holocene as well as Late and Middle Pleistocene littoral terraces of the Patagonian Atlantic coast

Since several early systematic studies (RADTKE et al. 1981, IKEYA & OMUHRA 1981), dating of molluscs is one of the most common applications of ESR dating and has been applied in several regions worldwide. Particularly typical for the example presented here, the Atlantic coast of Patagonia, is the phenomenon that the coarse littoral beach deposits frequently bear articulated mollusc shells (Photo 1). Such objects are very sensitive to movement and clearly indicate their *in situ* nature. This could be proven by radiocarbon dating of several bivalve mollusc shells found in one sediment layer (SCHELLMANN 1998, SCHELLMANN & RADTKE 2007). The dating of several *in situ* and isochronous mollusc shells (deposited within not more than a few decades) represents an ideal opportunity to test the accuracy of the ESR dating method. With this approach, it is possible to test the reproducibility of ESR dating with special regard to the dating of Pleistocene molluscs, where no other accurate dating methods are available.

Along the Patagonian Atlantic coast, there are several locations where Holocene and Pleistocene littoral deposits with articulated mollusc shells can be found at different elevations. The distribution of raised Pleistocene beach deposits in the Bay of Bustamante is shown in



Photo 1: Articulated mollusc shell (*Protothaca antiqua*) in Last Interglacial T3_[5] beach deposits near Bustamante, Patagonian Atlantic coast.

Foto 1: Muschelschalen von *Protothaca antiqua* in Lebendstellung in Strandablagerungen des letzten Interglazials T3_[5] an der patagonischen Atlantikküste nahe Bustamante.

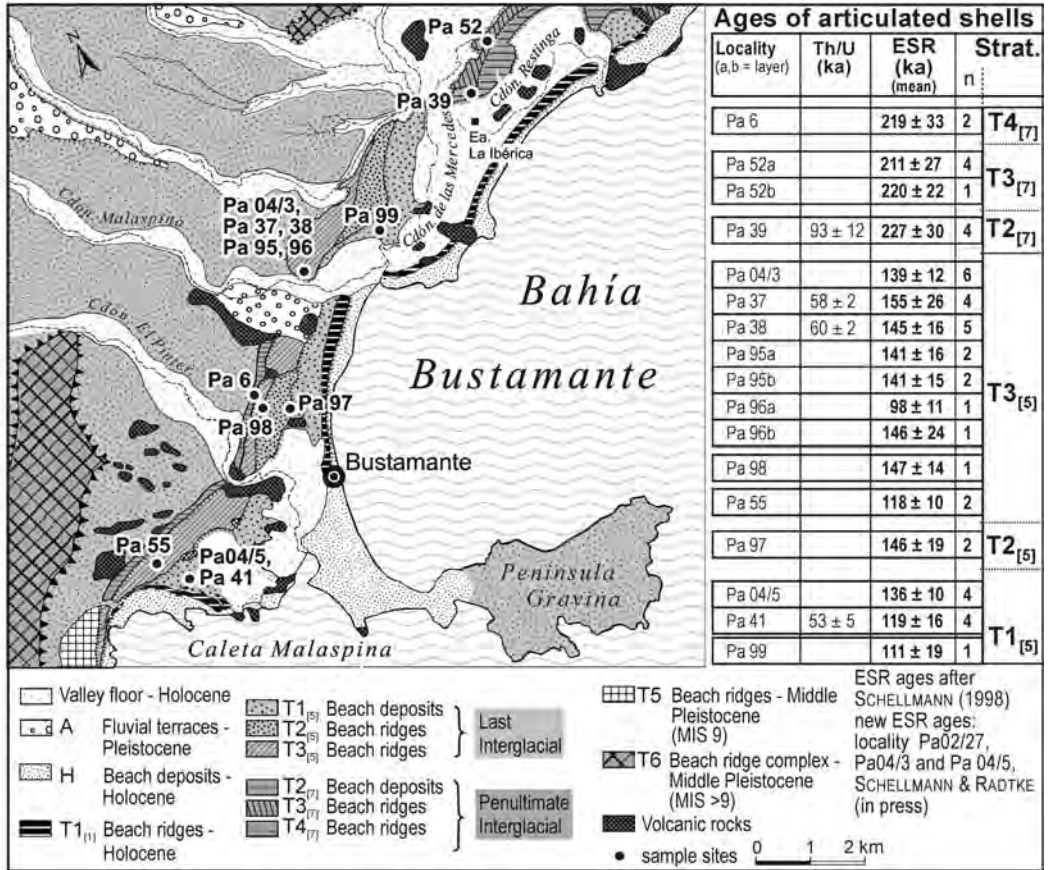


Fig. 7: ESR (mean ages) and TIMS Th/U ages of articulated mollusc shells of Last and Penultimate Interglacial beach ridge systems along the Patagonian Atlantic coast near Bustamante. TIMS Th/U dating by A. Rostami & A. Mangini (Institut für Umweltphysik, Universität Heidelberg); details in SCHELLMANN (1998) and SCHELLMANN & RADTKE (2000).

Abb. 7: ESR (gemittelte Alter) und TIMS Th/U Alter gemessen an Muschelschalen in Lebendstellung aus Strandwalledimenten des letzten und vorletzten Interglazials entlang der patagonischen Atlantikküste in der Nähe von Bustamante. TIMS Th/U Datierungen wurden von A. Rostami & A. Mangini am Institut für Umweltphysik, Universität Heidelberg durchgeführt. Details bei SCHELLMANN (1998) und SCHELLMANN & RADTKE (2000).

Fig. 7 together with the results of TIMS Th/U and ESR dating, mainly of bivalve molluscs found in the sediments (SCHELLMANN 1998). ESR dating confirms the general morphological and podostratigraphic differences between the individual beach ridge systems of the area (SCHELLMANN 1998). Beach ridges in distal position to the present shoreline (T4_[7] to T2_[7]) developed during the Penultimate Interglacial (ca. 220,000 yr ago). The beach ridge systems

closer to the sea, T3_[5] to T1_[5] were formed during the Last Interglacial (ca. 130,000 yr ago). The ESR ages are not precise enough to allow a differentiation in age between different beach ridge systems such as T4_[7] to T2_[7] and T3_[5] to T1_[5], respectively. Additionally, both Holocene (Fig. 9) and Last Interglacial ESR ages apparently tend to overestimate the real age of the sample. The results of TIMS Th/U however, appear much too young and do not allow a

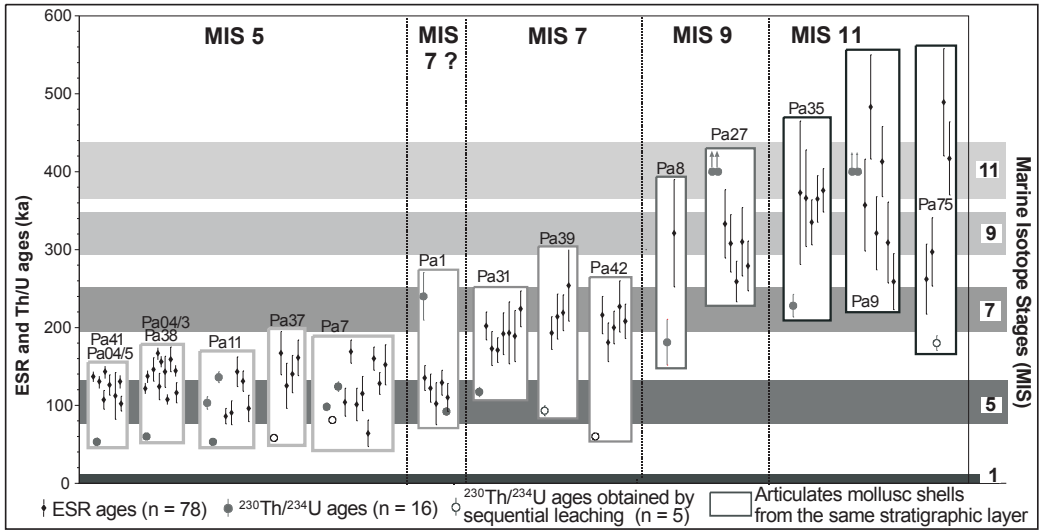


Fig. 8: Comparison of ESR and TIMS Th/U ages of mollusc shells from Late and Middle Pleistocene beach ridge systems along the Patagonian Atlantic Coast. TIMS Th/U dating by A. Rostami & A. Mangini (Institut für Umweltpophysik, Universität Heidelberg); details in SCHELLMANN (1998) with additional datings from SCHELLMANN & RADTKE (2007).

Abb. 8: Vergleich der ESR- und TIMS Th/U Alter von Muschelschalen aus spät- und mittelpleistozänen Strandwallsystemen entlang der patagonischen Atlantikküste. TIMS Th/U Datierungen wurden von A. Rostami & A. Mangini am Institut für Umweltpophysik, Universität Heidelberg durchgeführt; Details bei SCHELLMANN (1998) mit zusätzlichen Daten aus SCHELLMANN & RADTKE (2007).

differentiation between beach ridge systems of the Last and Penultimate Interglacial (Fig. 7). A similar picture is also revealed when comparing the whole data set of ESR and Th/U dating from different localities along the Patagonian Atlantic coast (Fig. 8). Only the ESR ages allow, with an error of 10-15 %, a geochronological differentiation between Last and Penultimate Interglacial littoral terraces. In cases where several datings are available, a differentiation of beach deposits belonging to Marine Isotope Stage (MIS) 9 is partly possible.

For Holocene mollusc shells, radiocarbon dating can be an independent reliability control for ESR dating. Although such a comparison shows fairly good agreement and reproducibility, it is obvious that significant discrepancies do occur (Fig. 9). It should be kept in mind that articulated mollusc shells within a sediment layer certainly are of the same age. As the external dose rate within the sediment

is equal for all samples and possible variations of internal dose rate cannot account for the observed age differences, the variation in age must be related to D_E calculation and some not yet known properties of the ESR dating signal at $g = 2.0007$. We can conclude that ESR ages of Holocene and Pleistocene mollusc shells can scatter substantially and result in significantly too high age estimates. It is hence necessary to date several shells out of a sediment layer to establish a reliable chronological frame for marine terraces. The given accuracy of ESR dating of mollusc shells will, however, only allow correlating the littoral deposits with certain interglacials but not more precisely.

3.2 ESR dating of Late Pleistocene land snail *Helix* sp. from aeolianites of the SE coast of Cyprus

Although first ESR dates for mollusc shells

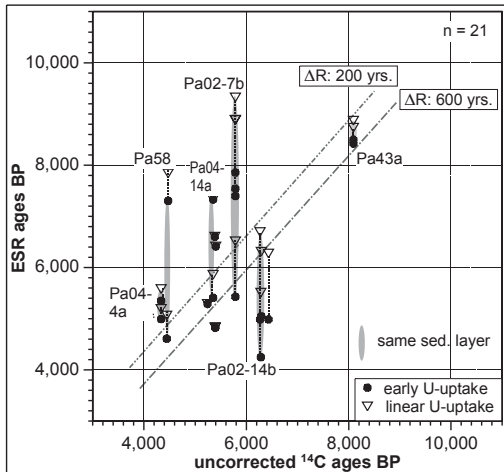


Fig. 9: Comparison of Radiocarbon and ESR ages on articulated Holocene mollusc shells from the Patagonian Atlantic coast (details in SCHELLMANN & RADTKE 2007).

Abb. 9: Vergleich von ^{14}C - und ESR-datierten, artikulierten Muschelschalen aus dem Holozän an der patagonischen Atlantikküste (Details bei SCHELLMANN & RADTKE 2007).

have been produced by RADTKE (1985), little research has been carried out on dating land snails (e.g. MOLODKOV 1993, SKINNER & SHAWL 1994, ENGIN et al. 2006). A comprehensive study on this topic was conducted by SCHELLMANN & KELLETAT (2001) on snail shells gathered from aeolianites from the SE coast of Cyprus. The Late Pleistocene aeolianites are spread over several kilometres along the coast and are exposed in cliffs caused by littoral erosion. In some areas, such as along the coast at Nissi Beach (Fig. 10), the basal part of the aeolianite is visible just below present sea level. As the southern coast of Cyprus has only been weakly uplifted since the Last Interglacial (SCHELLMANN & KELLETAT 2001), the deposition of the aeolianite probably occurred during times when the sea level was near the coast and therefore only a few ten metres below its present position. During the Late Pleistocene, such sea level stands occurred during the later parts of MIS 5.

Snail shells of *Helix sp.* have been used to confirm this geomorphologic interpretation of the

age of the aeolianites by means of geochronology (Fig. 10). According to ESR dating, the deposition of dune sands at Cape Greco took place between ca. 66,000 to 72,000 years ago. At Nissi Beach, snail shells from the aeolianites have been dated to ca. 84,000 to 95,000 yr. According to this data, deposition of the youngest aeolianites took place during the second half of MIS 5 when the sea level was not deeper than some ten metres below the present position (e.g. THOMPSON & GOLDSTEIN 2005; RADTKE & SCHELLMANN 2005: 99ff.). Radiocarbon dating of the mollusc shell gave much lower ages (Fig. 10) and cannot be used for geochronological interpretations. This confirms the well-known phenomenon that the upper dating limit of radiocarbon dating of carbonates is often reached at about 25,000 to 30,000 yr (RADTKE 1988). The reliability of ESR dating is impressively confirmed at Cape Greco, where the aeolianites are situated on top of beach deposits of the transgression phase of the Last Interglacial (Fig. 8). ESR dates of single shells from these marine deposits indicate an age of 130,000 to 137,000 yr. All together, the results from Cyprus underline the potential of ESR to date terrestrial mollusc shells.

3.3 ESR dating of Pleistocene corals from Barbados

Due to the fact that dose rate determination is relatively unproblematic, as already discussed above, the dating of aragonitic corals has a rather high potential. First test studies on ESR dating of corals were published in the late 1980s (IKEYA & OMUHRA 1983, RADTKE & GRÜN 1988, RADTKE et al. 1988, RADTKE 1989, GRÜN et al. 1992) and since then, several methodological improvements and the development of more stable and high-resolution ESR spectrometers have significantly increased the quality of the dating results. It is now not only possible to distinguish between the major periods of high sea level during the last 500,000 yrs but also to differentiate between sub-maxima, for example, during MIS 5 (e.g. SCHELLMANN

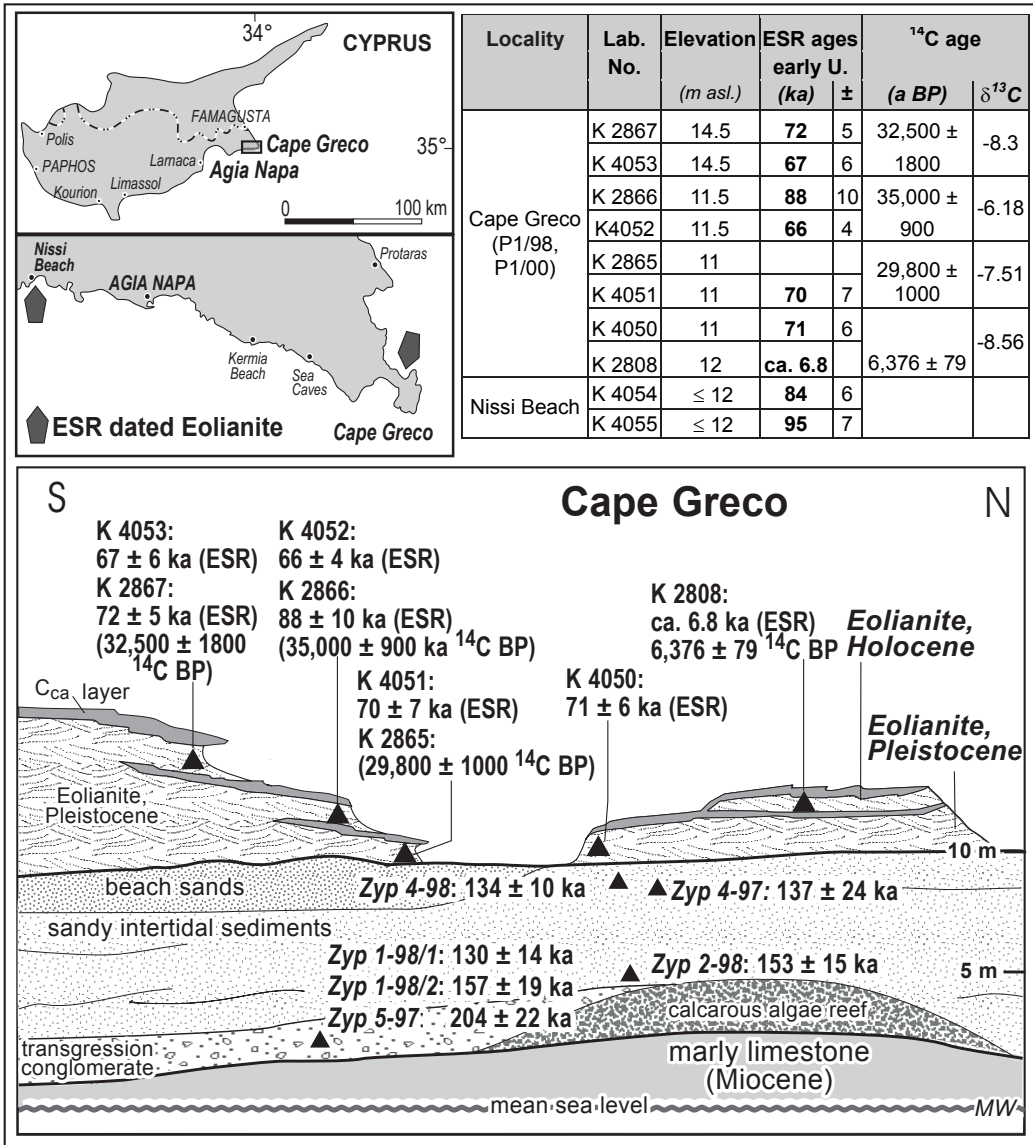


Fig. 10: Radiocarbon and ESR ages of Late Pleistocene aeolianites and beach deposits from the SE coast of Cyprus (SCHELLMANN & KELLETAT 2001).

Abb. 10: ¹⁴C- und ESR-Alter spät-pleistozäner äolischer Ablagerungen und Strndablagerungen von der Südost-Küste Zyperns (SCHELLMANN & KELLETAT 2001).

et al. 2004a; SCHELLMANN & RADTKE 2004a). Prerequisite is that numerous corals from one stratigraphic unit and ideally from several locations are dated. Only with this approach, can the effect of weak diagenetic alterations of the coral material and the resulting underestima-

tion of ESR ages be detected from the scatter of individual results. The high quality of ESR dating is proven by comparison with TIMS U/Th dating. For this purpose, up-lifted Late Pleistocene corals from different elevations and age from the southern coast of Barbados

were systematically investigated (Fig. 11). At an elevation of 21 to 43 m above present sea level (asl.), three different coral reefs that formed during the transgression maximum of MIS 5e were identified. Three coral reefs at an elevation between 4 and 17 m asl. are correlated with the sub-maximum of MIS 5c and two further reef terraces at 2-3 m asl. are interpreted to represent sub-stages MIS 5a₁ and MIS 5a₂ (SCHELLMANN & RADTKE 2004a, SCHELLMANN & RADTKE 2004b).

The relatively large scatter of ESR ages determined for individual coral reef terraces is most likely caused by weak diagenetic alterations, which cause re-crystallisation and/or up-take of Uranium. The result is that some ESR ages underestimate the real age of the sample. As a consequence, the oldest ESR samples are more likely to represent the actual age of a coral reef and should hence be used for the chronological interpretation. When more than 20 ESR ages were available for a coral reef terrace, this was accounted for by using the 90 percentile value, and if less than 20 ages were available, then the upper quartile value was used to determine the mean age of the reef. Comparing the results of this approach with the median of U/Th dating shows a rather good concordance (Table 2). Differences in

age are on average not more than 3000 yr and hence within the error of ESR dating.

Generally, TIMS U/Th of Late Pleistocene corals is considered to be a highly precise method with analytical errors of < 1 %. However, comparing the results of ESR and TIMS Th/U dating of Last Interglacial corals from Inch Marlowe Point (Fig. 12) and Batts Rock Bay (Fig. 13) reveals that the quality of TIMS U/Th is not better than that of ESR (SCHELLMANN et al. 2004a). At Inch Marlowe Point, ESR ages are about 1000 to 8000 yr younger than TIMS U/Th regardless the actual age of the samples. The upper quartile value of all ESR ages is 73,100 yr and the median of all U/Th ages is 76,700 yr. Interestingly, the spread of individual ages (not considering error) for both methods is about 4000 yr, although all ages were produced on individuals from the same branch of corals and are hence most likely of the same age (grown within not more than a few hundred years). Hence, the high precision of the TIMS U/Th measurements (~ 1 %) apparently does not completely account for the observed scatter in ages of isochronal samples. In contrast, the observed scatter of ESR dating is explained by the relatively high analytical error (low precision) (5-8 %). Hence, when considering the whole data set, the accuracy of

Table 2: ESR ages (upper quartile values) and TIMS U/Th ages (median values) from coral samples from the south and west coast of Barbados. TIMS U/Th dating by E.-K. Potter (Australian National University, Canberra); Details are provided by SCHELLMANN et al. (2004a).

Tab. 2: ESR Alter (Werte oberes Quartil) und TIMS U/Th Alter (gemittelte Werte) von Korallenproben der Süd- und Westküste von Barbados. Die TIMS U/Th-Datierungen wurden von E.-K. Potter (Australian National University, Canberra) durchgeführt. Für weitere Details siehe SCHELLMANN et al. (2004a).

Strat.	Terrace	ESR ages			U/Th ages		
		ka	±	n	ka	±	n
MIS 5a-1	T1a-1	73.4	5	14	76.7	0.6	12
MIS 5a-2	T1a-2	80.9	5	5	84.2	0.7	4
MIS 5a-2	N2	85.5	6	7	84.5	0.8	3
MIS 5c-3	T3	102.6	6	8	102.9	1	3
MIS 5c	N1 _[5c]	108.7	9	5	105.4	1	7

ESR = Upper quartile value (25% of all ESR ages are ranked above this value)

U/Th = Median of U/Th data with initial delta ²³⁴U values between 141 and 157 permil

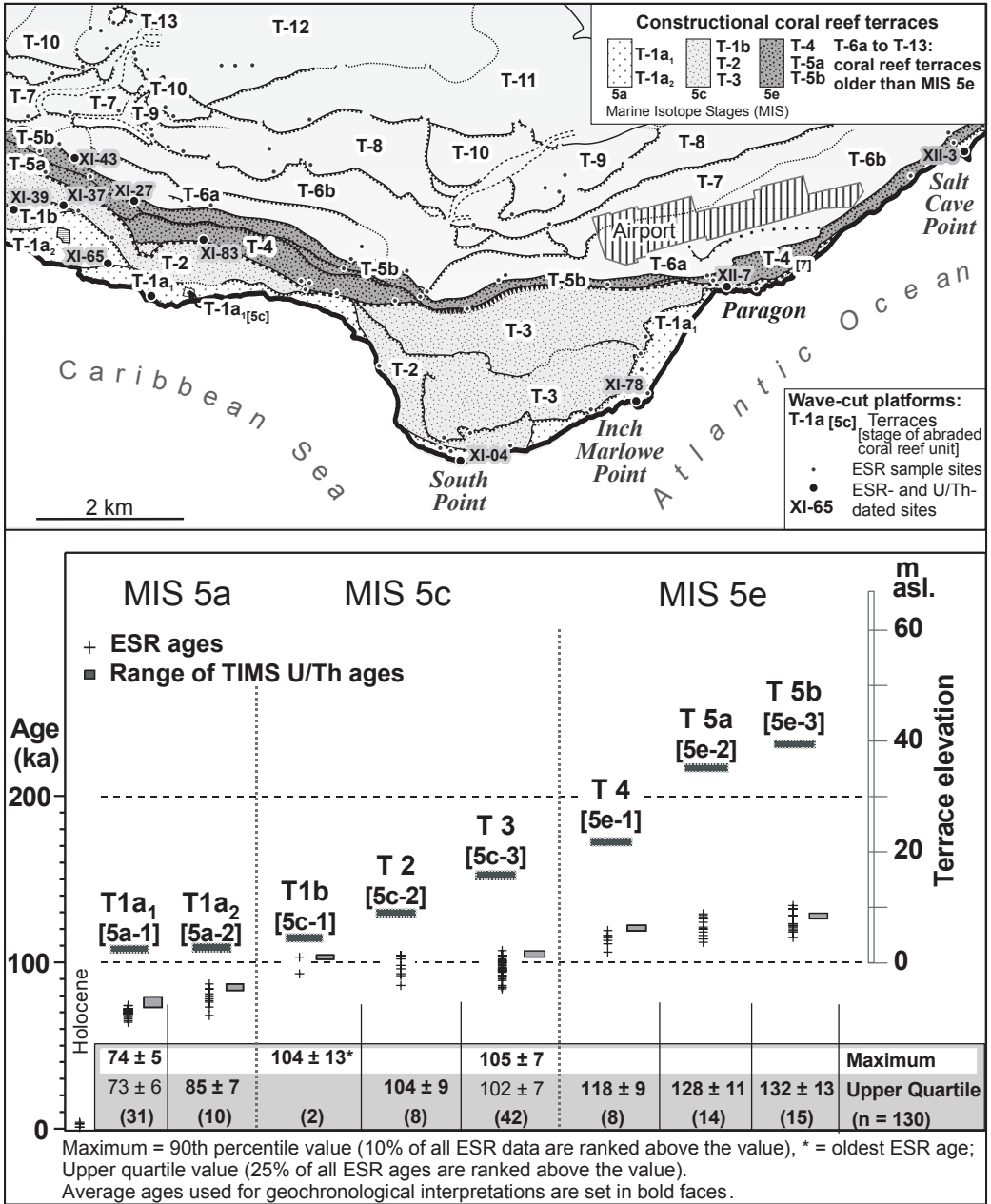


Fig. 11: Coral reef terraces along the southern coast of Barbados with location of ESR and TAMS U/Th dated corals (XI-No. = dated location). TAMS U/Th datings were carried out by E.-K. Potter (Australian National University, Canberra). Details are provided in SCHELLMANN et al. (2004a).

Abb. 11: Korallenriff-Terrassen entlang der Südküste von Barbados mit den Entnahmepunkten für die Datierung von Korallen mittels ESR und TAMS U/Th (XI-No. = datierte Lokation). TAMS U/Th-Datierungen wurden von E.-K. Potter (Australian National University, Canberra) durchgeführt. Für weitere Details siehe SCHELLMANN et al. (2004a).

individual TIMS U/Th ages is not better than that of ESR dating. The best possible resolution of both dating methods is also nicely demonstrated for branches of corals sampled at Batts Rock Bay, west coast of Barbados, that were formed during the two sea level sub-maxima of MIS 5c und MIS 5a. According to this data set, both dating approaches are associated with a relatively high non-systematic spread of ages of a few thousand years.

3.4 ESR dating of Holocene corals from the Netherlands Antilles (Aruba, Bonaire, Curaçao)

To further confirm the accuracy of ESR dating, 21 radiocarbon-dated sub-modern, as well as Late and Middle Holocene, coral samples from Tsunami deposits found on Aruba, Bonaire and Curaçao (Netherlands Antilles) were dated by ESR (Fig. 14). The calibrated radiocarbon ages spread between 0 and 3644

years and the ESR ages were between 9 and 3653 yr (not considering the individual uncertainties of each measurement). Despite one sample that is considered an outlier, the ages determined for both methods are consistent within an error range of ca. 250 yr. However, most ESR data shows a tendency towards slightly higher values in ages compared to radiocarbon, which were reservoir corrected assuming that $\Delta R = -49$ (ca. 392 yr). It is likely that the marine reservoir effect did considerably change in the past and that the assumed ΔR -value is not representative for all samples. The calibrated radiocarbon ages may hence not automatically represent the "true" age of a sample. The improved quality of ESR dating is also demonstrated by ESR dating of sub-modern corals that were collected alive by the Zoological Museum of Amsterdam University in the year 1920. For these samples, uncorrected ^{14}C ages were 586 ± 24 and 595 ± 24 , respectively. ESR ages were „recent“

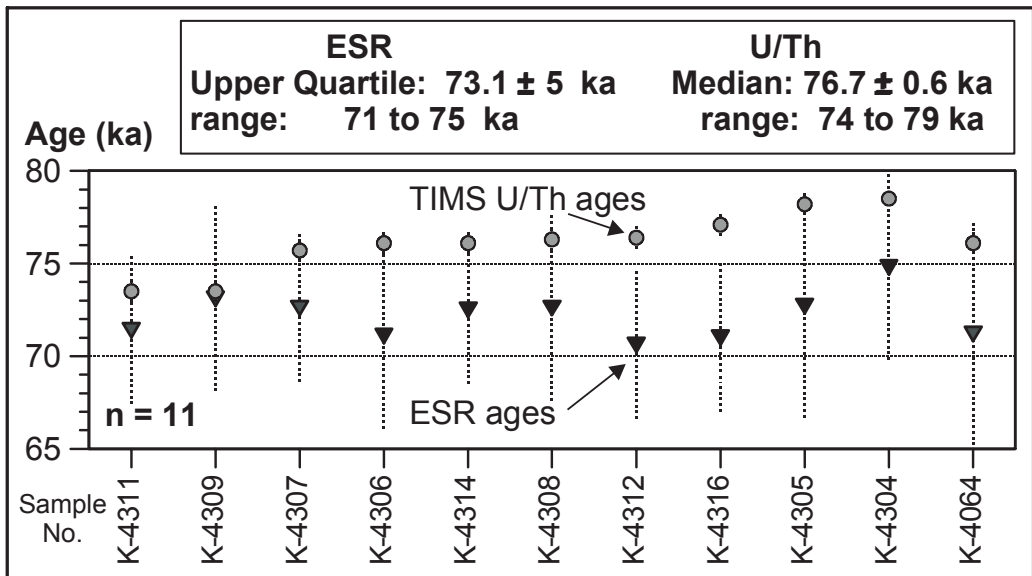


Fig. 12: ESR and TIMS U/Th ages ($\delta^{234}\text{U} > 141$ and $< 157\%$) of coral reef terrace T-1a₁ near Inch Marlowe Point, southern coast of Barbados. See sample site XI-78 in Fig. 11 (slightly modified after SCHELLMANN et al. 2004a).

Abb. 12: ESR und TIMS U/Th-Alter ($\sigma^{234}\text{U} > 141$ und $< 157\%$) der Korallenriffterrasse T-1a bei Inch Marlowe Point an der Südküste von Barbados. Die Lage des Entnahmepunktes XI-78 ist Abb. 12 zu entnehmen (leicht verändert nach SCHELLMANN et al. 2004).

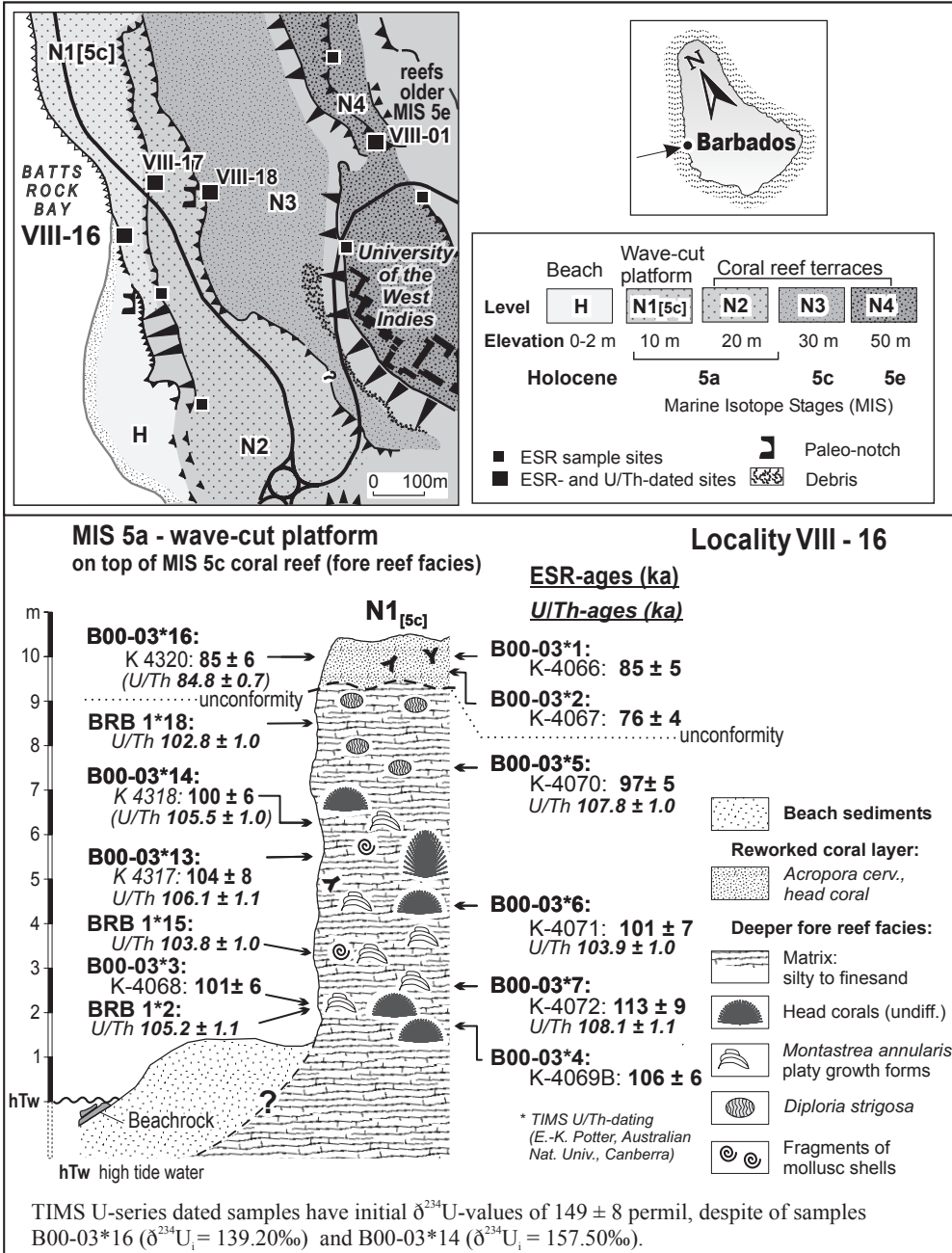


Fig. 13: ESR and TIMS U/Th ages of MIS 5a and MIS 5c coral reefs from Batts Rock Bay. TIMS U/Th datings were carried out by E.-K. Potter (Australian National University, Canberra). Details are provided in SCHELLMANN et al. (2004a).

Abb. 13: ESR- und TIMS U/Th-Alter von Korallenriffen (MIS 5a und MIS 5c) von Batts Rock Bay. Die TIMS U/Th-Datierungen wurden von E.-K. Potter (Australian National University, Canberra) durchgeführt. Für weitere Details siehe SCHELLMANN et al. (2004a).

and 232 ± 22 before present (~ 1778 AD) (RADTKE et al. 2003: Table 1). Considering the small amplitude of the ESR dating signal from such young samples, it is astonishing that the ESR ages are much closer to the „real“ age of 1920.

4 ESR dating of quartz

4.1 ESR dating of heated quartz

As ESR signals in quartz usually decay when heated, heating mechanisms in nature may provide an appropriate means of resetting the geological clock, enabling one to establish the age of the (last) heating. For instance, xenolithic quartz in volcanic rocks in the Eifel region (Germany) was successfully dated with the Al-centre (WODA et al. 2001). In general, the ESR ages were in good agreement with independent age control (Ar-Ar) up to 500 ka (Fig. 15).

Similarly, quartz-rich sediments, which were sufficiently heated by overlying lava flows, can be used to estimate the geological age of the lava flow. MIALLIER et al. (1994a) showed the potential of Ti- and Al-centres to date sediment baked by a Late Pleistocene lava flow by comparison with red TL and other independent age controls. However, dating attempts of heated quartz from a ~ 580 ka pumice ($^{40}\text{Ar}/^{39}\text{Ar}$) showed scattered ESR underestimates for these centres (MIALLIER et al. 1994b), leading to the conclusion that the dating range of ESR is below 500 ka for such materials.

ESR results for heated quartz were recently published by TOYODA et al. (2006). In this study, Al- and Ti-centres were used for ESR dating of Quaternary tephra from Japan, in comparison with red TL and other independent age controls (fission track, K/Ar). Although some degree of consistency could be observed for samples younger than 60 ka, the ESR results appeared very scattered, internally (inconsistencies between Al- and Ti-centres) as well as externally (inconsistencies between the ESR and TL data).

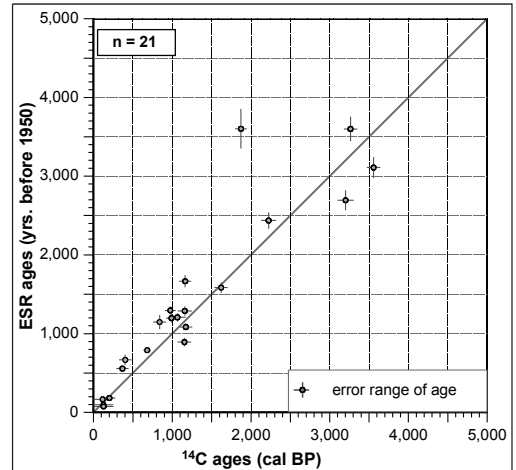


Fig. 14: ESR and radiocarbon ages of Holocene corals from Aruba, Bonaire and Curacao (Netherlands Antilles). Calibrated radiocarbon ages were corrected for marine reservoir effect. Radiocarbon dated coral samples were provided by D. Kelletat & A. Scheffers (Institut für Geographie, Universität Essen); dating and calibration by B. Kromer (Institut für Umweltp Physik, Universität Heidelberg); details in RADTKE et al. (2003).

Abb. 14: ESR- und ^{14}C -Alter holozäner Korallen aus Aruba, Bonaire und Curacao (Niederländische Antillen). Die kalibrierten ^{14}C -Alter wurden um den marinen Reservoireffekt korrigiert. Die ^{14}C -Alter wurden von D. Kelletat & A. Scheffers (Institut für Geographie, Universität Essen) zur Verfügung gestellt, Datierung und Kalibration durch B. Kromer (Institut für Umweltp Physik, Universität Heidelberg). Weitere Details bei RADTKE et al. (2003).

Reported ESR ages of heated quartz are often lower for the Al-centre, in comparison with the Ti-centre. This may be due to different thermal stabilities of Al- and Ti-centres (TOYODA & IKEYA 1994). Furthermore, high ambient temperatures such as those in geothermal areas may be sufficient to lower the natural ESR signal of volcanic quartz, eventually leading to age underestimates (TOYODA et al. 1995). RINK (1997) argued that this would lower the ESR dating range of volcanic rocks to around 50-60 ka. Alternatively, age overestimates may result from insufficient signal zeroing of ESR signals before or during the eruption (WODA et al. 2001). Several studies indicate that the

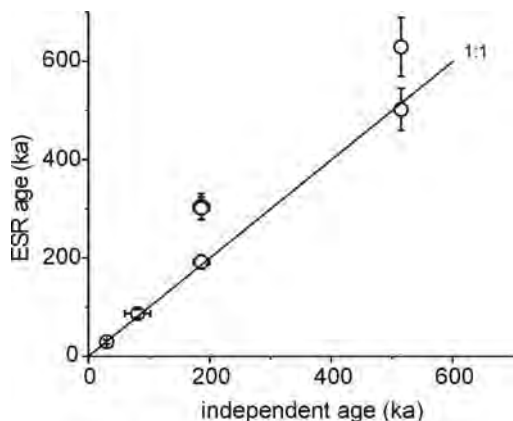


Fig. 15: ESR age plot for samples taken from volcanic deposits in the Eifel region, Germany. Replotted after WODA et al. (2001).

Abb. 15: Darstellung von ESR-Altern aus vulkanischen Ablagerungen der Eifel, Deutschland (aus WODA et al. 2001).

E_1' -centre can be used to critically reject insufficiently heated samples (FALGUÈRES et al. 1994, WODA et al. 2001).

4.2 ESR dating of strained quartz

ESR dating of faults is based on the principle of signal zeroing due to fault activity. Fault movements may strain quartz in fault gouge in such a way that the ESR signals are completely zeroed during faulting. Subsequently, the ESR signal starts to grow again, thus allowing the determination of the geological age of the last fault movement. ESR dating of fault gouge is of particular importance in seismic risk assessment. Generally, fault gouge is dated by the ESR plateau method (BUHAY et al. 1988). The methodology involves the use of several ESR centres (usually E_1' -, OHC- and Al-centres) from different quartz grain size fractions. The idea is that the smaller the grain size, the more the quartz is affected by strain and the more the geological clock has been reset (Fig. 16).

Usually, the best age estimates are obtained from the smallest grain size fractions (LEE & SCHWARCZ 1994). Sometimes however, even the smallest grain size fractions (i.e., around

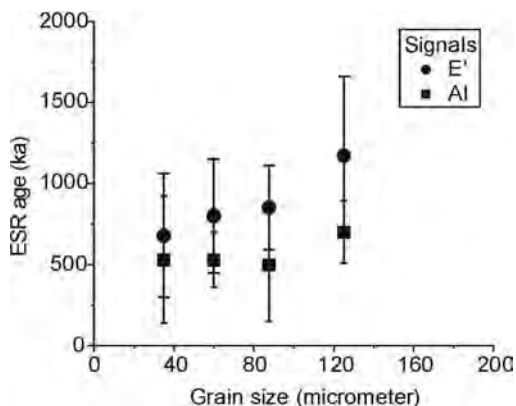


Fig. 16: ESR age plot for fault gouge from the Wangsan Fault (Southern Korea) according to the grain size and the ESR centre used. Note the systematically higher ages for the E_1' -centre. Replotted after LEE & YANG (2003).

Abb. 16: ESR Alter aus Proben der Wangsan Störung (Süd Korea) bezogen auf die Korngröße und das genutzte ESR Zentrum. Zu beachten sind die systematisch höheren Alter für das E_1' -Zentrum (nach LEE & YANG 2003).

30 μm) do not reflect the age of the last fault movement. For instance, this was shown by LEE & YANG (2003) in a study on ESR dating of the Wangsan Fault in Southern Korea. Here, ESR ages based on the E_1' - and Al-centres in quartz from fault gouge cluster around 500-600 ka, but the fault gouge is considered to have been lifted to the surface by later fault movements, which were not strong enough to reset the ESR signals. As such, the reported ESR ages are considered to reflect the latest global reactivation of the fault. In some cases, it appears only possible to calculate maximum ages for the last fault reactivation, as shown by LEE & YANG (2007).

The E_1' -centre is commonly observed in quartz and silica, and has been used to investigate the age of fault movements, heated flint and volcanic ash. However, ESR ages based on this centre should be carefully interpreted because of the possible occurrence of an overlapping unstable signal, the so-called counterfeited E_1' -signal (TOYODA & SCHWARCZ 1994)

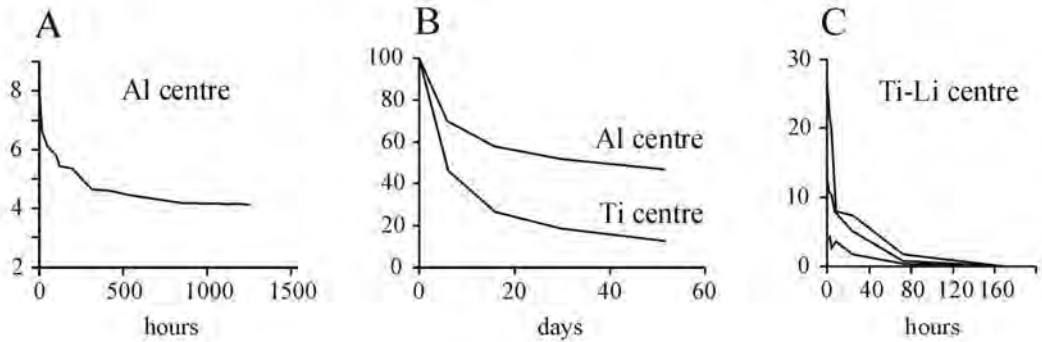


Fig. 17: Simplified bleaching curves for Al- and Ti-centres in quartz. Replotted after A: LAURENT et al. (1998), B: BRUMBY & YOSHIDA (1994) and C: TOYODA et al. (2000). A: UV-lamp, B: natural sunlight, C: halogen lamp.

Abb. 17: Vereinfachte Bleichkurve für die Al- und Ti-Zentren in Quarz. A: nach LAURENT et al. (1998). B: nach BRUMBY & YOSHIDA (1994) und C: nach TOYODA et al. (2000). A: UV-Lampe, B. natürliches Sonnenlicht, C: Halogenlampe.

4.3 ESR dating of sedimentary quartz

Quartz grains that are exposed to natural sunlight sufficiently long will have their ESR signals reset to a certain extent during transport, prior to burial (Fig. 17). This important principle forms the basis of ESR dating of sediments. It is one of the most promising sub-disciplines in ESR dating. The specific age range of ESR dating of quartz, possibly up to several Ma, makes sediment dating a potentially very valuable tool, with applications in geology, geography, prehistory and palaeoanthropology. The specific issue in ESR dating of sediments is the extent to which the geological clock is reset to zero, or to a value, which can be determined in the laboratory. Resetting of the geological clock proceeds through sunlight bleaching of ESR centres during transport, prior to burial. Experimental bleaching curves are shown in Fig. 17 for various ESR centres and various artificial light sources. The Al-centre contains an unbleachable residual, whereas Ti-centres can be fully (or to a negligible level) reset. Note that bleaching times are on the order of days to weeks.

4.3.1 Aeolian sediments

The most straightforward application of ESR dating of sedimentary quartz is that of aeolian sediments, because of the sunlight bleaching potential. Several studies report reasonable ESR ages for aeolian sand, based on the Al-centre, in the time frame up to several 100 ka (YOKOYAMA et al. 1985, TANAKA et al. 1995). Since the Al-centre contains an important unbleachable residual with respect to natural sunlight radiation, it is important to mention that the ESR ages reported in these studies are corrected for this residual. The principle of this method is shown in Fig. 18. Recent investigations in ESR dating of aeolian sand using the Al-centre also include Chinese loess deposits (YIN et al. 2007). Although the results show a strong underestimate relative to the independent age control of the loess, the method seems very promising if the residual dose can be determined very accurately.

The problem of residual doses is less pertinent with Ti-related ESR centres because bleaching studies indicate that the ESR signal can be reset completely or at least to a negligible level with respect to deposits older than Late Pleistocene. Recently for instance, it was shown that in-

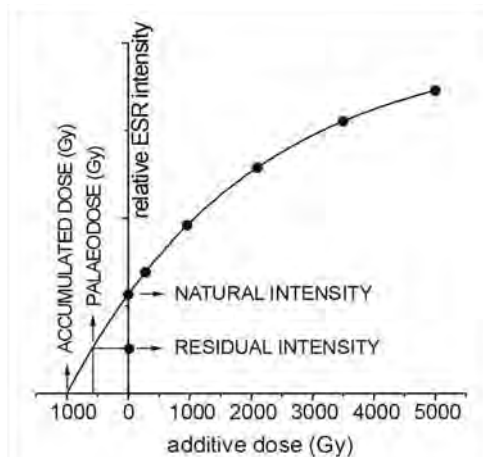


Fig. 18: Correction for the unbleachable residual in ESR dating of quartz sediments using the Al-centre. The residual intensity is measured after prolonged bleaching and this level is used to infer the true palaeodose. Replotted after LAURENT et al. (1998).

Abb. 18: Korrektur unbleichbarer Reste bei der ESR-Datierung von Quarzsedimenten unter Berücksichtigung des Al-Zentrums. Die Restintensität wird nach verlängerter Bleichung gemessen, wobei dieses Niveau zur Bestimmung der Paläostrahlungsrate genutzt wird (nach LAURENT et al. 1998).

dividual quartz grains show non-existing or only very small residual Ti-related doses in an Egyptian desert sand deposit (Fig. 19; BEERTEN & STESMANS 2005).

The potential and problems of multiple grain ESR dating using Ti-centres has been outlined in BEERTEN et al. (2006) for Australian sand dunes up to 350 ka. Large differences are often encountered for individual Ti-related sub-centres in the regenerative dose method, but it is not clear yet whether this is due to sensitivity changes or not. ESR ages based on Ti-H centres seem to underestimate the OSL ages for this profile, whereas the opposite is true for Ti-Li centres. Comparisons with the additive dose method are often hampered by improper dose response showing inflexion points (BEERTEN et al., in press).

The potential and problems of single grain ESR dating using Ti-centres has been outlined in BEERTEN & STESMANS (2007). The first results

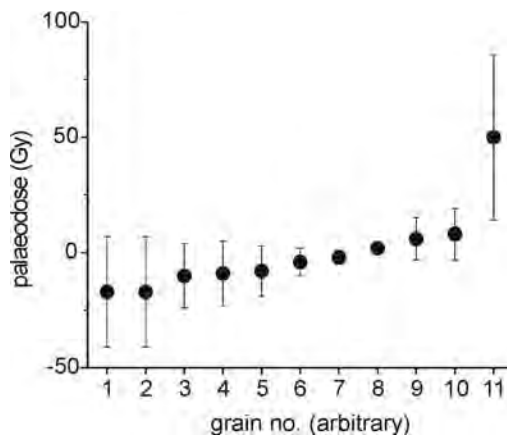


Fig. 19: ESR equivalent dose plot for Ti-centres from individual grains taken from a modern desert surface deposit, Eastern Desert, Egypt. Except for one grain (no. 11), all values are consistent with the expected dose of 0 Gy. Replotted after BEERTEN & STESMANS (2005).

Abb. 19: Darstellung der ESR Äquivalent Dosis für Ti-Zentren, gemessen an Einzelkörner moderner Oberflächenablagerungen aus der Östlichen Wüste, Ägypten. Mit Ausnahme einer Messung (Korn Nr. 11) zeigen alle Messungen konsistente Ergebnisse um den erwarteten Wert von 0 Gy (nach BEERTEN & STESMANS 2005).

of this new approach in ESR dating are very promising, but appropriate adaptations in instrumentation and equipment are necessary to make it more flexible and user friendly.

4.3.2 Fluvial and estuarine sediments

ESR dating has been shown to be a useful tool for establishing geochronological frameworks for fluvial and estuarine/marine deposits. Despite proven difficulties with bleaching residuals related to the Al-centre in quartz, this specific ESR centre has been used in many studies to date sedimentary sequences spanning the whole Quaternary period, and even up to the Miocene. A famous example of ESR dating of a fluvial terrace staircase is that from the Somme Basin, France, where consistent ESR results could be obtained relative to biostratigraphi-

cal, archaeological and palaeomagnetic data (Fig. 20; LAURENT et al. 1998, Antoine et al. 2000). The same methodology, i.e. ESR of the Al-centre with subtraction of the unbleachable residual, has been used for other terrace staircases as well, with promising results (VOINCHET et al. 2004, BAHAIN et al. 2007, TISSOUX et al. 2007). The Al-centre even has the potential to date Plio-Miocene estuarine deposits, as was shown by LAURENT et al. (1998). In this study, a consistent ESR dating pattern for deposits from the Tiglian up to the Tortonian (around 8-9 Ma) was obtained.

The quality of the ESR ages from these studies is guaranteed to some extent by bleaching experiments on recent fluvial deposits. LAURENT et al. (1998) and VOINCHET et al. (2003, 2007) showed that the Al-related ESR signal in quartz is at or very close to the unbleachable residual

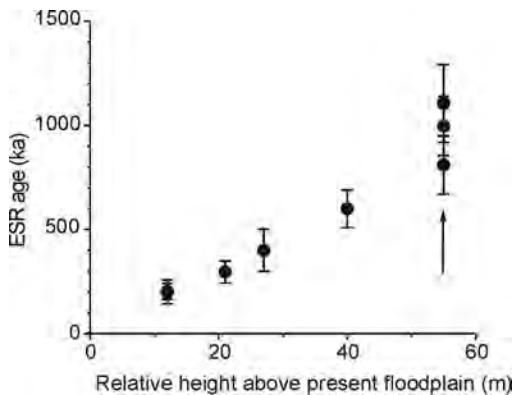


Fig. 20: ESR dating of the River Somme terrace staircase. Replotted from data in LAURENT et al. (1998) and ANTOINE et al. (2000). Only ESR ages on quartz are given. Other age control is available for the terrace sequence. The highest level in this diagram is supposed to be older than the B/M boundary, based on palaeomagnetic measurements. This is confirmed by the ESR dates (see arrow).

Abb. 20: ESR-Daten aus der Somme Terrassenstufe. (nach LAURENT et al. 1998 und ANTOINE et al. 2000). Es sind nur Alter von Datierungen an Quarz dargestellt. Für die Terrassensequenz sind auch andere Alterskontrollen möglich. Für die oberste Stufe wird durch paläomagnetische Altersbestimmung ein Alter älter als die B/M Grenze angenommen. Dies wird durch die ESR-Daten bestätigt (s. Pfeil).

level for various modern (i.e. zero-age) fluvial samples. Another point that should be kept in mind when evaluating such ESR dating results, is the targeted age range. Whereas, due to the slow bleaching behaviour, the Al-centre is probably inappropriate for detailed chronologies in the Late and late Middle Pleistocene, it may be a valuable tool for rough age determinations of extended Early Pleistocene and Pliocene sedimentary sequences, especially if other age-markers or suitable materials for dating are absent. Therefore, the highest potential of the Al-centre could be situated in the Tertiary, where a rough assignment to chronozones could be sufficient to tune estuarine/marine sedimentary sequences to the global sequence-stratigraphic timescale.

However, some fundamental issues in ESR dating using the Al-centre remain ambiguous. First, the determination of the bleaching residual usually proceeds through artificial bleaching experiments using some kind of 'solar simulator'. Up to the present, it is unclear how the irradiation spectrum of the solar lamp influences the magnitude of this residual. Second, the accuracy of the results is only guaranteed if this bleaching residual is a fixed value, in terms of absolute ESR defect concentration, as was pointed out by BRUMBY & YOSHIDA (1994b).

Lately, more attention has been paid to the use of Ti-centres for ESR dating of fluvial deposits from terrace staircases as well. A comparative study between ESR dating results of Al- and Ti-centres recently showed that both species have a large potential in geochronological studies (TISSOUX et al. 2007). However, from detailed studies it has become clear that individual ages at the single-grain scale may vary considerably if Ti-centres are used (BEERTEN et al. 2003). Ti-related ESR centres may even contain a small but detectable unbleachable residual, as was recently shown with artificial bleaching experiments on fluvial quartz (VOINCHET et al. 2007).

4.3.3 Glacial sediments

Grinding of quartz grains may occur not only

in a fault-related context, but also in some sedimentary environments. Together with sunlight bleaching, such grinding mechanisms could be an effective zeroing mechanism in ESR dating of glacially derived sediments. As such, the Ge-related impurity centre in quartz appears to generate promising results, e.g. in dating Pleistocene glaciations in Central Asia (ZHAO et al. 2006, ZHOU et al. 2006).

Whereas the basis for this specific ESR approach was laid by methodological studies on the behaviour of Ge-centres with respect to sunlight bleaching and pressure (TANAKA et al. 1985, BUHAY et al. 1988, YE et al. 1998), some fundamental points remain unclear. For instance, although growing with small artificial irradiation doses, it appears very difficult to find a natural Ge signal in quartz, as has been pointed out already by RINK (1997). The implication of this observation could be that the Ge centre is not stable at average burial temperatures.

4.4 General remarks

Various methodological issues are common to all types of quartz, irrespective of the geological context. Generally, the additive dose method is used to determine the equivalent dose in quartz. However, sometimes this approach may lead to large uncertainties for samples with a relatively large equivalent dose. In such cases, it would be advantageous to use the regenerative dose method. For ESR centres in quartz, artificial heating may be the most efficient and comfortable way of annealing the signal prior to regenerative dosing. Although this mechanism is not the annealing mechanism in sedimentary environments, this approach is currently being tested for ESR dating of sediments using Ti-related centres (BEERTEN et al. 2006). A major concern here is the possible presence of sensitivity changes (i.e. an alteration of the radiation-sensitivity of the ESR-signal) due to the heating step. Less comfortable in experimental terms, but much more related to the zeroing mechanism in sedimentary environments, is optical bleaching

of ESR centres prior to administering the regenerative dose. First results of this approach were recently published for Al-centres in loess (YIN et al. 2007). Unfortunately, the ESR dose response is often found to be problematic in various quartz samples. Sometimes, strong inflexion points are found in the dose response of the Ti-centre and the Al-centre. There are also observations on non-monotonic linear growth of Ti- and Ge-centres, indicating an ESR signal decrease with very high doses of several 1000 Gy (WODA & WAGNER, in press).

5 ESR dating of some other materials (speleothems, foraminifera, tooth enamel)

ESR dating has also been applied to a wide variety of other materials with great importance, especially in palaeoclimatological and archaeological contexts. The most common applications in this framework include speleothems, foraminifera, and tooth enamel.

Many ESR papers have published combined ESR and U-series dating results of speleothems (stalagmites, stalactites), which generally show good agreement. A detailed review is given by RINK (1997: 990f.). Nevertheless, as stated by PIROUELLE et al. (2007), further research is still needed about the best analytical routine for obtaining correct D_E values, which include systematic dating applications coupled with other (e.g. U-series) dating methods. However, as GRÜN (2007: 1512) stated, U series dating of speleothems and other secondary carbonates is much cheaper, faster and more accurate than ESR.

Studies on ESR dating of planctonic foraminifera from a deep-sea sediment core by MUDELSEE et al. (1992) show good agreement with $\delta^{18}O$ stratigraphy. The authors see the possibility to date planctonic foraminifera up to approx. 800 ka by using the ESR signal at $g = 2.0036$. More recently, HOFFMANN et al. (2001) stated that foraminifera could be dated up to 190 ka, just when using the CO_2 -signal at $g = 2.0006$. All in all, more studies on well-stratified deep sea cores in combination with independent age controls are needed to show

the potential and the limits of ESR dating of foraminifera.

Lately, ESR dating on human tooth enamel, in conjunction with laser ablation U-series dating, has become an important method for determining the age of human remains back to about 200 to 300 ka, beyond the time range of the ^{14}C dating method (e.g. GRÜN 2006, 2007; SORESSI et al. 2007). Most recently, GRÜN (2006) gives a detailed description of the ESR dating method itself in application on human teeth, which includes application examples and comparisons with other dating results (^{14}C , U-series, AAR). In addition, HAMEAU et al. (2007) see a good agreement of coupled ESR/Uranium-series dating of Middle Pleistocene *Rhinoceros* and *Tapirus* tooth enamel on East Java associated with lithic artefacts, which seems to provide a new geochronological framework of human occupation in South-East Asia. Details on ESR dating of teeth are summarized by GRÜN (2006) and RINK (1997).

There are further materials, e.g. travertines, calcretes or bones, which have been the object of ESR dating. However, the ages are less reliable due to complex Uranium uptake histories, to problems of recrystallisations or to carbonate impurities, which often lead to age underestimations (e.g. RINK 1997; GRÜN 2007).

6 Summary and outlook

Due the methodological advances of the past decade, ESR dating has become an important geochronological method, especially for dating deposits such as coral reefs and beach ridges as well as quartz from different geological settings. The reliability of ESR dating of carbonates was proven by direct comparison with other dating methods such as radiocarbon and TIMS U/Th. ESR dating of Holocene corals shows accuracy similar to radiocarbon dating when all sources of uncertainty are fully considered. ESR dating of Pleistocene corals allows not only to distinguish between MIS 1, 5, 7, 9, 11 and 13 deposits, but also between sub-stages $5e_{3/2}$, $5e_1$, $5c$, $5a_1$ and $5a_2$. The average error of ESR dating of corals is about 5-

8%. Late Pleistocene corals can be dated with a similar accuracy as with TIMS U/Th in this time range. The advantage of ESR, however, is that the upper dating limit for corals is probably above 500,000 yr. If diagenesis could be excluded, dating of coral with an age of several million years should be feasible from the physical point of view.

The resolution of ESR dating of molluscs and gastropods is with 10-15 % much lower. Additionally, ages tend to scatter quite substantially, which causes the need to carry out several datings for the same stratigraphic unit to allow accurate geochronological interpretation. Usually it is then possible to correlate layers to certain interglacials periods such as MIS 1, 5, 7 and 9. The high scatter associated with ESR ages of molluscs and gastropods is most likely related to yet not identified problems in palaeodose determination, since problems in dose rate apparently do not account for the scatter.

The examples shown in this paper demonstrate that ESR can be successfully applied to date quartz in various geological contexts. It should be clear that the large age range of ESR dating (up to several Ma), especially in relation to OSL dating (up to several 100 ka), underlines its important position next to other geochronological techniques. However, there are several issues that need further consideration. The accuracy with which the palaeodose can be determined is highly dependent on the degree of resetting of the ESR signal. Some examples show that incomplete resetting could undermine the reliability of the method. Therefore, it is important to incorporate criteria (external and internal) to evaluate the completeness of the zeroing process in nature, such as the plateau method for fault gouge and the use of multiple ESR centres and/or single grain measurements for sediment dating. Cross-checking of the results with independent age control is another crucial step in the development of a reliable ESR dating method. In the case of sediments, reference ages may be provided by luminescence dating (OSL, TL) for Late Pleistocene and in favourable circumstances even Middle Pleis-

tocene deposits. Similarly, ESR dating results of tephra layers could be evaluated against red TL measurements.

The reliability of the palaeodose determination method, purely experimentally, is another concern in ESR dating of quartz. Results of the regenerative dose method and additive dose method should be compared very carefully and a proper annealing mechanism should be looked for in the regenerative dose method (UV-bleaching for sedimentary quartz; thermal annealing for heated quartz). At present, experimental techniques in ESR dating of quartz produce relatively large errors (usually around 15%). In part, this may be related to the dose determination method (i.e. the additive dose technique in the case of older samples), but it is important to verify if intrinsic properties of ESR defects could be the cause of such large errors (i.e. inflexion points). In general, it is believed that the regenerative dose technique may produce much more precise results if there are no sensitivity changes.

There are even more materials, which have the potential for a dating by ESR, however the most reliable applications until now or in the near future seem to be coupled ESR/U-series dating of tooth enamel and perhaps speleothems. A great potential is inherited in ESR dating of foraminifera, which only needs more applications.

In summary, it is clear that the development of the ESR method is far from being complete and it is important to carry out methodological investigations before the full potential of the method can be used.

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Surface exposure dating with cosmogenic nuclides

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Abstract: In the last decades surface exposure dating using cosmogenic nuclides has emerged as a powerful tool in Quaternary geochronology and landscape evolution studies. Cosmogenic nuclides are produced in rocks and sediment due to reactions induced by cosmic rays. Landforms ranging in age from a few hundred years to tens of millions of years can be dated (depending on rock or landform weathering rates) by measuring nuclide concentrations. In this paper the history and theory of surface exposure dating are reviewed followed by an extensive outline of the fields of application of the method. Sampling strategies as well as information on individual nuclides are discussed in detail. The power of cosmogenic nuclide methods lies in the number of nuclides available (the radionuclides ^{10}Be , ^{14}C , ^{26}Al , and ^{36}Cl and the stable noble gases ^3He and ^{21}Ne), which allows almost every mineral and hence almost every lithology to be analyzed. As a result focus can shift to the geomorphic questions. It is important that obtained exposure ages are carefully scrutinized in the framework of detailed field studies, including local terrace or moraine stratigraphy and regional morphostratigraphic relationships; as well as in light of independent age constraints.

[Oberflächenexpositionsdatierungen mittels kosmogener Nukliden]

Kurzfassung: Im letzten Jahrzehnt hat sich die Methode der Oberflächendatierung mittels kosmogener Nuklide zu einer leistungsfähigen Methode in der Quartärchronologie und quantitativen Landschaftsanalyse entwickelt. Kosmogene Nuklide werden durch kosmische Strahlung in Fest- und Lockergestein gebildet. Die Konzentrationen der kosmogener Nuklide kann mittels Massenspektrometrie ermittelt werden. Dies ermöglicht - je nach Verwitterungsrate - die Datierung von Landschaftselementen und Landschaftsformen mit Altern zwischen einigen 100 Jahren bis über 10 Millionen Jahren. Neben einem Abriss der historischen Entwicklung und Theorie der Oberflächendatierung mittels kosmogener Nuklide enthält dieser Artikel eine ausführliche Übersicht der zahlreichen Anwendungsgebiete dieser Methode. Probenahme-strategien und die Eigenheiten der einzelnen Nuklide werden im Detail besprochen. Die Vielzahl der mit dieser Methode in den verschiedensten Mineralien bestimmbar Nuklide (Radionuklide ^{10}Be , ^{14}C , ^{26}Al und ^{36}Cl und Edelgase ^3He und ^{21}Ne) erlaubt die Beprobung und Analyse verschiedenster Lithologien. Der erreichte hohe Entwicklungsstand der Methode erlaubt es den Fokus auf die eigentlichen geomorphologischen Fragestellungen zu legen. Die Sensitivität der kosmogener Oberflächendatierungsmethode muss trotzdem sorgfältig im Rahmen ausführlicher Feldstudien erfolgen, wie zum Beispiel durch die Analyse von lokalen und regionalen Terrassen- oder Moränenstratigraphien oder durch den Vergleich mit anderen Datierungsmethoden.

Keywords: cosmogenic radionuclides, cosmogenic noble gases, surface exposure dating

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1 Introduction and history

Cosmogenic nuclides build-up predictably with time in minerals exposed to cosmic rays. Therefore measuring their concentrations allows determination of how long rocks or sediment have been exposed at or near the surface of the Earth (LAL 1991; GOSSE & PHILLIPS 2001). At present the most commonly utilized nuclides are the radionuclides ^{10}Be , ^{14}C , ^{26}Al , and ^{36}Cl and the stable noble gases ^3He and ^{21}Ne (Table 1). Because of the wide variety of nuclides available (with different half-lives or stable) and the fact that they can be measured in a variety of minerals a broad spectrum of geomorphological problems can be addressed (Fig. 1).

By measuring the concentrations of cosmogenic nuclides rock surfaces themselves can be directly dated. This is a unique and powerful tool never before available to geomorphologists.

Bedrock landforms, fluviially- or glacially-polished bedrock surfaces, fault footwall faces, and landslide bedrock detachment surfaces can be sampled and dated directly. There is no other method where this is possible. Sedimentary units such as moraines, landslide deposits, fluvial terraces, debris flows or alluvial fans can be directly dated by sampling boulder surfaces or by taking samples made up of numerous clasts. Sites unsuitable for luminescence techniques may be dated with cosmogenic nuclide methods, for example sediments that have not been exposed to light long enough or coarse-grained material. In the past geomorphology has relied on radiocarbon for the indirect dating of landforms. The upper age limit for radiocarbon dating of organic material is about 50 ka, whereas under certain conditions landform ages on the order of tens of millions years can be measured with exposure dating (SCHÄFER et al. 1999; DUNAI et al. 2005). For radiocarbon

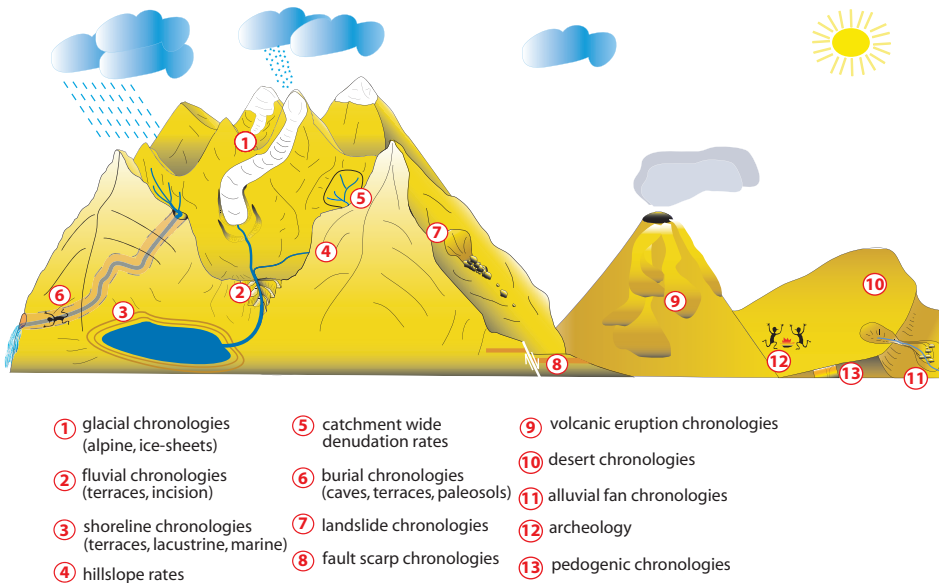


Fig. 1: Schematic diagram showing the various landforms that can be dated and approaches for using cosmogenic nuclides to address questions of timing and rates of landscape change (see also BIERMAN & NICHOLS 2004).

Abb. 1: Überblick über die Landschaftsformen und Landschaftselemente die mittels kosmogener Nuklide datiert oder deren Prozessraten quantifiziert werden können (siehe auch BIERMAN & NICHOLS 2004).

dating organic material must be present in the sediment, which is not the case in most high alpine early Holocene or Lateglacial deposits. Finally organic material included in sediments is not dating the landform itself, the relationship of the organic material to the landform can never be unequivocally established. Entrained wood fragments are simply older than the enclosing sediment.

Because of the presence of higher concentrations (production rates are higher in space than they are on earth) cosmogenic nuclides were initially investigated in lunar and meteorite samples already beginning in the 1950's (FINKEL & SUTER 1993 and references therein). Earliest attempts to measure cosmogenic nuclides in terrestrial rocks were made by DAVIS & SCHAEFFER (1955) and SRINIVASAN (1976). DAVIS & SCHAEFFER (1955) used low-level decay counting to measure ^{36}Cl in a high-Cl phonolite from unglaciated high elevation sites in the Rocky Mountains. In 1976, Srinivasan analyzed the cosmogenic noble gas ^{126}Xe in barite from a sedimentary unit (SRINIVASAN 1976) and highlighted the potential of noble gases in surface exposure dating (NIEDERMANN 2002). Routine measurement of cosmogenic nuclides and use of cosmogenic nuclides for determi-

nation of exposure histories and erosion rates only became possible after the development of accelerator mass spectrometry (AMS), and the construction of high sensitivity noble gas mass spectrometers between 1970 and 1980. These technical developments opened the way to measurements of exceedingly low nuclide concentrations. In the late 1970s first accelerator measurements were reported for ^{14}C (BENNETT et al. 1977; NELSON et al. 1977), ^{10}Be (RAISBECK et al. 1978), ^{26}Al (RAISBECK et al. 1979) and ^{36}Cl (ELMORE et al. 1979). Early measurements of cosmogenic nuclides in rock and sediment samples were made using ^3He (KURZ 1986), ^{10}Be and ^{26}Al (KLEIN et al. 1982), ^{21}Ne (MARTI & CRAIG 1987), ^{36}Cl (KUBIK et al. 1984; PHILLIPS et al. 1986), and ^{14}C (JULL et al. 1992). In 1991, LAL presented detailed terrestrial cosmic ray systematics, setting the standards for production rate and scaling formalisms as well as discussing potential applications and promising nuclide combinations (LAL 1991). GOSSE & PHILLIPS (2001) published a comprehensive review of cosmogenic nuclide methods in the Earth Sciences including the appropriate equations. Further summaries are given by NISHIZUMI et al. (1993), CERLING & CRAIG (1994a), BIERMAN et al. (2002), MORRIS

Table 1: Nuclide characteristics Mineral-Nuclide overview.

Tab. 1: Überblick über die kosmogenen Nuklide und deren Charakteristika.

Nuclide	Half-life	Other isotopes	meas. method	Target elements	Production rate atoms/g.yr*	Advantages/minerals used	Disadvantages
^{10}Be	1.51 Myr	^9Be	AMS	O Si	5	quartz resistant and ubiquitous	low production rate, ^{10}B interference in AMS generally restricted to quartz (no meteoric ^{10}Be)
^{26}Al	716 kyr	^{27}Al	AMS	Si	31	high production rate quartz resistant and ubiquitous	restricted to quartz (low Al) accurate determination of ^{27}Al required
^{36}Cl	301 kyr	^{35}Cl , ^{37}Cl	AMS	Ca K ^{35}Cl	composition dep. e.g. 10 granite e.g. 20 limestone	low detection limit (low AMS Bkgd) any rock type, silicates & carbonates	complicated production ^{36}S interference in AMS accurate determination of total Cl required determination of rock composition required
^{14}C	5.73 kyr	^{12}C , ^{13}C	AMS	O	16	useful for short timescales quartz resistant and ubiquitous	short half-life atmospheric ^{14}C contamination
^3He	stable	^4He	static mass spec.	many	120	high production rate useful for long time scales pyroxene, olivine	diffuses out of quartz or volcanic groundmass radiogenic/nucleogenic/magmatic correction beware pre-exposure
^{21}Ne	stable	^{20}Ne , ^{22}Ne	static mass spec.	Mg Si	20	useful for long time scales, > 50 ka quartz, olivine, pyroxene	nucleogenic/magmatic correction high air background possible beware pre-exposure

*production rates (GOSSE & PHILLIPS 2001)

et al. (2002), NIEDERMANN (2002), COCKBURN & SUMMERFIELD (2004). In this review we focus on surface exposure dating of landforms and deposits. The use of cosmogenic nuclides methods in Archeology is discussed in AKÇAR et al. (2008a). Burial dating is dealt with in the paper by DEHNERT & SCHLÜCHTER (2008). For readers interested in the use of cosmogenic nuclides to determine landscape denudation rates we refer to VON BLANCKENBURG (2005), GRANGER (2006; 2007) and GRANGER & RIEBE (2007).

2 Production of cosmogenic nuclides

The Earth is constantly being bombarded by cosmic rays (primarily protons, alpha particles as well as other heavier nuclei). The primary cosmic ray flux consists of galactic and solar cosmic rays with the former clearly being more important for production of cosmogenic nuclides in minerals (LAL & PETERS 1967). Interactions of high-energy cosmic ray particles with nuclei in the Earth's atmosphere result in a cascade of secondary particles (especially neutrons). This means that in traversing the atmosphere, the flux of cosmic ray particles first increases (in the first few kilometers) then steadily decreases. Consequently, nuclide production rates in rocks at the surface of the earth are lower at lower altitude. Primary galactic cosmic rays (especially the lower energy part) are modulated by the Sun's magnetic field. High solar activity reduces the primary cosmic ray flux. Cosmic ray particles are deflected by the Earth's predominantly-dipole magnetic field. The magnetic field impedes and deflects particles with lesser energies at lower latitudes. As a consequence cosmic ray intensity and therefore nuclide production is higher at the poles than at the equator. At sea level production rates are about half at the equator compared to what they are at the poles. The altitude and latitude dependence of cosmogenic nuclide production rates reflects this modulation of the cosmic ray flux by the earth's magnetic field and the atmosphere (Fig. 2) (GOSSE & PHILLIPS 2001, MASARIK et al. 2001). Several physical models have been presented for the scaling of

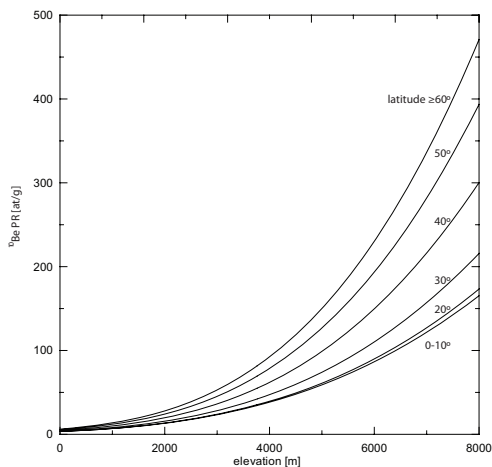


Fig. 2: Production of rate of ^{10}Be in quartz as a function of geomagnetic latitude and altitude (based on STONE 2000). The production rates have been normalized to sea level and high latitude. At low latitude, production rates are lower than at high latitude. Production rates increase exponentially with increasing altitude.

Abb. 2: Produktion von ^{10}Be in Quarz in Abhängigkeit der geomagnetischen Position und Höhe über Meeresspiegel (nach STONE 2000). Die Berechnungen sind skaliert auf Meeresspiegellhöhe und hohe geomagnetische Breiten. In niederen Breiten ist die Produktionsrate kleiner als in hohen Breiten. Die Produktionsraten steigen exponentiell mit der Höhe über dem Meer an.

production rates from their sea level and high latitude values to the altitude and latitude of the sampling site (LAL 1991; DUNAI 2000; 2001; STONE 2000; DESILETS & ZREDA 2001; PIGATI & LIFTON 2004; MUZIKAR 2005; DESILETS et al. 2006a) (see detailed discussion in BALCO et al. 2008). Key differences between the systems include the method of modelling the variation of the neutron flux with altitude and how past changes in the magnetic field are incorporated (BALCO et al. 2008). At present most studies use the production rates and scaling system of STONE (2000) for ^{10}Be and ^{26}Al . This allows a certain degree of intercomparison. Although the suitability of this protocol for samples at high altitude is under discussion (see BALCO et al. 2008). In any case it is crucial when

calculating an exposure age to use the same scaling formalities as were used for the original production rate calculations. The University of Washington/CRONUS-Earth website (<http://hess.ess.washington.edu/math/>) allows the consistent comparison of data from different sites as well as presents detailed discussion of the differences and similarities of the various scaling systems (BALCO et al. 2008). Another website where these calculations can be done is cosmocalc.googlepages.com (VERMEESCH 2007). Cosmogenic nuclides are produced within minerals by several reactions (Table 2). These include spallation, muon-induced reactions and low-energy (epithermal and thermal) neutron capture (LAL & PETERS 1967). During spallation a secondary cosmic ray neutron with sufficient energy hits the target element and one or more particles are ejected from the nucleus leaving the cosmogenic nuclide in the target element's site in the mineral lattice. Cosmogenic nuclides are also produced through interactions of muons with the target element (capture of slow muons and stopping of fast muons). Because they are reacting with target elements in rocks the flux of secondary cosmic ray particles decreases (is attenuated) with depth into

rock or sediment. Production due to spallation decreases exponentially with depth (LAL 1991) (Fig. 3). Muons are less apt to react than neutrons thus they penetrate deeper into the Earth's surface and production due to muons becomes increasingly important below depths of about 2 m (in a rock of density 2.7 g cm⁻³). The decrease of production depth profile for muons is described by equations with several exponentials (SCHALLER et al. 2001; 2002; GRANGER & SMITH 2000) (see also DEHNERT & SCHLÜCHTER 2008). ³⁶Cl differs somewhat from the other nuclides. In rocks with sufficient natural Cl (³⁵Cl and ³⁷Cl) ³⁶Cl is produced through low-energy (thermal and epithermal) neutron capture on ³⁵Cl and to a lesser extent on ³⁹K (Table 2); in addition to spallation and muon-related reactions. Low-energy neutrons can diffuse back out of a rock surface (FABRYKA-MARTIN 1988). As a result of this neutron leakage, production of ³⁶Cl by neutron capture peaks about 20 cm down into the rock (Fig. 4). The shape of this curve depends on rock composition (proportion of target elements) and density (FABRYKA-MARTIN 1988; LIU et al. 1994; PHILLIPS et al. 2001). Rocks with little natural Cl will have negligible low-energy neutron capture

Table 2: Main reactions to produce cosmogenic nuclides on the Earth.

Tab. 2: Hauptreaktionen kosmogener Nuklide an der Erdoberfläche.

Target element	Spallation	Negative-muon capture	Low-energy neutron capture
O	¹⁶ O(n,2pn) ¹⁴ C	¹⁶ O(μ ⁻ ,pn) ¹⁴ C	
	¹⁶ O(n,4p3n) ¹⁰ Be	¹⁶ O(μ ⁻ ,3p3n) ¹⁰ Be	
Si	²⁸ Si(n,p2n) ²⁶ Al	²⁸ Si(μ ⁻ ,2n) ²⁶ Al	
	²⁸ Si(n,2α) ²¹ Ne		
Na	²³ Na(n,p2n) ²¹ Ne		
Mg	²⁴ Mg(n,α) ²¹ Ne		
Al	²⁷ Al(n,3p4n) ²¹ Ne		
K	³⁹ K(n,2p2n) ³⁶ Cl	³⁹ K(μ ⁻ ,p2n) ³⁶ Cl	³⁹ K(n,α) ³⁶ Cl
Ca	⁴⁰ Ca(n,3p2n) ³⁶ Cl	⁴⁰ Ca(μ ⁻ ,2p2n) ³⁶ Cl	
Cl			³⁵ Cl(n,γ) ³⁶ Cl

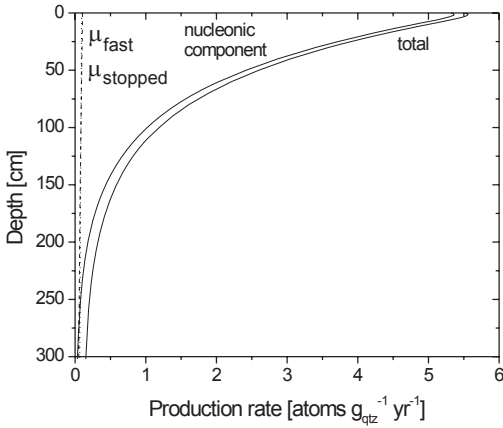


Fig. 3. Production rate of ¹⁰Be in quartz as a function of depth at sea level and high latitude. The total production is a composite of the production by neutron spallation, stopped muons, and fast muons.

Abb. 3: Produktion von ¹⁰Be in Quarz in Abhängigkeit der Tiefe, skaliert auf Meeresspiegelhöhe und hohe geomagnetische Breiten. Die Gesamtproduktion ist die Summe aus Spallations-, gestoppten und schnellen Muonenreaktionen.

production and the production depth profile will be spallation-dominated and resemble the curve for ¹⁰Be (Fig. 3). Complete equations for calculating depth profiles are given in GOSSE & PHILLIPS (2001).

Shielding by surrounding hillslopes and mountains reduces the cosmic ray flux to the sampling site leading to lower production. Similarly dipping of the rock surface decreases the production rate. Shielding corrections are done following DUNNE et al. (1999) (see also GOSSE & PHILLIPS 2001). Calculation of these correction factors is also possible through the internet (<http://hess.ess.washington.edu/math/>).

Cosmogenic nuclides [atoms g⁻¹] build-up in an exposed rock surface according to the following equation:

$$C(t) = \frac{P(0)}{\lambda + \frac{\rho \epsilon}{\Lambda}} \left(1 - e^{-\left(\lambda + \frac{\rho \epsilon}{\Lambda}\right)t} \right) + C_{in} e^{-\lambda t}$$

Eq. 1

where P₍₀₎ [atoms g⁻¹ a⁻¹] is the production rate at the sampling site, t [a] is the exposure age of the surface, λ [a⁻¹] is the decay constant, ρ [g cm⁻³] is the density of the irradiated material, ε [cm a⁻¹] is the erosion rate, and Λ [g cm⁻²] is the attenuation length. C_{in} [atoms g⁻¹] is the nuclide concentration already present at the beginning of exposure and is called inheritance. The presence of inherited nuclide concentrations will yield ages older than the true age (see also below).

Radionuclides build-up in exposed minerals until secular equilibrium is reached (saturation) which happens after about three to four half-lives. At secular equilibrium the number of nuclides produced per unit time is equivalent to the number that decays; the concentration is at steady state. This is seen in Figure 5 where the the upper concentration growth curve flattens out. When the rock surface is eroding (weathering), both erosion and decay lead to loss of the nuclide. Saturation is reached earlier (after a shorter exposure period) (Fig. 6).

In the first instance, exposure ages are calculated assuming zero inheritance and without erosion such that Eq. 1 simplifies to:

$$C(t) = \frac{P(0)}{\lambda} \left(1 - e^{-\lambda t} \right)$$

Eq. 2

Eq. 2 is solved for t. The production rate is scaled for site latitude (and when past magnetic field changes are considered longitude), altitude (Fig. 2), as well as, sample thickness, and topographic shielding. The attenuation length is 157 g cm⁻² and the density of crystalline rock is 2.7 g cm⁻³ (GOSSE & PHILLIPS 2001). As shown in Figure 7 the measured nuclide concentration is a direct measure of the length of the exposure period.

Where field evidence indicates that rock surface weathering (erosion) has been significant, an age is calculated by using an assumed or measured erosion rate with the following equation (Eq. 1 without the inheritance term):

$$C(t) = \frac{P(0)}{\lambda + \frac{\rho \epsilon}{\Lambda}} \left(1 - e^{-\left(\lambda + \frac{\rho \epsilon}{\Lambda}\right)t} \right)$$

Eq. 3

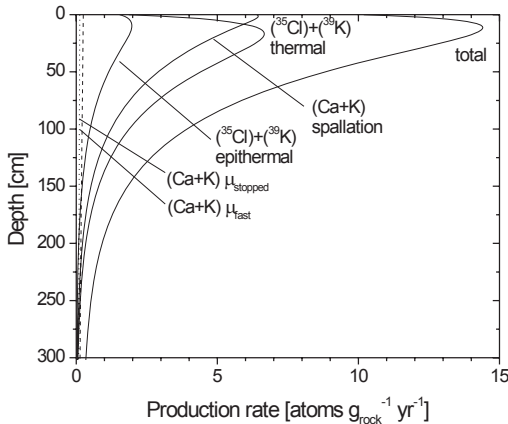


Fig. 4. Production rate of ^{36}Cl in granite with 200 ppm of Cl as a function of depth. The production rates are for sea level and high latitude. The total production is a composite of production by neutron spallation, thermal and epithermal neutrons, stopped and fast muons. The maximum production by thermal neutrons is highest at about 20 cm depth in a rock with a given density of 2.7 g cm^{-3} . Diffusion of thermal neutrons back out of the rock in the uppermost few centimeters causes this reduction in production rate. Variation of production rates with depth calculated after LIU et al. (1994), STONE et al. (1996, 1998), and PHILLIPS et al. (2001).

Abb. 4: Produktion von ^{36}Cl in einem Granit (200 ppm Cl) in Abhängigkeit der Tiefe, skaliert auf Meeresspiegelhöhe und hohe geomagnetische Breiten. Die Gesamtproduktion ist die Summe aus Neutronenspallations-, thermischen und epithermischen Neutronenreaktionen, sowohl als auch gestoppten und schnellen Muonenreaktionen. Das Maximum der Produktion durch thermische und epithermische Neutronenreaktionen wird in einer Tiefe von 20 cm erreicht (bei einer Dichte von 2.7 g cm^{-3}). Die Diffusion thermischer Neutronen in den oberen Zentimetern des Profils verringert die Gesamtproduktion (berechnet nach LIU et al. (1994), STONE et al. (1996, 1998), and PHILLIPS et al. (2001)).

Crystalline rock surface erosion rates are typically less than 10 mm ka^{-1} (COCKBURN & SUMMERFIELD 2004). For spallation-dominated nuclides rock surface erosion has the effect of lowering the concentrations so that the measured age is younger than the true period

of exposure. For ^{36}Cl exposure dating this is not always the case depending on the relative contribution of production due to low-energy neutron capture and the size of the hump in the depth profile (Fig. 4).

The upper and lower age limits for each nuclide are set by a combination of geological and methodological factors. The lower age limit for ^{10}Be , ^{26}Al and ^{36}Cl is limited predominantly by measurement capabilities. A certain amount of carrier must be added and a certain ratio of radionuclide to stable nuclide must be attained to be above the background levels. Under appropriate conditions exposure ages in the range of several hundred years can be determined (DAVIS et al. 1999). Upper age limits are constrained by the nuclide half-life and the weathering rate of the rock surface (Fig. 6). In areas where rock weathering is slow exposure ages up to several million years can be determined with ^{10}Be or ^{26}Al ; to tens of millions with ^{21}Ne . On the other hand, when a granitic rock surface is weathering at a rate of 5 mm ka^{-1} no ages older than several hundred thousand years can be calculated. But the calculated ages may be minimum ages for that landform. Although the noble gases are stable, they too are affected by erosion and will reach pseudo-saturation just like the radionuclides (Fig. 6).

Often additional information can be gleaned about the history of a rock surface by measuring more than one nuclide (LAL 1991; NISHIZUMI et al. 1993; BIERMAN et al. 1999; GOSSE & PHILLIPS 2001). As Eq. 3 contains two unknowns, the age and the erosion rate, using two nuclides can in some settings allow for the determination of both. One way to view two nuclide data (typically Al/Be, Ne/Be) is with the erosion island (or banana) plot (KLEIN et al. 1986; LAL 1991) (Fig. 8, 9). An important use of the erosion island plot is to readily distinguish data points that represent surfaces that have undergone continuous simple or single stage exposure (plot on or near the erosion island) versus those that reflect complex exposure involving burial (plot below the Al/Be erosion island, above the Ne/Be erosion

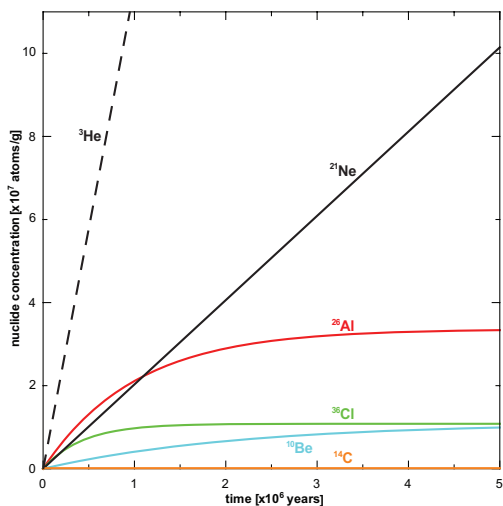


Fig. 5: Increase in concentration of the radionuclides ^{10}Be , ^{26}Al , ^{36}Cl and the stable nuclides ^3He and ^{21}Ne with time. Secular equilibrium, where production of radionuclides equals radioactive decay, is approached after 3–4 half-lives. The secular equilibrium concentration sets the limit of the maximum exposure age that can be determined with a given radionuclide.

Abb. 5: Anstieg der Konzentration der Radionuklide ^{10}Be , ^{26}Al , ^{36}Cl und Edelgase ^3He und ^{21}Ne in Abhängigkeit von der Zeit. Ein Gleichgewichtszustand, bei dem die kosmogene Radionuklidproduktion im Gleichgewicht mit dem radioaktiven Zerfall ist, wird nach 3–4 Halbwertszeiten erreicht. Die Gleichgewichtskonzentration bestimmt die maximal mögliche Altersspanne bei der Datierung mit Radionukliden.

island, respectively; Fig. 8, 9) (see DEHNERT & SCHLÜCHTER 2008). Spalling of slabs of rock of tens of centimeters thick may lead to data that plots inside the banana or in the complex field below the banana (BIERMAN et al. 1999; KOBER et al. 2007). The minimum exposure time represented by data plotting below the erosion island is made up of continuous exposure along the outer line (bold black line) followed by burial with complete shielding (zero production). But an infinite number of periods of exposure and burial are possible which require more time (cf. BIERMAN et al. 1999). Indeed data points for rocks or sediment that

were exposed then buried can arrive back inside the erosion island after a long enough period of (re)exposure. The erosion island plot can be used to estimate both exposure age and erosion rate, especially at sites where erosion rates are very low and where rocks have been exposed longer than 100 ka, for example in desert environments. For slowly eroding arid and hyperarid environments the use of ^{21}Ne (stable) measured in quartz in concert with ^{10}Be provides increased sensitivity for older, more slowly eroding surfaces (Fig. 9).

The basic assumptions that are implicit in the use of in situ-produced cosmogenic nuclides to address problems of landscape evolution are:

- the half-life of the radionuclide is known,
- production pathways and production rates including their variation in space (including with depth into the rock or sediment) are known,
- the initial nuclide concentration (inheritance) is zero or can be determined or estimated,
- the mineral has remained a closed system, i.e. there has been no gain or loss of the nuclide except due to production or decay (or through erosion).

3 Specific nuclide characteristics

3.1 Radionuclides

AMS (accelerator mass spectrometry) is used to determine concentrations of long-lived radionuclides ^{10}Be , ^{14}C , ^{26}Al , ^{36}Cl by measuring ratios relative to a standard material. In AMS interfering isobars or like-mass molecules are separated at the ion source (when they do not produce negative ions), through mass discrimination with magnetic or electrostatic analyzers (as in traditional mass spectrometry), during stripping and/or during detection (FINKEL & SUTER 1993).

Quartz is used in nearly all ^{10}Be studies. This is because it is a ubiquitous, resistant mineral that can be consistently cleaned of meteoric ^{10}Be . Meteoric ^{10}Be is produced in the atmosphere. Its presence in the analyzed mineral separate

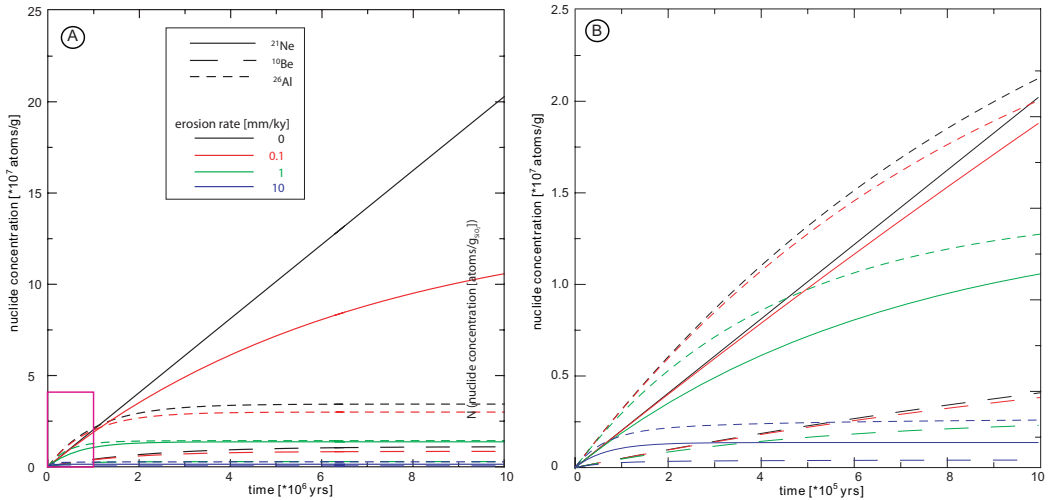


Fig. 6: Increase of the concentrations of ^{10}Be , ^{26}Al , and ^{21}Ne with time taking into account different erosion rates of the exposed surface. Secular equilibrium, where radionuclide gain due to production equals loss due to radioactive decay and erosion, is approached earlier for more rapid erosion rates. A blow-up for the region 0 to 1 Ma is shown.

Abb. 6: Anstieg der Konzentration der Radionuklide ^{10}Be , ^{26}Al und ^{21}Ne in Abhängigkeit von der Zeit und variierenden Erosionsraten an der Erdoberfläche. Ein Gleichgewichtszustand, bei dem die kosmogene Radionuklidproduktion im Gleichgewicht mit radioaktivem Zerfall und der Abnahme durch Erosion ist, wird früher erreicht, je höher die Erosionsrate ist (A). (B) zeigt einen detaillierten Ausschnitt von (A) im Zeitfenster von 0 bis 1 Million Jahre.

would lead to spurious age results. Minerals other than quartz have been tried; for example olivine and pyroxene (NISHIZUMI et al. 1990; BLARD et al. 2008). It is important to note that in contrast to quartz, these minerals chemically weather to clay minerals, consequently problems removing meteoric ^{10}Be have been reported (SEIDL et al. 1997; IVY-OCHS et al. 1998a). Several groups have tried to measure ^{10}Be in carbonate rocks (BRAUCHER et al. 2005); the affinity of ^{10}Be for clay minerals poses a significant obstacle (MERCHEL et al. 2008). The use of ^{26}Al is restricted to minerals with low ^{27}Al content. Too much ^{27}Al would yield a $^{26}\text{Al}/^{27}\text{Al}$ ratio too low to be measured with AMS. Conveniently, quartz satisfies the requirements for both ^{10}Be and ^{26}Al so that both can be extracted by dissolving a single quartz mineral separate. Pure quartz is obtained by selective chemical dissolution in a hot ultrasonic bath and/or on a shaker table (KOHL & NISHIZUMI 1992). Most

other minerals dissolve faster than quartz in a dilute HF (ca. 4%) solution. A carrier of ^9Be (about half a milligram in solution) is added to the pure quartz mineral separate which is then completely dissolved with concentrated HF and HNO_3 . Be and Al are separated and purified with ion exchange methods and selective pH precipitations (KOHL & NISHIZUMI 1992; IVY-OCHS 1996; VON BLANCKENBURG et al. 1996; BIERMAN et al. 2002) (see also <http://depts.washington.edu/cosmolab/chem>). Although it has a higher production rate (Table 1), one disadvantage of ^{26}Al is that a separate accurate measurement (with its own uncertainties) is required to determine the ^{27}Al content of the quartz. Ratios of $^{10}\text{Be}/^9\text{Be}$ or $^{26}\text{Al}/^{27}\text{Al}$ are measured with AMS.

Because Cl is hydrophylic contaminating meteoric ^{36}Cl (or ^{37}Cl or ^{35}Cl) can be removed with rinsing procedures. Under most conditions any rock type or mineral separate can be used

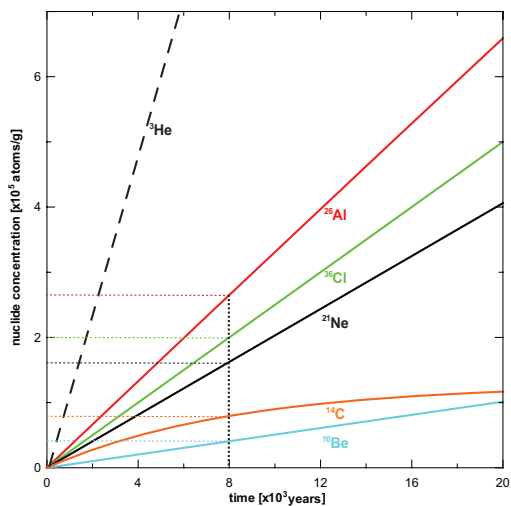


Fig. 7: Close-up of the region 0 to 20,000 years of exposure, illustrating the direct relationship between measured nuclide concentration and exposure age.

Abb. 7: Detaillierter Ausschnitt für das Zeitfenster von 0 bis 20000 Jahren, welcher die direkte Abhängigkeit des Anstiegs der Nuklidkonzentration von der Dauer der Exposition darstellt.

for ^{36}Cl exposure dating. On the other hand if secondary minerals which include meteoric ^{36}Cl precipitate (for example secondary calcite) then the system is no longer closed. Sample preparation procedures for ^{36}Cl are given in ZREDA (1994), IVY-OCHS (1996), STONE et al. (1996, 1998), IVY-OCHS et al. (2004) and DESILETS et al. (2006b) (see also <http://depts.washington.edu/cosmolab/chem/>). Crushed rock samples are first leached several times to release any non-in situ produced Cl. Several milligrams of carrier (in solution) of known isotopic composition (pure ^{35}Cl , ^{37}Cl , or a mixture of both) are added. Carbonate rocks are dissolved with HNO_3 and silicate rocks with HF . Sulfur is removed by precipitation of BaSO_4 (^{36}S interferes with AMS measurement of ^{36}Cl). A crucial improvement in ^{36}Cl methodology is the implementation of isotope dilution. By adding a spike of known isotopic composition but different from the natural ratio of about 3:1 (^{35}Cl : ^{37}Cl), both the total rock Cl concentration and ^{36}Cl can be determined in

a single target using an AMS set-up, through the measurement of $^{37}\text{Cl}/^{35}\text{Cl}$ as well as $^{36}\text{Cl}/\text{Cl}$ (ELMORE et al. 1997; IVY-OCHS et al. 2004). This has led to marked improvements in both precision and accuracy in ^{36}Cl results (DESILETS et al. 2006b). Because ^{36}Cl is produced from spallation of Ca and K and low-energy neutron capture on ^{35}Cl and ^{39}K and because each rock has a different chemistry, production rates for ^{36}Cl must be calculated individually. In addition to determination of major element oxides, concentrations of B, Gd, and Sm must be determined. These elements are strong neutron absorbers and influence the proportion of low-energy neutrons that are available for neutron capture reactions on ^{35}Cl and ^{39}K . U and Th concentrations are needed to correct for background (subsurface non-cosmogenic) neutron-capture ^{36}Cl production (FABRYKA-MARTIN 1988).

^{14}C is produced in quartz by spallation of ^{16}O (JULL et al. 1992; LIFTON et al. 2001; YOKOYAMA et al. 2004). Atmospheric ^{14}C contamination is removed from the surfaces and crevices of the quartz grains with acid etching (similar to that used for ^{10}Be studies; KOHL & NISHIZUMI 1992) and preheating. About five grams of quartz are flux melted in a flow of oxygen (LIFTON et al. 2001) to produce CO_2 , which is converted to graphite using standard procedures. Carbon ratios are measured with AMS. The possibility to use a gas ion source for direct analysis of the CO_2 with AMS is an exciting development. Because of difficulties in sample preparation and extraction, ^{14}C is currently used infrequently but has great potential.

3.2 Noble gases

For noble gas studies, mineral separates (tens to hundreds of milligrams) are pre-concentrated using heavy liquids and/or magnetic separation. Quartz is separated using the method of KOHL & NISHIZUMI (1992). Because of the small amounts necessary mineral separates can also be hand-picked under a binocular microscope. Noble gases are extracted and measured with high sensitivity static noble gas mass spectrometry (NIEDERMANN 2002). Mineral separates

Fig. 8: Plot of $^{26}\text{Al}/^{10}\text{Be}$ ratios versus ^{10}Be concentration showing the evolution of the $^{26}\text{Al}/^{10}\text{Be}$ ratios with time. Continuously exposed, non-eroding surfaces evolve along the bold black line. Continuously exposed surfaces eroding with steady-state erosion follow the trajectories (blue lines) that splay downward from the no-erosion line. The red line joins points of final $^{26}\text{Al}/^{10}\text{Be}$ ratios with the given erosion rates but is not an evolution line. The prescribed area is called the «steady-state erosion island». Samples that plot below the steady-state erosion island experienced a more complex exposure that involves periods of burial (see DEHNERT & SCHLÜCHTER 2008). Samples may also plot below the erosion island if thick slabs have spalled off.

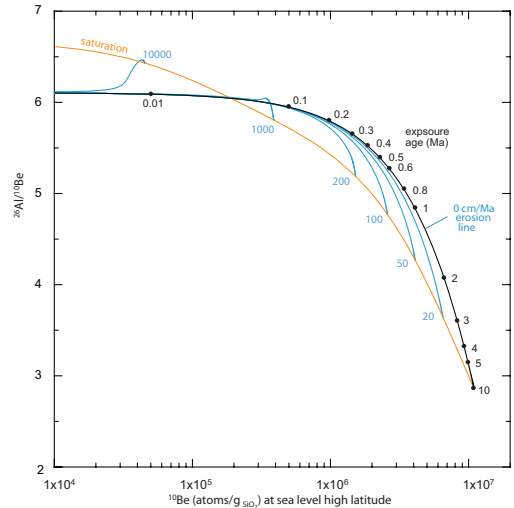


Abb. 8: Darstellung des Verhältnisses von $^{10}\text{Be}/^{26}\text{Al}$ zur ^{10}Be -Konzentration, welches die Entwicklung des Verhältnisses von $^{10}\text{Be}/^{26}\text{Al}$ mit der Zeit aufzeigt (als Funktion der Konzentration). Im Falle einer kontinuierlichen Exposition und Null-Erosion würde eine Probe auf der schwarzen, dicken Linie liegen. Kontinuierliche Exposition mit einer konstanten Erosionsrate würde eine Probe auf Trajekturen (blaue Linien) bewegen, die von der Null-Erosionslinie abzweigen. Die rote Linie verbindet die Endpunkte dieser Trajekturen (Gleichgewichtsendpunkt, Verhältnis von $^{10}\text{Be}/^{26}\text{Al}$ ist konstant) und ist artifizuell. Das Feld zwischen Null-Erosionslinie und der artifizuellen Linie wird als "Gleichgewichts-Erosionsinsel" bezeichnet. Probenpunkte unterhalb dieser Insel haben eine komplexe Expositionsgeschichte, einschliesslich Perioden mit Abschirmung zur kosmischen Strahlung (siehe auch DEHNERT & SCHLÜCHTER 2008). Probenpunkte unterhalb der "Gleichgewichts-Erosionsinsel" können auch durch eine schalige Abspaltung vom Festgestein verursacht werden.

are heated to various temperature steps until the final fusion of the sample. Step-wise heating or crushing *in vacuo* are performed to discriminate non-cosmogenic components (nucleogenic, radiogenic, or trapped). Gas purification (separation from e.g., CO_2 , water vapour, and heavy noble gases) is accomplished by a combination of cryogenic traps and hot getters.

^3He is produced by spallation reactions of nearly all elements. ^3He is not measured in quartz as it diffuses out (TRULL et al. 1991; BROOK & KURZ 1993). Olivine and pyroxene phenocrysts and microphenocrysts are retentive for ^3He (KURZ et al. 1990; CERLING & CRAIG 1994b; LICCIARDI 1999; FENTON et al. 2001; MARCHETTI et al. 2005). The presence of radiogenic ^3He may limit studies to rocks with young crystallization ages (cf. NIEDERMANN 2002; WILLIAMS et al. 2005).

^{21}Ne is produced by spallation of Si, Al, Mg and Na (Table 1) in quartz, olivine, pyroxene and sanidine. An important advantage of ^{21}Ne is that

in contrast to ^3He it can be measured in quartz (GRAF et al. 1991; STAUDACHER & ALLÈGRE 1991). Thus three cosmogenic nuclides, ^{10}Be , ^{26}Al and ^{21}Ne can be determined on aliquots of a single quartz mineral separate (HETZEL et al. 2002a,b; KOBER et al. 2007). As ^{14}C is measured in quartz the potential to measure four nuclides in quartz exists. ^{21}Ne is also measured in pyroxene and olivine (MARTI & CRAIG 1987; STAUDACHER & ALLEGRE 1991; POREDA & CERLING, 1992; BRUNO et al. 1997; SCHÄFER et al. 1999). KOBER et al. (2005) showed that the volcanic potassium feldspar sanidine retains ^{21}Ne . By using sanidine one can measure ^{10}Be , ^{36}Cl and ^{21}Ne in aliquots of the same mineral separate (IVY-OCHS et al. 2007b). Interferences due to trapped and/or nucleogenic (both non-cosmogenic) neon isotopes (HETZEL et al. 2002a) can in most cases be deconvoluted using stepwise heating of mineral separates and plotting data on the three isotope (^{20}Ne , ^{21}Ne , ^{22}Ne) plot (GRAF et al. 1991; NIEDERMANN 2002).

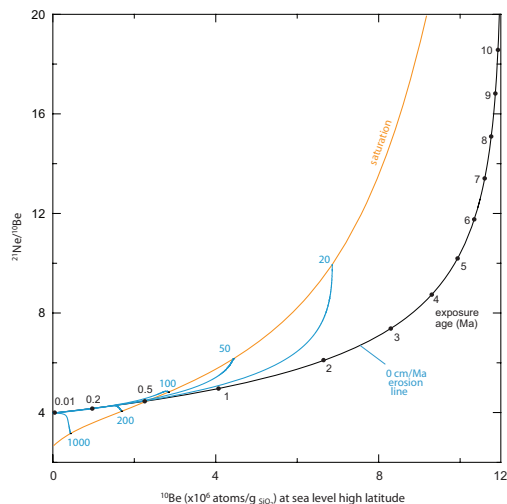


Fig. 9: Plot of $^{21}\text{Ne}/^{10}\text{Be}$ ratios versus ^{10}Be concentration showing the evolution of the $^{21}\text{Ne}/^{10}\text{Be}$ ratios with time. Continuously exposed, non-eroding surfaces evolve along the bold black line. Continuously exposed surfaces eroding with steady-state erosion follow the trajectories (blue lines) that splay upward from the no-erosion line. The red line joins points of final $^{21}\text{Ne}/^{10}\text{Be}$ ratios with the given erosion rates but is not an evolution line. The prescribed area is called the «steady-state erosion island». Samples that plot below the steady-state erosion island experienced a more complex exposure that involves periods of burial. Samples may also plot above the erosion island if thick slabs have spalled off.

Abb. 9: Darstellung des Verhältnisses von $^{21}\text{Ne}/^{10}\text{Be}$ zur ^{10}Be -Konzentration, welches die Entwicklung des Verhältnisses von $^{21}\text{Ne}/^{10}\text{Be}$ mit der Zeit aufzeigt (s. Legende Fig. 8).

4 Sampling considerations

Cosmogenic nuclides can be used to address a variety of problems in the Earth Sciences (Fig. 1). Two factors are key in deciding which cosmogenic nuclide (Table 1) is best suited to the geological/geomorphological problem at hand: i) the half-life of the nuclide (or stable), and ii) the bedrock geology (mineralogy) of the study area. As described above certain nuclides can only be measured in certain minerals.

Depositional landforms such as moraines (Fig. 10), fluvial terraces or alluvial fans can be dat-

ed with cosmogenic nuclides. For the exposure date to represent as close as possible the true formation or abandonment age of the landform, the sampled object (boulder, clasts or bedrock) surface must have i) undergone single-stage exposure (no pre-exposure/inheritance), ii) been continuously exposed in the same position (not shifted), iii) never been covered, and iv) undergone only minimal surface weathering or erosion (not spalled). Sampling large (>1.5 m high), broad, and flat-topped boulders located in stable positions (moraine crest; flat tread of a terrace) best satisfy these conditions. Large boulders have a smaller chance of having sunk in or shifted. To avoid edge effects (MASARIK et al. 2000; MASARIK & WIELER 2003), flat surfaces at least 20-30 cm away from an edge are sampled. Similarly pointed knobs are avoided. Careful scrutiny of candidate boulders may help one to rule out those that have spalled or toppled. The base of boulders is inspected for flakes, although this may be difficult to judge in forested settings. Sample surfaces are evaluated for post-depositional weathering, when there are step-like surfaces the highest one is sampled. A hammer and chisel, a saw or a drill are used to take several hundred grams to 1 kg of the upper few centimeters of the rock surface (Fig. 10). The amount of material needed depends on location (i.e. the local production rate), estimated exposure age and mineralogy (% of the mineral needed) of the sampled surface. Because of the possibility of the data scattering, where enough suitable boulders are present at least three to five different boulders are sampled.

In most data sets the prevalence of «too young» ages far overshadows «too old» ages. This has been determined based on independent age controls for the landform (e.g. ^{14}C age for a landslide) or intercomparison of dates from a single or related landform (when inversions occur etc). Most commonly, weathering of the sampled rock surface and/or degradation of the landform leads to «too young» ages. The magnitude of this effect is related to the age, location (climate, treeline location with respect to the landform), lithology (mineralogy and texture)

and morphology of the landform (flatness of the upper surface steepness of the margins). \diamond Too old \diamond ages arise when the initial nuclide concentration in the sampled rock surface was not zero

(inheritance). In boulders, inheritance can be acquired in bedrock exposures before the boulder falls onto the glacier or before the landslide, or when the boulder is reworked from older depo-

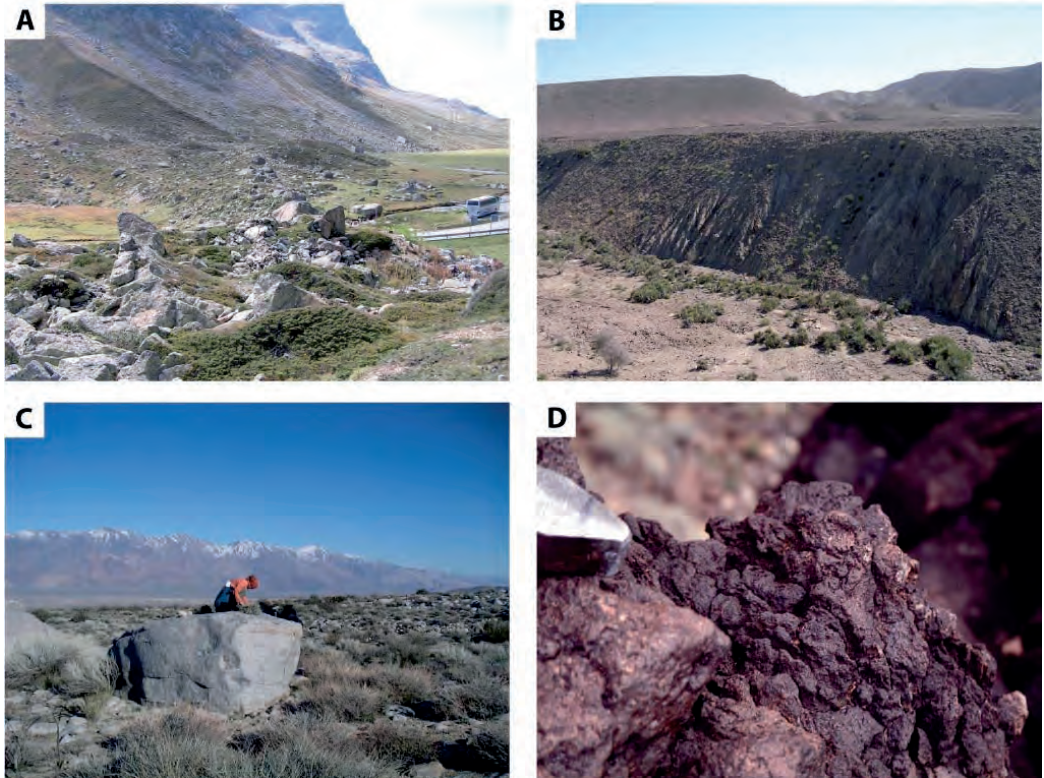


Fig. 10: Examples of exposure dated landforms:

- A. View along the boulders making up the latero-terminal moraine complex at Julier Pass, left lateral moraine is seen in the upper left. Numerous boulders were dated with ^{10}Be , ^{26}Al and ^{36}Cl . Exposure dates indicate moraine stabilization at 11.4 ka (IVY-OCHS et al. 2006),
- B. Terrace levels (T2 in the center, T3 in the background) in the Pishamak area, Makran Range, SE Iran, exposure dated with both ^{10}Be and ^{21}Ne (F. KOBER, unpublished data),
- C. ^{10}Be dated boulder on the Shephard Creek debris-flow fan in Owens Valley (DÜHNFORH et al. 2008),
- D. Pahoehoe flow top from the ca. 90,000 year old (^3He) Bar Ten flow, Arizona (FENTON et al. 2001).

Abb. 10: Beispiele von Landformen die mit kosmogenen Nukliden datiert wurden:

- A: Blick entlang eines Seiten/Endmoränenkomplexes am Julier Pass, linke Seitenmoräne ist im linken oberen Bildausschnitt. Zahlreiche Blöcke wurden mit ^{10}Be , ^{26}Al und ^{36}Cl datiert. Expositionsalter geben ein Alter von 11400 Jahren an, welches als das Alter der Stabilisierung der Moränen interpretiert wird (IVY-OCHS et al. 2006).
- B: Terrassenniveaus (T2 im Zentrum, T3 im Hintergrund des Bildes) im Gebiet um Pishamak, Makran, SE-Iran, datiert mit ^{10}Be und ^{21}Ne (F. KOBER et al. in Bearbeitung).
- C: Blöcke eines Schuttstromes auf einem Fächer des Shephard Creeks, Owens Valley (DÜHNFORH et al. 2008).
- D: Oberer Bereich eines Pahoehoe Lavastroms des Bar Ten Stroms, Arizona, datiert auf ca. 90000 Jahre (FENTON et al. 2001).

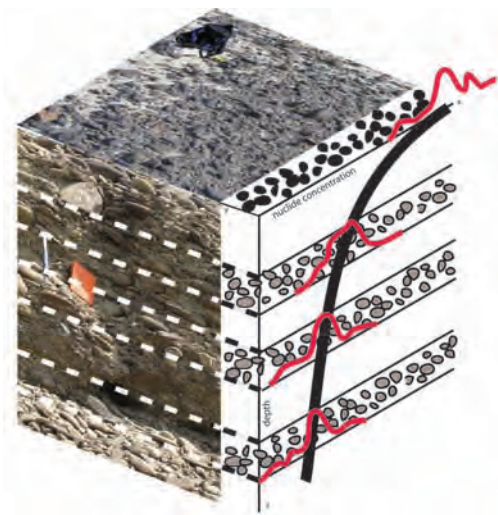


Fig. 11: Schematic sketch of a depth profile (z-axis) and cosmogenic nuclide concentrations (x-axis) in an alluvial sequence deposited in a single event. Red curves show hypothetical frequency distribution of nuclide concentrations of individual clasts, illustrating the need to amalgamate tens of clasts (ANDERSON et al. 1996; REPKA et al. 1997). Amalgamated clast samples taken in certain depth intervals show a decreasing concentration with depth.

Abb. 11: Schematische Darstellung der Nuklidkonzentration (x-Achse) mit der Tiefe (y-Achse) in einer alluvialen Ablagerungssequenz (singuläres Ereignis). Die rote Kurve zeigt die hypothetische Nuklidkonzentrationsverteilung, welche aus einer Anzahl von individuellen Kiesgeröllen gewonnen wurde. Dies zeigt, dass eine Mischung einer größeren Anzahl von Kiesgeröllen zur Datierung nötig ist (ANDERSON et al. 1996, REPKA et al. 1997). Mischproben zeigen eine Abnahme der Nuklidkonzentration mit der Tiefe.

sits. For example boulders can be pushed into new moraines without having their orientation changed (see also IVY-OCHS et al. 2007a).

Where large boulders are not present amalgamated clast samples are analyzed (ANDERSON et al. 1996; REPKA et al. 1997). This method is suitable for older landforms (> 100 ka) that probably never had boulders; for example fluvial terraces. The effects of unrepresentative concentrations (too high or too low) should be smoothed out by the amalgamation of >50 clasts of similar size (several centimeters in diameter) (ANDERSON et

al. 1996; REPKA et al. 1997). Clasts are collected from the flat part of the landform surface well away from modifying channels. In many cases the nuclide concentration due to inheritance is revealed by measuring a depth profile. Amalgamated clast samples are taken every 10-20 centimeters down to 2 meters depth (Fig. 11) (ANDERSON et al. 1996; REPKA et al. 1997; RYERSON et al. 2006). Exposure during hill slope erosion, sediment transport, and storage in terraces may result in inheritance in clasts. Scavenging (reworking) of material from older deposits at higher elevation can also lead to inheritance in clasts (WARD et al. 2005). Where field relationships indicate that the top of the depositional surface has undergone little erosion (flat tread morphology as opposed to convex tops on terraces) or little aggradation (minimal silt layer below the top surface pavement) the shape of the depth curve reveals both age and inheritance (Fig. 12). Appropriate sediment densities must be used to calculate the model curves. Commonly the concentration in the upper several tens of centimeters is constant due to mixing (often bioturbation) (PHILLIPS et al. 1998) (Fig. 12). Thick (more than tens of meters) rapidly deposited units may also be burial dated by measuring the concentrations of two nuclides from samples at greater depth (> 10 m) (GRANGER & MUZIKAR 2001; WOLKOWINSKY & GRANGER 2004; GRANGER 2006) (see DEHNERT & SCHLÜCHTER 2008). Several landscape evolution questions can be addressed by sampling bedrock. Bedrock samples are taken from areas that have never been covered by sediment, ice or snow. This may require sampling a steeply dipping surface and the making of the appropriate shielding corrections (NISHIZUMI et al. 1989; KELLY et al. 2006). In general sampling of steep surface is avoided to circumvent the additional uncertainties associated with the dip correction.

5 Dating of Quaternary landforms

5.1 Glacial landscapes

Surface exposure dating has been used in a broad spectrum of settings in glacial landscapes. This

includes the dating of boulders on moraines (for a recent compilation see REUTHER et al. 2006a), boulders on glacial outwash fans (PHILLIPS et al. 1997), boulders on the former margins of ice-dammed lakes (DAVIS et al. 2006) and boulders deposited during catastrophic outburst of ice-dammed lakes (CERLING et al. 1994; REUTHER et al. 2006b). Glacially-polished bedrock is also analyzed for determining rates of ice retreat, and depth of subglacial erosion. The method of burial dating has great potential, especially

in the dating of old, buried glacial deposits (see DEHNERT & SCHLÜCHTER 2008). BALCO et al. (2005) used ^{26}Al and ^{10}Be ratios in quartz to determine the age of deeply buried paleosols and underlying till units.

5.1.1 Dating of moraines

Moraines record the location of the margins of a glacier in the past. Changes in glacier volume and length themselves reflect changes in tem-

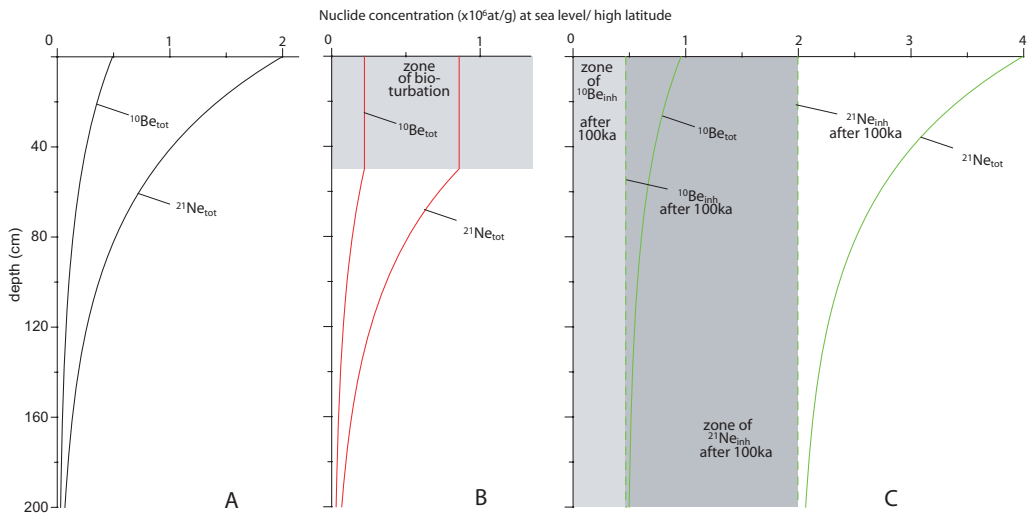


Fig. 12: ^{10}Be and ^{21}Ne concentrations with depth into a rock surface. The nuclide concentration in a sample is a composite of the inherited and the post-depositional nuclide concentration. The inherited concentration can be approximated by measuring several samples from different depths, tens of centimeters apart down to about 2 m.

- ^{10}Be and ^{21}Ne concentrations in a depth profile after exposure for 100 ka with a constant erosion rate of 0.20 mm/ka.
- The upper 50 cm have been mixed by bioturbation resulting in constant nuclide concentrations.
- Curve showing the effect of inheritance on both the ^{10}Be and ^{21}Ne concentrations.

Abb. 12: Entwicklung der Nuklidkonzentration (^{10}Be und ^{21}Ne) in Abhängigkeit von der Tiefe. Die Gesamtnuklidkonzentration ist die Summe aus ererbten Konzentrationen und der Konzentration aus der Produktion nach Ablagerung der Sequenz oder eines vormalig exponierten Tiefenprofils im Festgestein. Die ererbte Nuklidkonzentration kann durch die Beprobung in unterschiedlichen Tiefen (mehrere Proben über eine Tiefe von mindestens 2 m) ermittelt werden.

- ^{10}Be und ^{21}Ne Nuklidkonzentrationen in einem Tiefenprofil nach einer Expositionszeit von 100000 Jahren unter einer konstanten Erosionsrate von 0,20 mm/1000 Jahre.
- Der obere Bereich des Profils (50 cm) ist durch Bioturbation homogenisiert, was zu einer konstanten Nuklidkonzentrationen über dieses Intervall führt.
- Darstellung des Einflusses einer ererbten Nuklidkonzentration auf die ^{10}Be und ^{21}Ne Nuklidkonzentrationen.

perature and precipitation patterns in a region with time (KERSCHNER 2005). Therefore if one can directly date moraines one can construct a chronological structure to past glacier fluctuations and therefore past climatic fluctuations (KERSCHNER & IVY-OCHS 2008). Depending on the detailed structure of the moraine complex the innermost moraine records the onset of glacier downwasting (GOSSE 2005). For example near synchrony of glacier downwasting at the end of the Last Glacial Maximum world-wide has been established with ^{10}Be dating (SCHAEFER et al. 2006). As described above large boulders in stable positions along the moraine crest are sampled preferentially. A boulder must be large and stable enough not to have toppled or shifted and high enough to have protruded above the matrix since moraine deposition (HALLET & PUTKONEN, 1994; PUTKONEN & SWANSON, 2003; PUTKONEN & O'NEAL, 2006). Key limitations include weathering of the boulder surface and degradation of the moraine itself (ZREDA et al. 1994; ZREDA & PHILLIPS, 1995; REUTHER et al. 2006a; IVY-OCHS et al. 2007a). Sampling several small clasts on a young (<20ka) moraine is unlikely to give the depositional age (IVY-OCHS et al. 2007a). Moraines ranging in age from hundreds of years to hundreds of thousands of years in both hemispheres have been dated (e.g. PHILLIPS et al. 1990; IVY-OCHS et al. 1999, 2006; BARROWS et al. 2002; KAPLAN et al. 2004; BRINER et al. 2005; BALCO & SCHAEFER 2006; Akçar et al., 2008b). The older the moraine the greater the spread in ages amongst the exposed boulders. This may make interpretation of the deposition age of moraines that are older than 100 ka difficult (KAPLAN et al. 2005; SMITH et al. 2005). In such cases the oldest age is assumed to be closest to the landform age. But this age may still be a minimum age for the landform. In a suite of ages from a single moraine outliers that are too old reflect inheritance. Boulders that are deposited in moraines may have acquired inheritance in the bedrock setting or because they are reworked from older moraines. For the case of a boulder surface exposed in the bedrock setting, the amount of inheritance is dependent on how long the bedrock surface

was exposed (and the bedrock weathering rate) and how deep the boulder originated inside the bedrock surface (IVY-OCHS et al. 2007a). The effect of this will be greater in younger (Holocene) moraines. Based on a compilation of numerous published moraine boulder exposure ages, PUTKONEN & SWANSON (2003) found that pre-exposure was observed in moraine boulders in only a few percent of cases.

5.1.2 Glacially-modified bedrock surfaces and rates of sub-glacial erosion

Initial deglaciation of the valley bottom and rates of glacier downwasting can be determined by analyzing cosmogenic nuclides in glacially-scoured bedrock surfaces (e.g. roche moutonnées) (GOSSE et al. 1995; GUIDO et al. 2007). However, results from bedrock surfaces should be viewed with caution. If three meters or more of bedrock have not been removed by sub-glacial erosion during the last glaciation, then the rock surface may contain inherited nuclide concentrations. This has been noted where the rock is highly resistant (GUIDO et al. 2007). In the case where the timing of deglaciation is independently known (for example from exposure dating of erratics), the nuclide concentration measured in glacially-scoured bedrock can be used to determine sub-glacial erosion rates. Determined rates are on the order of 0.1 to 1 mm per year (BRINER & SWANSON 1998; COLGAN et al. 2002; FABEL et al. 2004). Direct determination of such rates is only possible with cosmogenic nuclides.

Cosmogenic nuclides have a unique characteristic in that they can be used to elucidate fundamental information about the thermal regime of past ice sheets. Lower ^{26}Al than ^{10}Be concentrations in bedrock reveals areas where ice was frozen to its bed and unerosive (BIERMAN et al. 1999; FABEL et al. 2002; MARQUETTE et al. 2004; STAIGER et al. 2005; SUGDEN et al. 2005; LINGE et al. 2006; PHILLIPS et al. 2006). Production ceased when the surfaces were covered by ice, ^{26}Al decays faster than ^{10}Be thus $^{26}\text{Al}/^{10}\text{Be}$ ratios plot below the erosion island (see also DEHNERT & SCHLÜCHTER 2008).

5.2 Alluvial, lacustrine and marine systems

Determining the age of alluvial landforms (for example fluvial terraces and alluvial or debris-flow fans) provides fundamental information about timing and rates of depositional processes. Constructing an age sequence also allows estimation of the contribution of the various external forcing mechanisms, such as tectonically induced base-level or regional slope changes, climatically (discharge) induced or sediment supply dependent variations. Fluvial or marine terraces; or alluvial fans can be dated using depth profiling and measuring one or more nuclides (samples from the upper 2 meters) (PERG et al. 2001; WARD et al. 2005; RYERSON et al. 2006; FRANKEL et al. 2007) or using burial dating (WOLKOWINSKY & GRANGER 2004) (see also DEHNERT & SCHLÜCHTER 2008).

5.2.1 Wave-cut bedrock platforms and paleoshorelines

Lake and marine paleoshorelines delineate higher water levels and thus record past changes in the balance between precipitation vs. evaporation plus lake drainage and inflow or fluctuations in sea-level, respectively. In order to integrate these periods of lake/sea highstands into existing regional chronologies a time frame is required. Such highstands can be exposure dated in two ways by exposure dating wave-cut bedrock platforms or by determining the age of cobble deposits that mark ancient strandlines. The only way to directly date a wave-cut bedrock platform is with cosmogenic nuclides. Uplifted marine wave-cut platforms in northern Spain were studied by ALVAREZ-MARRÓN et al. (2007) with ^{10}Be , ^{26}Al , and ^{21}Ne . Minimum ages of 1-2 Ma were calculated. Based on a comparison of ^{10}Be and ^{21}Ne data the authors suggested that the surfaces have undergone complex exposure histories and may even be Pliocene in age when the burial time is included. The data of KONG et al. (2007) from wave-cut shore lines of lake Sumxi Co, Western Tibet, agree with regional chronologies; the highest bedrock terrace was formed during enhanced discharge of

melting glaciers around 12 ka. This implies that no inherited nuclides were present. Wave-cut bedrock platforms and associated boulders of the Provo shoreline which delineates the latest Pleistocene extent of Lake Bonneville have been dated with a number of cosmogenic nuclides, including ^3He (CERLING & CRAIG 1994a). At that site consistent ages have been obtained with ^{14}C in quartz (LIFTON et al. 2001) and ^{10}Be making it an excellent site for production rate determination and nuclide cross-calibrations. Timing of abandonment of the Provo shoreline is known from radiocarbon dating (GODSEY et al. 2005). Cobble and pebble deposits that delineate ancient strandlines can be exposure dated either by analyzing individual clasts or by amalgamating tens of clasts together. Because of the dependency on preservation of the strandlines most of these studies are in arid regions. The primary difficulty in dating such deposits is inheritance. Inheritance is recognized when the clast exposure ages are significantly scattered and/or when they are older than the estimated age of the shoreline based on field relationships or correlations with independently dated sections. Inheritance can be acquired in the catchment when the denudation rate is low. Thus the clasts arrived in the lacustrine system with a non zero initial nuclide concentration. Clasts may have been exposed during intermittent storage in river terraces between erosion in the catchment and final deposition in the shoreline. Finally, inheritance may indicate that clasts were moved from shoreline to shoreline and thus do not give the correct age for the strandline from which they were collected. MATMON et al. (2003) dated five individual chert clasts from the ancient strandline of paleo Lake Lisan age scatter and disagreement with independent age estimates due to inheritance points to intermediate storage of clasts.

5.2.2 Alluvial fans

Patterns of alluvial fan deposition record variations of erosional processes in the fan catchment and changes in incision of the fan head over time (DÜHNFORTH et al. 2008). When individual lobes

can be clearly mapped then surface exposure dating is a useful tool for dating of abandoned fan lobes (DÜHNFORH *et al.* 2007). Original depositional forms such as flow snouts and levees are often still present on debris-flow fans that are less than 100 ka old. To date the different fan lobes large boulders in clear position on snout or levee are sampled (BIERMAN *et al.* 1995; ZEHFUSS *et al.* 2001; DÜHNFORH *et al.* 2007). On older alluvial fans degradation of fan surfaces and boulder grusification leads to smoothing out of the original bar and swale morphology. After a long enough period of time (50 ka?) desert pavement of interlocking clasts develops (RYERSON *et al.* 2006) with associated desert varnish. In these cases amalgamated clast samples are analyzed. An important question is whether or not the dates represent the interval of lobe construction or whether they point to timing of fan abandonment. Another serious concern for the dating of alluvial and debris-flow fans is inheritance. Similar to the case of the strandline clasts inheritance can have been acquired in the catchment, in intermediate storage, or as clasts are moved from older to younger lobes. Reflecting the bedrock rezeroing effect of glaciers, inheritance is often greater in fans originating in unglaciated catchments.

5.2.3 Fluvial incision rates

Fluvial incision rates and their variation with time are calculated by using the exposure age and height of fluvial (WARD *et al.* 2005) or strath terraces (BURBANK *et al.* 1996; LELAND *et al.* 1998; PRATT-SITULA *et al.* 2004; REUSSER *et al.* 2004). Boulders or amalgamated clast samples on debris-flow deposits on ancient bedrock straths have been dated to determine incision rates (FENTON *et al.* 2004; MARCHETTI & CERLING 2005). Based on ^{10}Be in samples from three strath terraces REUSSER *et al.* (2004) reported that incision rates of the Susquehanna River (eastern U.S.A.) more than doubled to 0.5 mm a^{-1} during the last glaciation (32 to 16 ka). In Taiwan ^{36}Cl measured in samples from fluvially-sculpted limestone channel walls in Taroko Gorge indicate incision rates of 26 mm a^{-1} (SCHALLER *et al.*

2005). Steep gorge walls may provide a more continuous record of incision than strath terraces as the latter is a step-like sequence. In any case it is often difficult to verify that strath terraces were never covered by sediment during their exposure histories. Past incision rates can also be calculated by combining burial ages of cave sediments with the height of the cave above the active river channel (GRANGER *et al.* 1997; STOCK *et al.* 2004) (see DEHNERT & SCHLÜCHTER 2008).

5.3 Tectonic and mass movement studies

Cosmogenic nuclides can be used to determine rates of tectonic activity in two ways i) by dating landforms that have been offset by movement along faults and ii) by dating bedrock fault surfaces directly.

5.3.1 Dating of offset landforms

Slip rates on strike-slip faults or rates of uplift on normal faults have been determined by taking the age and offset distances of moraines (LASSERRE *et al.* 2002; BROWN *et al.* 2002), fluvial terraces (HETZEL *et al.* 2002a) and alluvial fans (BIERMAN *et al.* 1995; SIAME *et al.* 1997; BROWN *et al.* 1998; VAN DER WOERD *et al.* 1998; ZEHFUSS *et al.* 2001; RITZ *et al.* 2003; MÉRIAUX *et al.* 2005; RYERSON *et al.* 2006; FRANKEL *et al.* 2007). Cautious interpretation of field evidence for offset distances (piercing point, offset terrace risers vs. treads; offset stream channels) is important (MÉRIAUX *et al.* 2004).

5.3.2 Direct dating of fault surfaces

Limestone bedrock fault surfaces are dated directly with ^{36}Cl (ZREDA & NOLLER 1998; MITCHELL *et al.* 2001; BENEDETTI *et al.* 2002; 2003; PALUMBO *et al.* 2004). Suitable fault surfaces are several meters high several kilometers long, fresh, and uneroded. Samples are taken at centimeter intervals along the exposed fault surface often with a circular saw. In principle, the recurrence interval of earthquakes along the fault segment can be reconstructed based on step changes in ^{36}Cl concentrations determi-

ned in tens of samples from a single fault face. Earthquakes that occurred as recently as only a thousand years ago are dated. In principle, such a study is also possible with ^{10}Be in quartz, but may be limited by sample preparation time for the large number of samples required. The age of the fault surface that can be determined is controlled by the karst weathering rate of the limestone and is generally restricted to those less than tens of thousands of years old.

5.3.3 Landslides

Landsliding is an important process for the modification of valley slopes and cross profiles, as well as overall landscape denudation especially in steep terrain. By determining the timing of recurrence intervals we can begin to understand the mechanisms of large-scale downslope movement of rock, and the interplay between various possible causes and triggers. These include inherited tectonic structures, debuttressing following retreat of glaciers, climate (precipitation, temperature, permafrost extent etc.), and/or seismic events. Several catastrophic landslides have been dated using cosmogenic nuclides: the Koefels landslide in Austria (IVY-OCHS et al. 1998b), the Flims landslide in Switzerland (IVY-OCHS et al. 2008), the Fernpass landslide in Austria (PRAGER et al. 2008), the Almtal landslide in Austria (VAN HUSEN et al. 2007); the Beinn Alligin rock avalanche, in Scotland (BALLANTYNE & STONE 2004), the Blackhawk landslide in California, U.S.A. (NICHOLS et al. 2006), several slides in Argentina (HERMANN et al. 2001; 2004), and northern India (BARNARD et al. 2001), as well as both deep-seated and shallow landslides in Hong Kong (SEWELL et al. 2006).

Although new rock surfaces are created during mass movements, exposure dating of landslides can have mixed results (IVY-OCHS et al. 2008). Each possible sampling site (bedrock detachment zones, sliding planes, large boulders) has its own advantages and disadvantages. In the breakaway zone recent rockfall can be difficult to identify. Sliding planes with low dip angles may yield too young ages due to past coverage

by snow and/or vegetation. Because of the movement mechanisms of landslides, rocks from the outer surface of the pre-slide bedrock frequently end up as boulders on top of the deposit. As a result, «too old» exposure ages with respect to independent age controls occur much more frequently in landslide boulders than in moraine boulders (NICHOLS et al. 2006; SEWELL et al. 2006; IVY-OCHS et al. 2008). Nevertheless exposure dating is an irreplaceable tool in landslide studies, often there is no other way to date the mass movement. Although repeated sliding may be identified during mapping, it is impossible to determine the recurrence interval without direct dating. In the Alps, post-Last Glacial Maximum landsliding in the early Lateglacial cannot be dated with radiocarbon because of a lack of organic material. Holocene landslides can be dated when entrained trees or wood fragments are discovered or by dating basal sediments of impounded lakes. Intercomparison of dating results from various methods allows better constraining of the event age.

5.4 Volcanic sequences

Based on detailed mapping relative eruption sequences are constructed. Lava flows can often be ^{14}C dated with entrained charcoal or by analyzing underlying burnt soil, when such material is found. Time of eruption of lava flows and domes are dated with K/Ar and $^{40}\text{Ar}/^{39}\text{Ar}$ (KELLEY 2002). Not all flows are suitable for Ar/Ar dating: i) high-K minerals may be lacking, ii) excess Ar or iii) Ar loss may be a problem. As erupted volcanic rocks are newly formed surfaces with no pre-exposure, their initial nuclide concentration is zero, they are well-suited for surface exposure dating. The most challenging aspect is to unequivocally identify the original eruptive surface to sample. This may be established by the presence of primary eruptive features such as spatter (Fig. 10), frothy glassy texture of the cooling rinds on flows or bombs, vesicles near the tops of flows, or the ropey texture of pahoehoe flows (CERLING & CRAIG 1994a; FENTON et al. 2001; PHILLIPS 2003). Mafic lava flows with olivine and/or

pyroxene phenocrysts or microphenocrysts are dated with ^3He and/or ^{21}Ne (KURZ et al. 1990; ANTHONY & POTHS 1992; CERLING & CRAIG 1994b; LICCIARDI et al. 1999; 2006; FENTON et al. 2004; WILLIAMS et al. 2005; DUFFIELD et al. 2006). ^{21}Ne and ^{10}Be in quartz and/or sanidine phenocrysts have been used to date ignimbrites (welded tuffs) (LIBARKIN et al. 2002; KOBER et al. 2005). Volcanic rocks of intermediate composition lacking quartz or olivine or pyroxene or flows lacking phenocrysts completely can be exposure dated with ^{36}Cl (PHILLIPS 2003; ZREDA et al. 1993). This also affords the opportunity for intercomparison between the eruptive (K–Ar or $^{40}\text{Ar}/^{39}\text{Ar}$ age) and the exposure age (FENTON et al. 2001; PHILLIPS 2003). For the dating of landforms (moraines, flood deposits, etc.) comprised of volcanic rocks ^3He and ^{36}Cl are well suited.

5.5 Ancient Landscapes

It is only since the advent of cosmogenic nuclides that rock surfaces in the arid and hyper-arid deserts of southern Africa (COCKBURN et al. 1999; 2000; FLEMING et al. 1999; BIERMAN & CAFFEE 2001), south-central Australia (BIERMAN & TURNER 1995; BIERMAN & CAFFEE 2002; BELTON et al. 2004), western South America (NISHIZUMI et al. 2005; KOBER et al. 2007) and Antarctica (SUMMERFIELD et al. 1999; SUGDEN et al. 2005) can be directly dated. The long-lived radionuclides (^{10}Be and ^{26}Al) and especially the noble gases (^3He and ^{21}Ne) are well suited to dating landforms that have been exposed for millions of years. Combining nuclides, for example ^{10}Be and ^{26}Al or ^{10}Be and ^{21}Ne , is critical to check for continuous exposure and rule out intermittent coverage. Studies using two or more nuclides indicate that many of the ancient bedrock surfaces in the deserts have been exposed continuously, but are nevertheless weathering, albeit remarkably slowly. In most cases only minimum ages can be determined. Maximum erosion rates are calculated by assuming that nuclide concentrations are in steady state. At several of these sites erosion rates range down to less than 1 mm

kyr $^{-1}$ (COCKBURN & SUMMERFIELD 2004). With cosmogenic nuclide methods, fundamental information about the age and mode of formation of ancient landscapes has been gained. High nuclide concentrations measured in Australia led to the conclusion that inselbergs are direct descendents of early Cenozoic or even Mesozoic landforms (BIERMAN & CAFFEE 2002). Combined fission-track and low cosmogenic nuclide-derived erosion rates across the Namibian escarpment indicate that great escarpments do not form by rapid (and ongoing) escarpment retreat, but must have formed early on after continental break-up (COCKBURN et al. 2000). ^{10}Be in combination with ^{26}Al data showed that many surfaces in Antarctica have experienced single-stage, continuous exposure with remarkably low erosion rates for at least the last several million years (NISHIZUMI et al. 1991; BROOK et al. 1995; IVY-OCHS et al. 1995; SUMMERFIELD et al. 1999; MATSUOKA et al. 2006). The high nuclide concentrations found in rocks in the Dry Valleys Antarctica provide irrefutable support for the premise that the East Antarctic Ice Sheet has been a stable feature since its inception (SCHÄFER et al. 1999; ACKERT & KURZ 2004). Measured pre-Pleistocene apparent exposure ages and corresponding low denudation rates characterize the deserts of northern Chile, despite active uplift (DUNAI et al. 2005; KOBER et al. 2007; NISHIZUMI et al. 2005).

6 Summary and outlook

The ability to use cosmogenic nuclides to determine how long minerals have been exposed at the surface of the earth provides an unrivaled tool for determining ages of landforms and rates of geomorphic processes. Depending on rock and landform weathering rates, landforms ranging in age from a few hundred years to tens of millions of years can be dated. Because of this unique capability, the variety of applications of cosmogenic nuclides will continue to grow. Concern about methodological uncertainties, such as those associated with the production rates, the site latitude and

altitude scaling factors, as well as the effect of past changes in the Earth's magnetic field, has led to the establishment of an international consortium made up of CRONUS-Earth (www.physics.purdue.edu/cronus) and CRONUS-EU (www.cronus-eu.net). Analysis of artificial targets and samples from natural sites with independent age control are underway to refine production rates. Scaling factors are being evaluated with neutron monitors and analysis of same age natural samples taken along altitudinal transects (for example lava flows). Numerical modeling is being used to constrain production rates and scaling factors both now and in the past. The half-lives of radioactive nuclides must be accurately known. In the case of ^{10}Be , two different half-lives have been published, 1.51 and 1.34 Ma (GRANGER 2006; NISHIZUMI et al. 2007). When these factors are better constrained the errors of the final ages will be closer to the range of the AMS and noble gas mass spectrometry measurement uncertainties (of the order of 1-4 %). With improved knowledge of production rates and their scaling to the site, the precision of obtained ages will improve. But the accuracy of the ages remains a question of geological uncertainties. The degradation of both rock surfaces and the landforms with time imposes clear limitations on the time range and accuracy of dating. Similarly, the natural variability of samples depends on landform morphology and its age. Obtained exposure ages must be evaluated individually for conformity with field relationships, including local terrace or moraine stratigraphy and regional morphostratigraphic relationships; as well as with independent age constraints for the same or correlative features. For older landforms (more than a hundred thousand years) measurement of multiple cosmogenic nuclides can reveal fundamental information, such as non-continuous exposure, which must be factored into interpretations (ALVAREZ-MARRÓN et al. 2007; KOBER et al. 2007). Cosmogenic nuclides provide a powerful and multifaceted tool whose potential has yet to be fully realized. But this power is tempered with the need for careful sampling based on detailed field mapping.

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Sediment burial dating using terrestrial cosmogenic nuclides

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Abstract: Burial dating using in situ produced terrestrial cosmogenic nuclides is a relatively new method to date sediments and quantify geomorphological processes such as erosion, accumulation and river incision. Burial dating utilises the decay of previously in situ produced cosmogenic nuclides and can be applied to sedimentary deposits such as cave fillings, alluvial fans, river terraces, delta deposits, and dunes. Using the established $^{10}\text{Be}/^{26}\text{Al}$ nuclide pair allows numerical dating of quartz bearing material from ~100 ka to 5 Ma, where other dateable material is often unavailable. To date, a number of studies have demonstrated the successful application of in situ produced cosmogenic nuclides in various scientific disciplines, such as Quaternary geology, geomorphology and palaeoanthropology. However, insufficiently defined physical properties such as nuclide half lives and complex depth dependent nuclide production rates result in relatively large uncertainties. Nevertheless, burial dating represents a promising method for determining numerical ages.

[Datierung des Überdeckungsalters mit Hilfe von terrestrischen kosmogenen Nukliden]

Kurzfassung: Die Methode der Bestimmung des Überdeckungsalters mit Hilfe von in situ produzierten terrestrischen kosmogenen Nukliden stellt ein verhältnismäßig neues Datierverfahren dar. Es ermöglicht die Altersbestimmung von Sedimenten und damit die Quantifizierung von geomorphologischen Prozessen, wie Erosion, Akkumulation und Flusseintiefung. Das Verfahren bedient sich dabei des Zerfalls von zuvor in situ produzierten kosmogenen Nukliden und kann auf sedimentäre Ablagerungen wie Höhlenfüllungen, Schwemmfächer, Flussterrassen, Deltaschüttungen und Dünen angewendet werden. Durch die Verwendung des erprobten $^{10}\text{Be}/^{26}\text{Al}$ Nuklidpaares erlaubt die Methode die Bestimmung eines numerischen Alters von quarzführendem Material über einen Zeitbereich von ~100 ka bis 5 Ma. In diesem Zeitabschnitt ist datierfähiges Material für andere Methoden oftmals nicht oder nur unzureichend vorhanden. Viele Studien konnten bereits die erfolgreiche Anwendung von in situ produzierten kosmogenen Nukliden in den verschiedensten wissenschaftlichen Bereichen, darunter zum Beispiel in der (Quartär)Geologie, Geomorphologie und Paläoanthropologie, belegen. Dennoch können die zur Zeit nur ungenügend genau bestimmten physikalischen Größen, wie zum Beispiel die Nuklidhalbwertszeiten oder die tiefenabhängigen Nuklidproduktionsraten zu vergleichsweise großen Unsicherheiten führen. Trotz dieser Nachteile stellt die Methode eine vielversprechende Möglichkeit der numerischen Altersbestimmung dar.

Keywords: cosmogenic nuclides, burial dating, numerical age determination, sediment, geochronology

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1 Introduction

Sedimentary archives, for example marine, terrestrial and glacial deposits, provide information regarding the climatic and environmental history as well as the tectonic development of a given area. In this context, the youngest part of the Earth's history, the Neogene and the Quaternary, are of particular interest. This time have been characterised by massive mountain forming processes, large changes in the temperature of oceans and the atmosphere as well as by important biological evolution, particularly with the appearance of early and modern hominids (ASFAW et al. 1999; CLARK et al. 2003; DÉZES et al. 2004; RAVELO et al. 2004; GIBBARD et al. 2005). All this information, however, is only of minor value if it can not be integrated into a global chronological framework. Only with reliable dating can such comparisons be made and the dating of sediments of the past million years is, therefore, one of the most important tasks in modern Quaternary research.

Dating of terrestrial sediments over long time periods, however, can often be highly complex, imprecise or even impossible. Available methods, for example radiocarbon and luminescence dating, cover up to 50 ka and a few 100 ka, respectively, only a relatively short part of the Quaternary (PREUSSER et al. 2008, HAJDAS 2008). We are presenting here a brief summary on recent developments in using terrestrial cosmogenic nuclides for dating the burial age of sediments. Burial dating is based on the assumption that rocks that have been exposed to cosmic radiation for a given time are enriched with various cosmogenic radionuclides (Fig. 1). When these rocks, or their erosional products, are shielded from cosmic radiation, nuclide production ceases and the radionuclides decay according to their individual half lives (Fig. 2). Shielding may be due to transportation of sediment into a cave, by covering with overburden or by deposition in a deep water body (Fig. 3). By measuring the concentration of two nuclides (at least one radionuclide), it is possible to date the time elapsed since shielding.

Cosmogenic nuclides are produced by nuclear reactions in the Earth's atmosphere as well as in the upper parts of the Earth's crust. These nuclear reactions are initiated by high energy secondary cosmic radiation (LAL & PETERS 1967). The development of accelerator mass spectrometry (AMS) over the past few decades has enabled the detection of very small amounts of cosmogenic nuclides (FINKEL & SUTER 1993) and is fundamental to the effective use of cosmogenic nuclides in modern geosciences.

The radionuclides ^{10}Be and ^{26}Al are good candidates for burial dating: both are produced in situ in quartz and possess relatively long half lives. Quartz is one of the most common minerals in the Earth's crust and so is present in almost all sedimentary deposits. The long half lives of both radionuclides enable dating over a time period of 5 Ma and, in addition to this, the nuclide production ratio has been quantified and is independent of latitude, altitude, depth below surface and time (GOSSE & PHILLIPS 2001). For these reasons, the method of burial dating is independent of production rate changes in time and space (LAL & ARNOLD 1985; KLEIN et al. 1986; LAL 1991), which causes most of the inaccuracies in surface exposure dating (GOSSE & PHILLIPS 2001; IVY-OCHS & KOBER 2008).

The principle of burial dating was proposed by LAL & ARNOLD (1985) and underwent a major revision by LAL (1991). The first application of burial dating published (KLEIN et al. 1986) was able to show, with the analysis of the $^{26}\text{Al}/^{10}\text{Be}$ -ratio, that the Libyan Desert Glass Field was occasionally covered by shifting sand dunes, although it was not possible at this time to give discrete burial ages for the sampled glass. GRANGER et al. (1997) were the first to date a deposition event by using the $^{26}\text{Al}/^{10}\text{Be}$ -ratio. They determined river down-cutting rates for the past 1.5 Ma using relocated sediments from cave fillings. Since these first applications a few studies have demonstrated the successful application of burial dating. In addition to the more application focused studies, reviews by GRANGER & MUZIKAR (2001) and more recently by GRANGER (2006) provide the basic knowledge needed for age calculations in various burial scenarios.

As already mentioned, the method of burial dating with cosmogenic nuclides is based on the decay of radionuclides. This requires dependable accuracy in half life determination and precision of the nuclide measurement itself. The half life of ^{26}Al of 0.702 ± 0.056 Ma (MIDDLETON et al. 1983), 0.705 ± 0.024 Ma (NORRIS et al. 1983), or 0.716 ± 0.032 Ma (RIGHTMIRE et al. 1958) is well defined and widely accepted in the cosmogenic nuclide community. However, the traditionally recognised half life of ^{10}Be of 1.51 ± 0.06 Ma (HOFMANN et al. 1987) is still undergoing discussion. Some authors (e.g. PARTRIDGE et al. 2003; HÄUSELMANN & GRANGER 2005; GRANGER et al. 2006) believe that this figure is too high and suggest a shorter ^{10}Be half life of ~ 1.34 Ma (MIDDLETON et al. 1993). NISHIIZUMI et al. (2007) support this, having recently re-evaluated the commonly used ICN (ICN Chemical & Radioisotope Division)

and NIST (National Institute of Standards and Technology) reference material, suggesting that the ^{10}Be half life should be lowered to 1.36 ± 0.07 Ma. On the other hand, FINK & SMITH (2007) also re-evaluated the same material but hesitated to lower the ^{10}Be half life, stating that the direct and accurate specific activity measurement of the parent solution of both standards is needed to calculate the ^{10}Be half life, but this is not yet available.

2 In situ production of cosmogenic ^{10}Be and ^{26}Al

The Earth's atmosphere is undergoing permanent bombardment by primary cosmic radiation. This high energy nucleon radiation originates mainly in the Milky Way (with $E \approx 1 - 10^{10}$ GeV), with a much smaller fraction descended from beyond our galaxy ($E < 10^{20}$ eV) (GOSSE & PHILLIPS

Table 1: Constants for production rate and burial age calculations with in situ produced ^{10}Be and ^{26}Al extracted from quartz minerals. SLHL = sea level, high latitude ($> 60^\circ$)

Tab. 1: Zusammenstellung der Konstanten für die Produktionsraten- und Altersbestimmung mit in situ produziertem ^{10}Be und ^{26}Al aus Quarz. SLHL (sea level, high latitude) = Meeresspiegel, hohe Breiten ($> 60^\circ$)

Constant	^{10}Be	^{26}Al	reference
half life $t_{1/2}$ (Ma)	1.36 ± 0.07 * 1.51 ± 0.06 ^	0.705 ± 0.024 #	* NISHIIZUMI et al. (2007) ^ HOFMANN et al. (1987) # NORRIS et al. (1983)
total surface production rate P_{SLHL} at SLHL (atoms $\text{g}^{-1} \text{a}^{-1}$)	5.1 ± 0.3	31.1 ± 1.9	STONE (2000)
negative muonic surface production rate $P_{1\mu^-}$ at SLHL (atoms $\text{g}^{-1} \text{a}^{-1}$)	0.096	0.723	GRANGER & SMITH (2000)
negative muonic surface production rate $P_{2\mu^-}$ at SLHL (atoms $\text{g}^{-1} \text{a}^{-1}$)	0.021	0.156	GRANGER & SMITH (2000)
fast muonic surface production rate P_{μ^+} at SLHL (atoms $\text{g}^{-1} \text{a}^{-1}$)	0.026	0.192	GRANGER & SMITH (2000)
nucleonic surface production rate P_{N} at SLHL (atoms $\text{g}^{-1} \text{a}^{-1}$)	4.957	30.029	with $P = P_{\text{N}} + P_{1\mu^-} + P_{2\mu^-} + P_{\mu^+}$
nucleon attenuation length Λ_{N} (g cm^{-2})	160 ± 10		GRANGER & SMITH (2000)
negative muon attenuation length $\Lambda_{1\mu^-}$ (g cm^{-2})		738.6	GRANGER & SMITH (2000)
negative muon attenuation length $\Lambda_{2\mu^-}$ (g cm^{-2})		2688	GRANGER & SMITH (2000)
fast muon attenuation length Λ_{μ^+} (g cm^{-2})		4360	GRANGER & SMITH (2000)

2001). Interactions of incoming primary radiation with atoms of the Earth's atmosphere result in low-energy particles, traditionally known as "secondary cosmic radiation" ($E \approx 100$ MeV). For the in situ production of ^{10}Be and ^{26}Al , both at the rock surface and subsurface level, there are two kinds of relevant particles: nucleons (neutrons and protons) and muons (fast and negative). According to strict nomenclature rules, muons belong to tertiary cosmic radiation, as they are a byproduct of decaying secondary cosmic radiation pions (π mesons) (GOSSE & PHILLIPS 2001). Cosmogenic ^{10}Be and ^{26}Al are generated in quartz (^{16}O as the main target for ^{10}Be and ^{28}Si transforms to ^{26}Al) by spallation reactions (neutrons), negative muon capture, and a cascade of reactions called Coulomb-reactions (fast muons). These processes are explained in detail in LAL & PETERS (1967) and GOSSE & PHILLIPS (2001), among others. Nuclide production in near surface material is dominated by nucleons ($\sim 97.5\%$) (HEISINGER 1998) and changes with increasing depth, as muons display greater penetration depth as a result of their lower reactivity. The nucleonic production as a function of depth can be shown as a simple exponential law (LAL 1991; BROWN et al. 1992; GOSSE & PHILLIPS 2001):

$$P_{10N}(z) = P_{10N}(0)e^{-\rho z/\Lambda_N} \quad [1]$$

$$P_{26N}(z) = P_{26N}(0)e^{-\rho z/\Lambda_N} \quad [2]$$

with production rate in depth in atoms $\text{g}^{-1} \text{a}^{-1}$ ($P(z)$), scaled surface production rate in atoms $\text{g}^{-1} \text{a}^{-1}$ ($P(0)$), depth in cm (z), attenuation length in g cm^{-2} (Λ) and density of the overburden in g cm^{-3} (ρ). The index of 10 and 26 represents the nuclides ^{10}Be and ^{26}Al , respectively, and N nucleonic production. Constants for all equations are listed in Tab. 1.

The nuclide production by muons is valid within a depth range of 200 – 5000 g cm^{-2} and is well described by the sum of three exponential functions (GRANGER & SMITH 2000). At shallower depths nuclide production by muons is negligible, as nucleonic production is predominant. Nuclide production by negative muon

capture is described in the first two terms. The last one represents production by Coulomb-reactions (GRANGER & SMITH 2000; GRANGER & MUZIKAR 2001):

$$P_{10\mu}(z) = P_{10/1\mu-}(0)e^{-\rho z/\Lambda_{1\mu-}} + P_{10/2\mu-}(0)e^{-\rho z/\Lambda_{2\mu-}} + P_{10\mu+}(0)e^{-\rho z/\Lambda_{\mu+}} \quad [3]$$

$$P_{26\mu}(0) = P_{26/1\mu-}(0)e^{-\rho z/\Lambda_{1\mu-}} + P_{26/2\mu-}(0)e^{-\rho z/\Lambda_{2\mu-}} + P_{26\mu+}(0)e^{-\rho z/\Lambda_{\mu+}} \quad [4]$$

The index μ represents muonic production, + and – show fast and negative muons respectively.

The scaled surface production rate, sometimes called local production rate, is dependent on altitude above sea level and latitude, due to the effects of the Earth's magnetic field on the se-

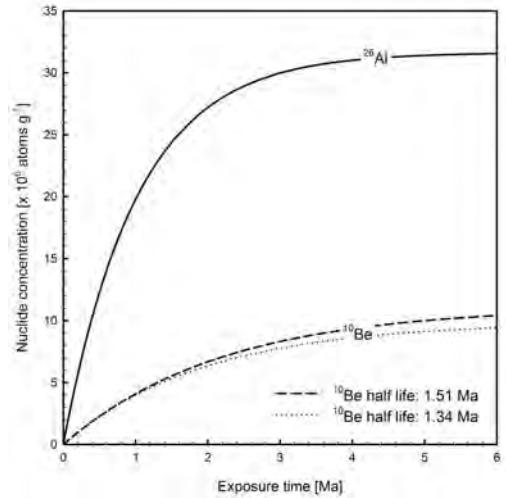


Fig. 1: Increasing nuclide concentration of ^{10}Be and ^{26}Al in quartz over time. Plotted for the ideal case for exposure at SLHL with unshielded flat surface and no erosion. Used parameters are listed in Tab. 1.

Abb. 1: Darstellung der Konzentrationsentwicklung der Nuklide ^{10}Be und ^{26}Al für den Fall der Bestrahlung unter SLHL Bedingungen einer nicht erodierenden, nicht abgeschirmten, ebenen, Quarzoberfläche. Zu Grunde liegende Parameter können Tab. 1 entnommen werden.

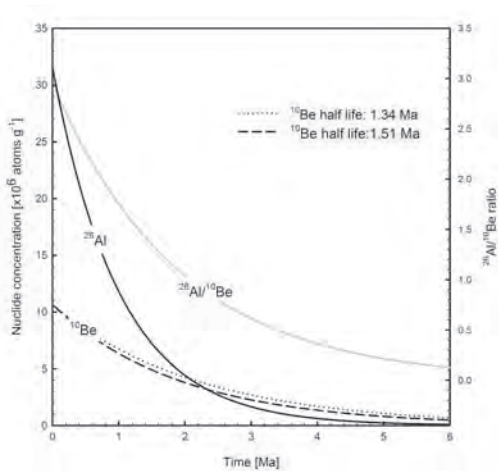


Fig. 2: Decreasing $^{26}\text{Al}/^{10}\text{Be}$ ratio (in gray) and individual nuclide concentration of ^{10}Be (dashed and dotted) and ^{26}Al (solid) in quartz over time with complete shielding from cosmic radiation after reaching individual saturation. Used parameters are listed in Tab. 1.

Abb. 2: Darstellung der Abnahme des $^{26}\text{Al}/^{10}\text{Be}$ Verhältnisses (in grau) sowie der Nuklidkonzentration von ^{10}Be (gestrichelt und gepunktet) und ^{26}Al (durchgezogen) in Quarz bei vollständiger Abschirmung der kosmischen Strahlung. Ausgangskonzentrationen entsprechen vollständiger Sättigung. Zu Grunde liegende Parameter können Tab. 1 entnommen werden.

condary cosmic rays and the attenuation effect of the atmosphere itself. Its value and scaling for the sampling position is also under debate at the moment, and is discussed in more detail in LAL (1991), DUNAI (2000), STONE (2000), WAGNER et al. (2000), DESILETS & ZREDA (2001), and references therein.

The nuclides ^{10}Be and ^{26}Al are also produced in situ by non-cosmogenic such as radiogenic reactions. SHARMA & MIDDLETON (1989) stated that only α -induced nuclear reactions could provide a significant portion of ^{10}Be and ^{26}Al . Lithium as target nuclei for production of ^{10}Be ($^7\text{Li}(\alpha,p)^{10}\text{Be}$) is normally present at trace levels ($\mu\text{g g}^{-1}$) but sodium, as a source of ^{26}Al ($^{23}\text{Na}(\alpha,p)^{26}\text{Al}$), is nearly always present at concentrations of one or more percent. Hence, ^{10}Be production is quite small compared to the

cosmogenic compound and therefore negligible, while that of ^{26}Al may be significant. BROWN et al. (1991), however, argued that the steady-state concentrations of radiogenically produced ^{26}Al in average sandstones is $\sim 6.0 \times 10^4$ at g^{-1} . This low concentration, with respect to steady-state concentrations of cosmogenically produced ^{26}Al of $\sim 3.2 \times 10^7$ at g^{-1} (Fig. 1 & 2), will only be significant in samples with a “short” exposure history. Beside this, no relevant radiogenic ^{26}Al contamination in quartz has ever been observed and reported so far.

3 Application of burial dating

The following chapter looks at different kinds of burial situation and is designed to give an overview of the required formulas, followed by selected examples. For more detailed information see GRANGER & SMITH (2000) and GRANGER & MUZIKAR (2001).

3.1 Single stage exposure history – cave sediments

The simplest case of burial is provided by cave sediments (Fig. 4), in that shielding from cosmic rays occurs quickly and effectively to inwashed sediments. The great advantage of these deposits is that they remain almost unaffected by cosmic radiation and thus only negligible post burial production results. Assuming steady-state erosion conditions for the sediments, the burial age is directly dependent on nuclide half-lives. Thus today's $^{26}\text{Al}/^{10}\text{Be}$ ratio gives us the burial age (t in years) solving equation 5 and 6 for ^{10}Be and ^{26}Al .

$$N_{10}(t) = \frac{P_{10}}{1/\tau_{10} + \rho\epsilon/\Lambda} e^{-t/\tau_{10}} \quad [5]$$

$$N_{26}(t) = \frac{P_{26}}{1/\tau_{26} + \rho\epsilon/\Lambda} e^{-t/\tau_{26}} \quad [6]$$

Equation 5 and 6 can be combined to equation 7

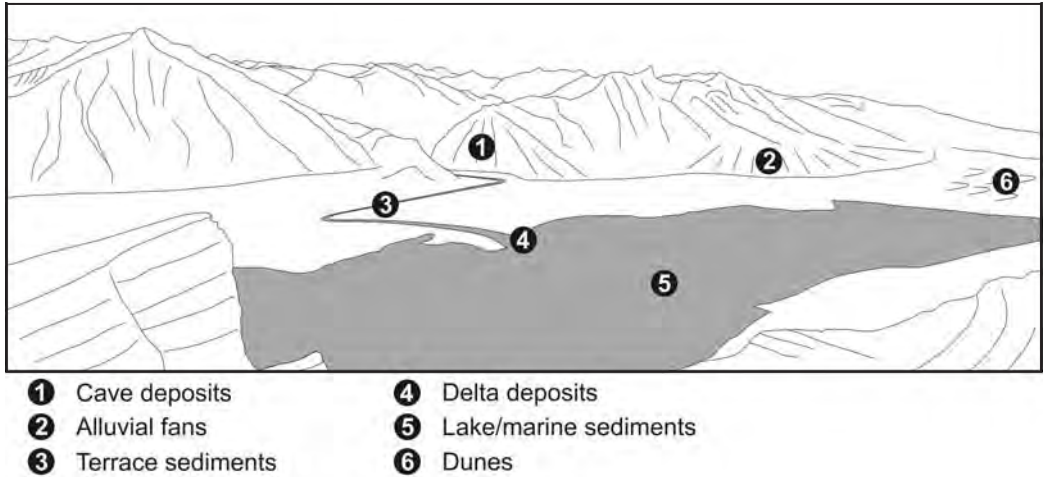


Fig. 3: Overview sketch of potential dateable sedimentary deposits with the cosmogenic burial dating technique. Quick shielding from cosmic radiation is achieved by washing into deep underground (1), covering with sediment (2, 3, 4, 6) or water (4, 5).

Abb. 3: Übersichtsdarstellung der potenziellen geologischen Archive, welche mit Hilfe des burial dating Verfahrens datiert werden können. Die notwendige rasche Abschirmung der kosmischen Strahlung kann erreicht werden durch Umlagerung in den tieferen Untergrund (1), Überdeckung durch Sediment (2, 3, 4, 6) oder Wasser (4, 5).

$$\frac{N_{26}(t)}{N_{10}(t)} = \frac{P_{26}}{P_{10}^{\tau_{10}/\tau_{26}}} (1/\tau_{10} + \rho\varepsilon/\Lambda)^{\tau_{10}/\tau_{26}}$$

$$(1/\tau_{26} + \rho\varepsilon/\Lambda)^{-1} (N_{10}(t))^{\tau_{26}/\tau_{10} - \tau_{10}\tau_{26}} \quad [7]$$

GRANGER et al. (1997) were the first to use the ^{26}Al - ^{10}Be pair to date burial events of sediments. They dated quartz pebbles found in various caves within dolomites along the New River, between Eggleston and Pearisburg, Virginia, USA. The pebbles, quartz vein remnants of metamorphic host rocks in New River's headwaters, were sampled in numerous cave levels up to 35 m above the modern river. By dating the inwash events of these pebbles from 0.29 ± 0.18 Ma to 1.47 ± 0.22 Ma, GRANGER et al. (1997) were able to calculate river incisions rates for the New River of 30.2 ± 5.5 m Ma^{-1} at Pearisburg and 19.7 ± 3.2 m Ma^{-1} at Eggleston, with a mean rate of 27.3 ± 4.5 m Ma^{-1} .

GRANGER et al. (2001) conducted similar studies at the Mammoth Cave multi level system

in Kentucky, USA. By analysing the $^{26}\text{Al}/^{10}\text{Be}$ ratio of 29 quartz gravel and sand deposits throughout the cave system and from its surface, they were able to reconstruct a water table history for the nearby Green and Ohio Rivers over the past 3.5 Ma. Furthermore, they demonstrated a glacial influence on the Ohio River behaviour with increasing incision (~ 1.5 Ma and ~ 1.2 Ma) followed by aggradation ($0.7 - 0.8$ Ma).

More recently, HAEUSELMANN et al. (2007) looked at glacially accelerated valley incision. Cave infill sediments were used to identify accelerated incision rates (up to 1.2 km Ma^{-1}) in the River Aare valley, between 0.8 to 1.0 Ma, in the Siebenhengste-Hohgant cave system, Switzerland.

Archaeological, as well as geological, questions can be solved using burial dating with ^{10}Be and ^{26}Al (see also AKÇAR et al. 2008). Dating of embedded bones is mostly done using U/Th dating of surrounding calcite layers (PICKERING et al. 2007). In the absence of speleothems or with unsuitable U/Th con-

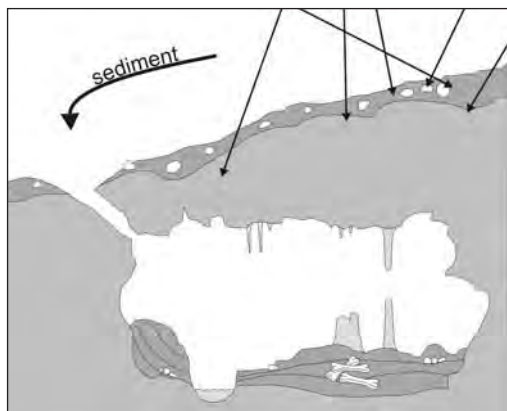


Fig. 4: Cave scenario: Exposed quartz-containing sediment or soil gets complete shielded by washing into a deep cave. Embedded fossils or artefacts can be dated by detecting the $^{26}\text{Al}/^{10}\text{Be}$ ratio of the surrounding sediment.

Abb. 4: Höhlenszenario: Bestrahtes(r) quarzführendes(r) Sediment oder Boden wird in eine Höhle umgelagert und hierdurch vollständig von der kosmischen Strahlung abgeschirmt. Die Altersbestimmung von eingebetteten Fossilien oder Artefakten kann dann durch die Bestimmung des $^{26}\text{Al}/^{10}\text{Be}$ Verhältnisses des umgebenen, zuvor eingespülten Materials erfolgen.

ditions, dating can also be done with the help of inwash sediments containing quartz. Lower Pliocene hominid remains from Sterkfontein cave, South Africa were dated indirectly by burial dating of the surrounding sediment by PARTRIDGE et al. (2003). The fossils are encased in a breccia of dolomite, chert and surface soil that accumulated as debris dropped into the cave. PARTRIDGE et al. (2003) utilised this fact to obtain burial ages from the quartz bearing material. Palaeomagnetic signals in calcitic flowstones dated the sediment layer containing hominid fossils into a timeframe ranging from 3.22 to 3.58 Ma. PARTRIDGE et al. (2003) dated this layer using the $^{26}\text{Al}/^{10}\text{Be}$ ratio to 4.17 ± 0.14 Ma in the Silberberg Grotto and the previously undated fossil layer in Jacovec Cavern to 4.02 ± 0.27 Ma. Although, these burial ages are in good agreement they are currently under debate (see WALKER et al. 2006).

3.2 Multiple stage exposure history – Nuclide profiling

Post burial nuclide production can not be ignored where sediment has only few metres of overburden, as can be done in the cave scenario described above. The post burial production is depth- and time-dependent (see chapter 2) and thus the relevant equations for both nuclides become:

$$N_{10}(z, t) = N_{10/\text{inh}} e^{-t/\tau_{10}} +$$

$$\int_0^t \left[P_{10N}(z + \rho \epsilon t') + P_{10\mu}(z + \rho \epsilon t') \right] e^{-t'/\tau_{10}} \partial t' \quad [8]$$

$$N_{26}(z, t) = N_{26/\text{inh}} e^{-t/\tau_{26}} +$$

$$\int_0^t \left[P_{26N}(z + \rho \epsilon t') + P_{26\mu}(z + \rho \epsilon t') \right] e^{-t'/\tau_{26}} \partial t' \quad [9]$$

Following GRANGER & SMITH (2000) the above integrals can be expressed as equation 10:

$$\begin{aligned} N_i(z, t) = & N_{i/\text{inh}} e^{-t/\tau_i} \\ & + \left[P_{iN} e^{-\rho z/\Lambda_N} / (1/\tau_i + \rho \epsilon/\Lambda_N) \right] \left[1 - e^{-t(1/\tau_i + \rho \epsilon/\Lambda_N)} \right] \\ & + \left[P_{i/1\mu-} e^{-\rho z/\Lambda_{1\mu-}} / (1/\tau_i + \rho \epsilon/\Lambda_{1\mu-}) \right] \left[1 - e^{-t(1/\tau_i + \rho \epsilon/\Lambda_{1\mu-})} \right] \\ & + \left[P_{i/2\mu-} e^{-\rho z/\Lambda_{2\mu-}} / (1/\tau_i + \rho \epsilon/\Lambda_{2\mu-}) \right] \left[1 - e^{-t(1/\tau_i + \rho \epsilon/\Lambda_{2\mu-})} \right] \\ & + \left[P_{i\mu+} e^{-\rho z/\Lambda_{\mu+}} / (1/\tau_i + \rho \epsilon/\Lambda_{\mu+}) \right] \left[1 - e^{-t(1/\tau_i + \rho \epsilon/\Lambda_{\mu+})} \right] \end{aligned} \quad [10]$$

The index i represents either the nuclide ^{10}Be or ^{26}Al . Each of equation 8 and 9 contains three unknowns (burial age, inherited nuclide concentration and erosion rate). Thus, a combination of [8] and [9] can not be solved uniquely by only analysing ^{10}Be and ^{26}Al in one sample at a specific depth. The solution for this problem lies in sampling a vertical profile within the deposit. By analysing two samples from the same profile and considering suitable constraints for the inherited nuclide concentration [8] and [9] can be solved uniquely. These assumptions are site-specific and may comprise of, for example, a constant inherited nuclide concentration

across the profile or a known inherited $^{26}\text{Al}/^{10}\text{Be}$ ratio. The model solutions from [8] and [9] need to be fitted to the field observations (measured nuclide concentrations). This can be done by least squares regression or other suitable optimisation techniques. Collecting more than two samples overconstrains the model solution, resulting in reduced random errors and yields a more robust detection of systematic deviations.

The first use of nuclide profiling to obtain sediment burial ages was done by GRANGER & SMITH (2000). They measured the ^{10}Be and ^{26}Al concentrations of nine samples in a ~ 10 m profile of a river terrace of Old Kentucky River at Rice Station, Kentucky, USA. The sandy sediment was rapidly deposited, perhaps at the shores of a rising proglacial lake, and has remained exposed since deposition. By solving an equation similar to [8] and [9] and least-square-fitting of the solutions, they dated the

terrace formation to $1.50^{+0.32}_{-0.25}$ Ma with a post-depositional terrace erosion rate of $6.2 \pm 0.2 \text{ m Ma}^{-1}$. This is in good agreement with the nearby Green River incision around 1.5 Ma (GRANGER et al. 2001, see above).

This initial study was followed by dating of alluvial deposits above the San Juan River near Bluff and Mexican Hat, Utah, USA by WOLKOWINSKY & GRANGER (2004). Their cosmogenic ^{10}Be and ^{26}Al data suggest a deposition age of $1.36^{+0.20}_{-0.15}$ Ma and an erosion rate of $14 \pm 4 \text{ m Ma}^{-1}$ for the ~ 12 m thick Bluff site. Additional to t and ϵ , they allowed a fit within the model for bulk density and the addition of, at most, 20 cm material on top of the sampled profile. They were not able to realise dates for the Mexican Hat site due to insufficient burial depth of only 5.5 m following GRANGER & MUZIKAR (2001).

The more recent work of HÄUSELMANN et al. (2007) utilises the nuclide profiling technique

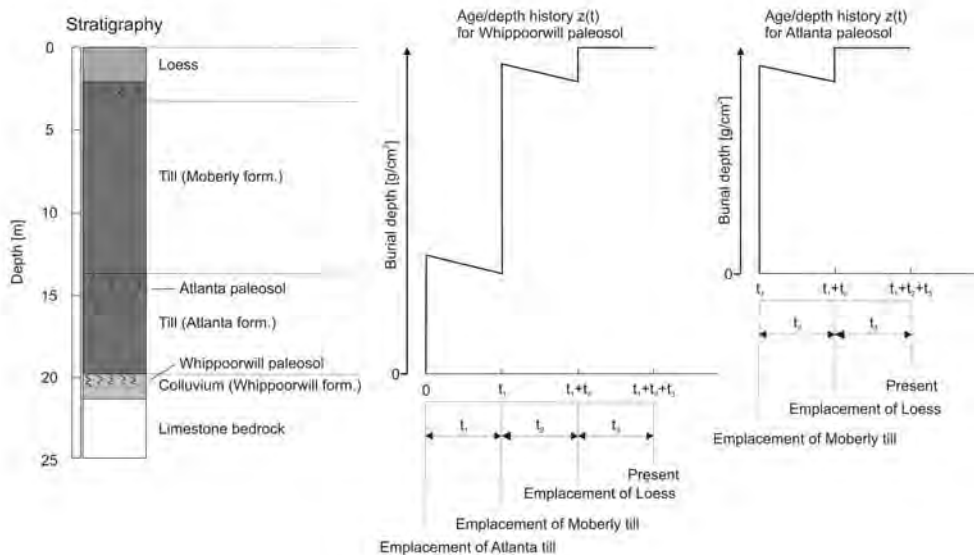


Fig. 5: Overview of the Musgrove section, Missouri, USA. Left part shows idealised stratigraphic sketch with used stratigraphic units. Right part visualise the used three-step density build-up model with rapid burial followed by moderate surface erosion. Modified after BALCO et al. (2005).

Abb. 5: Schematische Darstellung des Musgrove Profils, Missouri, USA. Links: vereinfachtes stratigraphisches Profil mit Tiefenangaben. Rechts: Veranschaulichung der Entwicklung der Überlagerungsmächtigkeiten im angewendeten dreiphasigen Modell mit rascher Überdeckung und anschließender gemäßigter Oberflächenerosion. Verändert nach BALCO et al. (2005).

to date glacially related gravel successions in the Bavarian foreland of the Alps, Germany, at an abandoned gravel quarry near Bad Grönenbach, and in a second site, Böhener Feld. Both sites are made up of a mixture of limestone and quartz gravels in a carbonate matrix. The pioneer work on alpine Quaternary geology by PENCK & BRÜCKNER (1909) named these gravels “Deckenschotter” (cover gravels) and placed them within the classical Mindelian (Bad Grönenbach) and the Günzian (Böhener Feld) glaciation, respectively. The age and terrace erosion rate determination of HÄUSELMANN et al. (2007) follows the nuclide profiling approach of GRANGER & SMITH (2000), as described above, followed by a χ^2 minimisation. This resulted in a fitted burial age of $0.68^{+0.23}_{-0.24}$ Ma and a terrace erosion rate of 123^{+139}_{-32} m Ma⁻¹ at Bad Grönenbach, and $2.35^{+1.08}_{-0.88}$ Ma and an erosion rate of 18^{+10}_{-4} m Ma⁻¹ for the section at Böhener Feld. This work was the first attempt to absolutely date these gravel deposits and demonstrates the suitability of the burial dating approach using ¹⁰Be and ²⁶Al within the time span of the Quaternary, although relatively high measuring uncertainties, due to the abundance of common Al impurities in the processed quartz, result in large model uncertainties and subsequently in large errors. This work also demonstrated that sample preparation and nuclide measurements are still delicate tasks.

The three examples described above, can explain the build-up of terraces in a single depositional event, which may occur over hundreds or even thousands of years. Cosmogenic burial dating of more complex deposit evolution is also possible, as long as a well constrained stratigraphy is available. In such a sedimentary sequence, each package will have its own deposition age and burial history that is determined by the deposition age of the following layer. BALCO et al. (2005) describe the age determination of such a multiple package set-up. They dated a profile containing three palaeosols buried beneath Laurentide Ice Sheet sediments and a loess cover. At the Musgrove clay pit, Missouri, USA, two tills, the Atlanta

and Moberly formations, overlie deeply weathered bedrock, as well as locally derived colluvium of the Whippoorwill formation (Fig. 5). BALCO et al. (2005) developed a three step burial history for the Musgrove section with rapid burial and moderate surface erosion (Fig. 5). Using Monte Carlo simulations in MATLAB’s Optimization Toolbox enables minimisation of χ^2 discrepancies between model and measurements. In doing so, they determine a burial age of 2.41 ± 0.14 Ma for the lower till section (Atlanta formation) and, one between 1.6 – 1.8 Ma for the upper till (Moberly formation).

It should be noted, however, that it was necessary to assume some initial conditions in order to complete this well-developed model. BALCO et al. (2005) chose an age of 125 ka for the deposition of the covering loess layer based on regional stratigraphy (identified by t_3 in Fig. 5). This assumption consequently affects all other age calculations in the used stepwise model (age of Atlanta till = $t_1 + t_2 + t_3$). This approach is, therefore, not an independent age determination as is normally assumed with the burial dating method, although it should be noted that a stratigraphical age of 125 ka for the loess is convincing and that BALCO et al. (2005) subsequently correlated the age of 2.41 Ma with a (global) ice sheet build up from 2.7 to 2.4 Ma, as suggested by marine oxygen isotope data (JOYCE et al. 1993).

3.3 Complex sediment burial histories

The history of sediments in the natural environment is often highly complex. The material undergoes several kinds of transportation usually involving multiple stages of exposure and burial from its source until its modern day position. It is often impossible to describe the complete transportation history with confidence.

In these situations the in situ cosmogenic nuclides ¹⁰Be and ²⁶Al may provide useful information regarding, whether sediment has been buried or not. In some cases it is possible to evaluate cumulative burial and exposure durations.

The first use of the ¹⁰Be/²⁶Al ratio was done by KLEIN et al. (1986). They looked at 12 Libyan

Dessert Glass samples, subdivided into three groups. Fission-track dating placed the age at 28.5 Ma, but in situ cosmogenic ^{10}Be and ^{26}Al concentrations did not agree with this estimate. KLEIN et al. (1986) attempted to solve this discrepancy with regard to the history of the Libyan Desert Glass and the distribution of nuclide concentrations within the individual groups. By analysing the ratio of ^{10}Be and ^{26}Al they could clearly show that burial by sand dunes is the most plausible explanation for the obtained nuclide concentrations. Furthermore they presented the first (minimum) burial ages based on the simplification of a single exposure followed by a single burial. The article of KLEIN et al. (1986) marks the advent of “burial dating with in situ produced cosmogenic nuclides”.

A second interesting application of the $^{10}\text{Be}/^{26}\text{Al}$ ratio in the early stage of burial dating development is documented by ALBRECHT et al. (1993), working on volcanic ash-flow tuffs at the Pajarito plateau of the Valles caldera, New Mexico, USA. These surfaces are part of the Tshirege member of the Bandelier Tuff, which was deposited during a caldera eruption at ~ 1.14 Ma. The Tshirege member can be subdivided into four subunits, each with its own erosive character. The analysis of nuclide concentrations of ^{10}Be and ^{26}Al identified two to five times lower concentrations than a 1.14 Ma exposure history should yield, which was mainly due to erosion and previous burial of the investigated surfaces. ALBRECHT et al. (1993) suggested a burial period of 0.6 ± 0.3 Ma for most of the studied samples by using the $^{26}\text{Al}/^{10}\text{Be}$ ratio. The former shielding of the sampled surface is explained by an eroded tuff and soil cover consisting of the overlaying subunits of the Tshirege member with an average thickness of ~ 6 m.

Burial by overlaying sediment is not the only explanation for a complex burial history. The shielding material does not necessarily have to be rock or sediment to result in the attenuation of secondary cosmic ray particle energy, which is dependent on the density of the overburden material. Thus any matter may shield cosmic radiation, with rock causing the most effective

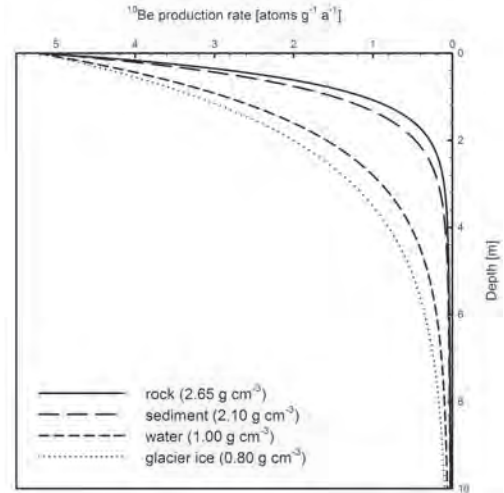


Fig. 6: Illustration of decreasing ^{10}Be production rate (at SLHL) caused by increasing shielding for typical geological overburdens with average densities at various depths. “Hard” overburdens such as rock or sediment obtain shielding rates of 90% in depth of ~ 1.8 m, while “soft” materials such as water or glacial ice need to be of ~ 4.5 m in thickness.

Abb. 6: Darstellung der ^{10}Be Produktionsratenabnahme bei zunehmender Überdeckung und damit steigender Abschirmung der kosmischen Strahlung (gültig für SLHL-Bedingungen).

Gezeigt sind ausgewählte geologisch typische Deckschichten mit ihren mittleren Dichten. „Harte“ Deckschichten wie Gestein oder Sediment erreichen Abschirmungsraten von 90% bereits in Tiefen von $\sim 1,8$ m, während „weiche“ Materialien, wie Wasser oder Eis, eine Mächtigkeit von $\sim 4,5$ m aufweisen müssen um die gleiche Abschirmung zu erreichen.

attenuation followed in order by sediment, water and glacial ice (Fig. 6).

Any shielding history is clearly shown by the position of the sample on a two-isotope diagram, for example $^{26}\text{Al}/^{10}\text{Be}$ ratios vs. ^{10}Be concentrations (Fig. 7), referred to as a “steady-state erosion island plot” (LAL 1991), “simple exposure island plot” or “banana plot”. The two-isotope diagram allows the detection of any shielding of a sample during its history. The “zero erosion line” and the “steady-state erosion line” frame a banana shaped area on a plot with logarithmic abscissa referred to

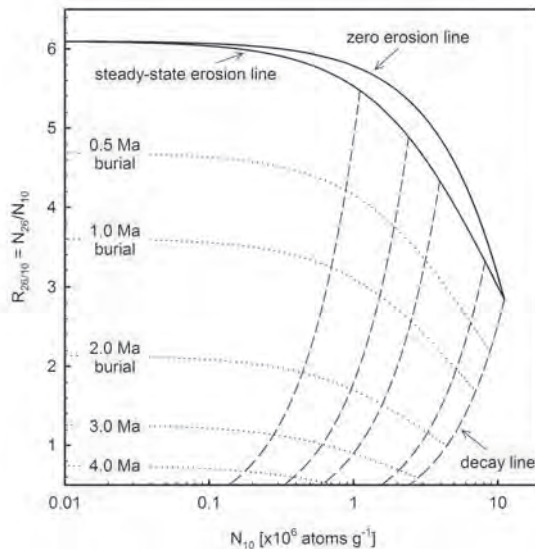


Fig. 7: Two-isotope diagram for in situ produced cosmogenic ^{10}Be and ^{26}Al in quartz as proposed by LAL (1991) with selected burial isochrones (dotted) and decay lines (dashed). Production rates are normalised to SLHL values with a $^{26}\text{Al}/^{10}\text{Be}$ production ratio of 6.1. Decay lines are given for complete shielding after single exposure with selected erosion rates (in mm ka^{-1}) until reaching saturation. Samples that have been shielded will plot below the steady-state erosion line in that the $^{26}\text{Al}/^{10}\text{Be}$ ratio will decrease resulting from the shorter half live of ^{26}Al with respect to ^{10}Be . Used parameters are listed in Table 1.

Abb. 7: Zwei-Isotopen-Diagramm für in situ produziertes ^{10}Be und ^{26}Al in Quarz nach LAL (1991), mit ausgewählte Überlagerungsisochronen (gepunktet). Produktionsraten sind normalisiert auf SLHL-Bedingungen mit einem $^{26}\text{Al}/^{10}\text{Be}$ Produktionsverhältnis von 6,1. Zerfallsgeraden (gestrichelt) sind dargestellt für den Fall der vollständigen Abschirmung nach einmaliger, kontinuierlicher Bestrahlung für ausgewählte Erosionsraten (in mm ka^{-1}). Proben die während oder nach ihrer Bestrahlung abgeschirmt wurden, plotten unter der Gleichgewichts-Erosionsgeraden, da das $^{26}\text{Al}/^{10}\text{Be}$ Verhältnis abnimmt, gemäß der kürzeren Halbwertszeit des ^{26}Al Nuklids. Zu Grunde liegende Parameter können Tabelle 1 entnommen werden.

as the steady-state erosion island (LAL 1991). All samples with a nuclide inventory resulting from simple exposure will plot within this area, while samples that have also undergone some shielding will fall below that. A determination of burial duration is only possible for a single burial event. A discrete observation of multiple shielding events is not possible with the steady-state erosion island plot, as the measured nuclide inventory is a composite of all exposure and shielding periods. Nuclide concentrations and production rates have to be normalised to sea level, high latitude ($> 60^\circ$; SLHL) values to avoid any affects of production rate changes resulting from scaling to sample location (for more detailed discussion of two-isotope

diagrams see LAL 1991; BIERMAN et al. 1999; GOSSE & PHILLIPS 2001).

Shielding by material other than sediment or rock was studied by BIERMAN et al. (1999), who measured ^{10}Be and ^{26}Al concentrations at two locations at the northern and southern margin of the former Laurentide Ice Sheet. The northern site around Pangnirtung on Baffin Island, Canada consists of in situ deeply weathered Precambrian gneissic bedrock. In the area around these tor samples neither bedrock nor boulders display striations, grooves or chattermarks, although erratic cobbles and boulders confirm the presence of glacial ice. The southern part near Pipestone, Minnesota, USA is made up of very erosion resistant Sioux Quartzite. Its surface

preserves ice-flow indicators which have subsequently been wind polished and cross-cut by shallower grooves, and earlier studies suggested that the areas were ice-free during the last glaciation. BIERMAN et al. (1999), however, determined $^{26}\text{Al}/^{10}\text{Be}$ ratios that are less than the production ratio of ~ 6.1 within the bedrock that do not support a single exposure history, and suggest the areas must have been shielded from cosmic radiation at some point during and/or after initial exposure. On the one hand, young exposure ages (see IVY-OCHS & KOBER 2008) of the sampled erratics, lying on the modern surface, suggest deposition by ice during the Last Glacial Maximum (LGM, 14 – 30 ka). On the other hand, high nuclide concentrations within the bedrock do not support glacial cover during the LGM, due to the lack of glacial erosion and therefore the lowering of nuclide concentration. To explain these opposing observations BIERMAN et al. (1999) suggest cover by cold-based glaciers that would have not eroded the underlying bedrock and therefore would have not reset the “cosmogenic exposure clock”. By modelling different exposure-burial scenarios they identified that the Sioux Quartzite samples have minimum total burial times that are more than twice as long as minimum exposure times, with an average minimum burial duration of 414 ± 29 ka. For the northern edge of the former Laurentide Ice Sheet on Baffin Island they conclude from ^{10}Be and ^{26}Al data, a non-erosive cold-based ice or deep snowfield cover for at least 400 ka.

A similar approach was used by STROEVEN et al. (2002) and FABEL et al. (2002) who investigated ancient landscapes in Northern Sweden as relicts of the Fennoscandian Ice Sheet, to test the hypothesis of landscape preservation through multiple glacial cycles. STROEVEN et al. (2002) investigated three tors and a bedrock outcrop in a meltwater channel in the Parkajoki area. The meltwater channel data are an exposure age of 11 ± 3 ka for both ^{10}Be and ^{26}Al , as a reliable deglaciation age. The lowered $^{26}\text{Al}/^{10}\text{Be}$ ratios of the tors, however, suggest that the sampled relict landscapes did not undergo a single exposure history, as re-

ported in the regional literature. FABEL et al. (2002) looked at bedrock outcrops and erratics on relict surfaces in the northern Swedish mountains. These yield deglaciation ages of $\sim 8 - 12$ ka with ^{10}Be surface exposure dating of the erratics but much older surface ages of $\sim 33 - 60$ ka for the bedrock, similar to STROEVEN et al. (2002). These researchers refined the approach described by BIERMAN et al. (1999) by using the marine benthic foraminifer oxygen isotope record of global ice volume from DSDP 607, as a proxy for the duration of periods of ice sheet cover vs. ice free conditions. They postulated 11 exposure and 10 burial events with a combined duration of 128 and 477 ka respectively for the Parkajoki area and a mean exposure-burial duration of 45 and 800 ka for the northern Swedish mountains.

4 Summary and outlook

The recent advances in terrestrial cosmogenic nuclides in geology and geomorphology have only been possible following the development of accelerator mass spectrometry in the late 1970's and early 1980's (see FINKEL & SUTER 1993), although the first burial and exposure histories were too complex and so difficult to interpret and provide reliable ages. A $^{26}\text{Al}/^{10}\text{Be}$ ratio significant lower than the production ratio was believed to indicate that the samples had been buried before, and only minimum burial ages could be constrained. After identifying that cave environments provide the ideal shielding scenario (instantaneous and sufficient), which prevents post burial nuclide production, it was possible to produce absolute burial ages from ^{10}Be and ^{26}Al determinations (GRANGER et al. 1997). This step, around 10 years ago, marked the beginning of “real” cosmogenic burial dating. In the last few years, burial dating has been applied using the nuclide profiling approach, to more complex environments such as alluvial fans (MATMON et al. 2005), fluvial terraces (WOLKOWINSKY & GRANGER 2004) or to relict surfaces beneath glacial ice that undergo partial shielding (STAIGER et al. 2005; BRINER et al. 2006; DAVIS et al. 2006). Nevertheless, this

often produces relatively large uncertainties that result in scepticism outside the cosmogenic community. As already shown, however, dating with cosmogenic nuclides offers unique advantages: application to quartz, the most common mineral on Earth's surface, and a time range of 100 ka to 5 Ma, making it very attractive to geomorphologists, (Quaternary) geologists and palaeoanthropologists.

Although fundamentals underlying its application have been recognised for almost 70 years, burial dating using cosmogenic nuclides is a relatively new technique and it is likely that in future nuclide pairs other than the $^{10}\text{Be}/^{26}\text{Al}$ one will be used. The shorter half-life of radiocarbon, 5730 ± 40 years (GODWIN 1962), would enable burial dating in the order of thousands to tens of thousands of years and allow the dating of more recent processes using the $^{10}\text{Be}/^{14}\text{C}$ pair in quartz. Additionally, slowly eroding rocks should have inherited ^{14}C concentrations close to saturation and would allow burial age determination without any other nuclides as initial nuclide concentration is reasonably well known. The $^{10}\text{Be}/^{36}\text{Cl}$ pair is also of interest, and would allow burial age determinations within carbonate rich and mafic environments. The first step towards the use of this nuclide pair has been made recently by BRAUCHER et al. (2006) who investigated the in situ ^{10}Be production rate and the chemical behaviour of ^{10}Be in carbonates as well as clinopyroxene samples. They identified a normalised ^{10}Be production rate of 37.9 ± 6.0 atoms $\text{g}^{-1} \text{a}^{-1}$ in calcite and 3.1 ± 0.8 atoms $\text{g}^{-1} \text{a}^{-1}$ in clinopyroxenes using the longer half life of ~ 1.5 Ma, and also presented laboratory protocols for the in situ ^{10}Be extraction from calcite and pyroxene samples.

New cosmogenic nuclide systems or combinations will be of interest in the coming years and the ongoing evaluation of physical constraints such as nuclide half lives (see chapter 1), nuclide production rates, muon production effects in depth and general long term production effects will also improve the precision of the general cosmogenic nuclide approach. The burial dating method stands today at a reasonably well-understood experimental level, although model

evaluations and sensitivity tests are often not considered, or only partially understood, as a result of lack available data, compared to other well established dating techniques, such as radiocarbon, uranium/thorium or luminescence dating. This situation will change in the future through further research and the discovery of new applications.

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Application of in-situ produced terrestrial cosmogenic nuclides to archaeology: A schematic review

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Abstract: The wide applicability of in-situ produced Terrestrial Cosmogenic Nuclides (TCNs) to geological problems and experiences in development and testing gained over the past decade is encouraging for its application to archaeological questions, where there is a distinct need for an additional independent dating tool beyond the limits of radiocarbon (~ 40 ka). Just as TCNs are applicable to a broader time period with considerable precision in archaeology, so also are they applicable to all lithologies. Application of TCNs to archaeological problems is relatively simple: either surface exposure dating (using cosmogenic nuclide production) or burial dating (using decay of radioactive cosmogenic nuclides) can be applied. For a successful application, close collaboration between archaeologists and TCN experts is required. The total exposure from 100 a to 5 Ma of a given surface of archaeological origin can be determined by surface exposure dating. The range of burial dating is from ~0.1 to 5 Ma. TCNs have been successfully applied to many archaeological problems during the last decade and both surface exposure dating and burial dating show high potential in the solving of archaeological problems.

[Anwendung in-situ produzierter, terrestrischer kosmogener Nuklide in der Archäologie: Ein schematischer Überblick]

Kurzfassung: Die breite Anwendbarkeit von in-situ produzierten Terrestrischen Kosmogenen Nukliden (TCN) in geologischen Fragestellungen und die damit verbundenen Erfahrungen in der Entwicklung und Eignungsprüfung die im letzten Jahrzehnt erlangt wurden, ermutigen diese Methode auch für archäologische Fragestellungen anzuwenden. In diesem Zusammenhang gibt es ein konkretes Bedürfnis für eine zusätzliche unabhängige Methode der Altersbestimmung jenseits der Grenze von Radiokohlenstoff (~ 40 ka). TCN sind nicht nur in einem Grenzbereich mit einer gewissen Präzision anwendbar, sie sind auch geeignet für die unterschiedlichsten Lithologien. Die Anwendung von TCN in archäologischen Fragestellungen ist relativ einfach: entweder wird der Ansatz der Oberflächendatierung gewählt (Nutzung der Produktion von kosmogenen Nukliden) oder man verwendet den Ansatz der Begrabungsalter (Nutzung des Zerfalls von radioaktiven kosmogenen Nukliden). Für eine erfolgreiche Anwendung der Methode ist jedoch eine enge Zusammenarbeit zwischen Archäologen und TCN Spezialisten notwendig. Eine Expositionszeit zwischen 100 a bis zu 5 Ma einer archäologisch geschaffenen Fläche kann durch Oberflächendatierung bestimmt werden. Der Altersbereich der Begrabungsaltermethode liegt zwischen ~0.1 to 5 Ma. TCN wurden während des letzten Jahrzehnts erfolgreich zur Datierung vieler archäologischer Fragestellungen angewendet und sowohl die Oberflächendatierung als auch Begrabungsaltermethode zeigen ein hohes Potenzial um archäologische Fragestellungen lösen zu können.

Keywords: Terrestrial Cosmogenic Nuclides, Archaeology, Numerical Dating

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1 Introduction

Shrewder sampling, improved chemical sample preparation and analysis as well as a better understanding of the physical processes responsible for in-situ produced Terrestrial Cosmogenic Nuclides (TCNs) have significantly enhanced the reliability of the method since its conception (cf., GOSSE & PHILLIPS 2001). The best known application of TCNs is the dating of the Quaternary ice volume fluctuations from the records archived by mountain glaciers (e.g., IVY-OCHS et al. 2006) and continental ice sheets (e.g., BRINER et al. 2006). Moreover, TCN methods have been employed in dating volcanic (e.g., LICCIARDI et al. 1999) and palaeoseismic events (e.g., BENEDETTI et al. 2003) as well as to quantify surface and/or rock uplift (RITZ et al. 2006) and incision rates (e.g., SCHALLER et al. 2005). Due to the sensitivity of the cosmogenic nuclide concentration to surface erosion and depth below the surface, the method has led to significant breakthroughs in establishing the rates and styles of local and large-scale erosion (SCHALLER et al. 2001), soil development (HEIMSATH et al. 1997) and landscape evolution (e.g., BIERMAN et al. 2002). The method can also be applied to the study of surface processes over a broad range of climate settings, employing different lithologies and mineralogies (IVY-OCHS & KOBER 2008). In addition, several geologic anomalies have been dated by cosmogenic nuclides such as Libyan desert glass (KLEIN et al. 1986) and the Canyon Diablo meteorite impact (NISHIZUMI et al. 1991; PHILLIPS et al. 1991). Depending on the surface preservation and exposure history, this dating technique has an effective range from the Pliocene to the late Holocene (IVY-OCHS & KOBER 2008). Its rapid and wide acceptance in using ^3He , ^{10}Be , ^{14}C , ^{21}Ne , ^{26}Al , and ^{36}Cl is not only due to its wide applicability to problems in surface geology but also to the reproducibility of many of its early results (GOSSE & PHILLIPS 2001 and references therein).

Cosmogenic nuclides can be used to exposure date fossils, lithic artefacts or monuments directly, to exposure date rock surfaces, or to

date sediments associated with fossils or stone tools, or to determine nuclide concentrations to assess tool procurement strategies. Therefore, cosmogenic nuclide methods have a largely unrealized potential in archaeology (STUART 2001). Although several dating techniques such as radiocarbon (e.g., GONZALEZ et al. 2003), U-series (e.g., SHEN et al. 2001), electron spin resonance (e.g., MOLODKOV 2001), $^{40}\text{Ar}/^{39}\text{Ar}$ (e.g., CLARK et al. 2003) and luminescence (e.g., VALLADAS et al. 2003), either directly or indirectly, can be applied to archeological problems, there is a substantial need for an additional dating tool in archaeology beyond the limits of radiocarbon and independent of the dating methods mentioned above (Fig. 1). Among several dating techniques, the TCN technique seems to be very promising due to its longer application period and efficacy without the need for any organic material (Fig. 1).

The primary focus of this review paper is on the evaluation of the application of TCNs to archaeological questions considering basically surface exposure dating and burial dating. Following the basic information on these two techniques, we discuss in detail how these techniques can be applied with assumptions, requirements, potential problems, disadvantages and advantages of each technique. This discussion is complemented with respective reviews of previous studies of TCN applications to archaeology.

2 How to apply TCNs to archaeology?

High-energy cosmic rays originating predominantly from super nova explosions within our galaxy are continuously bombarding the Earth. Interactions between these high-energy cosmic rays and the Earth's atmosphere create secondary and tertiary cosmic rays, including neutrons and muons. These high-energy particles can penetrate meters into rock and sediment when they reach the Earth's surface. Located in the upper surface of a rock, in-situ produced terrestrial cosmogenic nuclides are the product of the interaction of cosmic radiation, primarily neutrons, with a variety of target atoms within

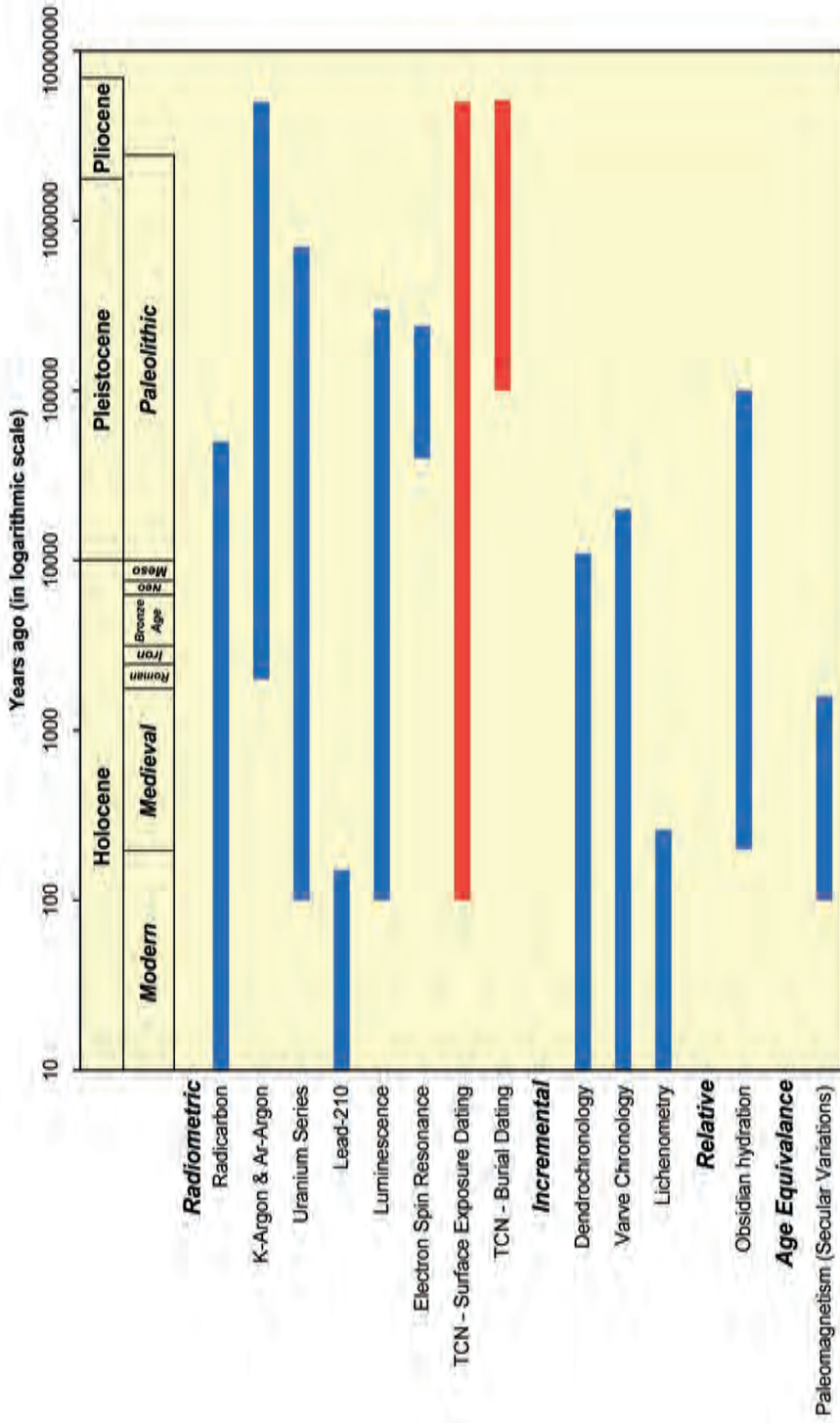


Fig. 1: The effective time ranges of the different dating methods applied (or can be applied) both in Quaternary Geology and in Archaeology (modified from ROBERTS 1998 and WALKER 2005).

Abb. 1: Effektive Zeiträume die von den verschiedenen Datierungsmethoden im Rahmen der Quartärgeologie und Archäologie abgedeckt werden oder abgedeckt werden könnten (modifiziert nach ROBERTS 1998 und WALKER 2005).

minerals (e.g., quartz, calcite, K-feldspar and olivine). This interaction results in nuclear reactions such as spallation (the splitting of nuclei), muon-induced reactions and neutron capture reactions (LAL & PETERS 1967; REEDY 1987; LAL 1988). In consequence of these reactions, unstable cosmogenic nuclides such as ^{10}Be , ^{14}C , ^{26}Al , and ^{36}Cl and stable cosmogenic nuclides such as ^3He and ^{21}Ne are produced in respective mineral lattices (Table 1) (cf., IVY-OCHS & KOBER 2008).

Theoretically, application of TCNs to archaeological problems is relatively simple. Either the production (both stable and unstable) or decay (in the case of unstable) cosmogenic isotopes can be applied. Surface exposure dating is the most common field of the cosmogenic nuclide technique (cf., IVY-OCHS & KOBER 2008), while burial dating, based on TCN decay, is now also increasingly used (cf., DEHNERT & SCHLÜCHTER 2008). Essentially, surface exposure dating can be applied over the timescales of around 100 a to 5 Ma and burial dating over around 0.1 to 5 Ma. The difference between the lower limits originates directly from the time required for a

sufficient decay of unstable cosmogenic nuclides in order to detect different isotopic concentrations due to various half-lives. Both surface exposure and burial dating tools require that the archaeological sample to be dated is destroyed during the sample preparation, and this can make the application of TCNs to archaeology at first seem strange. However, a destruction of this type can be minimized with careful planning of the sampling strategy and analysis of the scientific problem. In spite of this, the number of samples required for such analysis should be in the order of five.

2.1 Surface exposure dating

The application of surface exposure dating to archaeology at first sight appears to be restricted to large archaeological structures such as buildings, monuments and standing stones (STUART 2001). However the situation is simpler: if the concentration of the required TCN can be measured with accelerator mass spectrometry, the total exposure duration (from 100 a to 5 Ma) of a given surface can be deter-

Table 1: Characteristics of unstable cosmogenic nuclides ^{10}Be , ^{26}Al , and ^{36}Cl and stable cosmogenic nuclides ^3He and ^{21}Ne (modified after IVY-OCHS & KOBER 2007).

Tabelle 1: Charakteristiken der instabilen kosmogenen Nuklide ^{10}Be , ^{26}Al , und ^{36}Cl und der stabilen kosmogenen Nuklide ^3He und ^{21}Ne (verändert nach IVY-OCHS & KOBER 2007).

TCN	Production Rate* (atoms $\text{g}^{-1} \text{a}^{-1}$)	Half-life (Ma)	Target Elements	Target Minerals
^3He	119 (LICCARDI <i>et al.</i> 1999)	Stable	Many	Olivine Pyroxene
^{10}Be	5.1 (STONE 2000)	1.51 (HOFMANN <i>et al.</i> 1987)	O Si	Quartz
^{21}Ne	20.33 (NIEDERMANN 2000)	Stable	Si Mg	Quartz, Olivine Pyroxene
^{26}Al	30.1 (STONE 2000)	0.716 (SAMWORTH <i>et al.</i> 1972)	Si	Quartz
^{36}Cl	Lithology dependent ca. 10 in granite 20 in limestone	0.301	K Ca ^{35}Cl	Whole Rock

*Nuclide production rates at sea level and high latitude (GOSSE & PHILLIPS 2001).

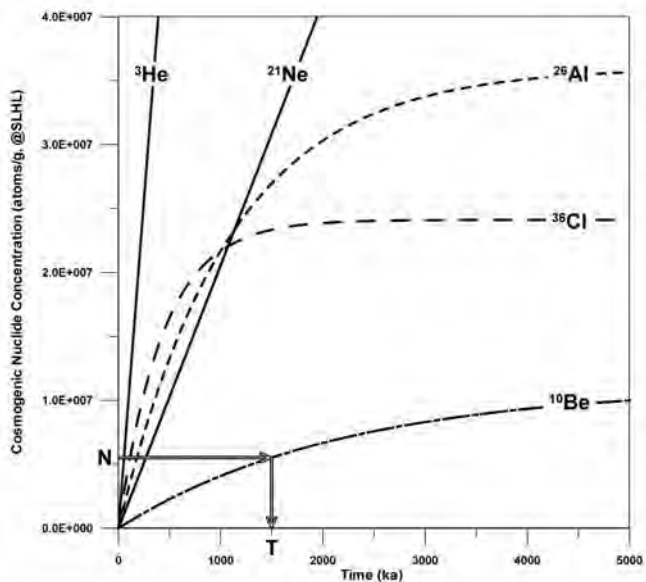


Fig. 2: Accumulation of ^3He , ^{10}Be , ^{21}Ne , ^{26}Al and ^{36}Cl in a rock surface as a function of exposure time at sea level high latitude (@SLHL). N is the concentration of the cosmogenic nuclide and T is the equivalent time of exposure. TCN concentrations were calculated with CosmoCalc 1.0 (VERMEESCH 2007), using STONE (2000) scaling factors and default values.

Abb. 2: Akkumulation von ^3He , ^{10}Be , ^{21}Ne , ^{26}Al und ^{36}Cl in Gesteinsoberflächen als Funktion der Expositionszeit im Meeresspiegelniveau (@SLHL). N ist die Konzentration des kosmogenen Nuklids und T ist die Äquivalenzzeit an Exposition. TCN Konzentrationen wurden mit Hilfe von CosmoCalc 1.0 (VERMEESCH 2007) kalkuliert, unter Anwendung der Skalierungsfaktoren und Vorgaben nach STONE (2000).

mined (Fig. 2) using the local production rate of the respective isotope (Table 1) (IVY-OCHS & KOBER 2008). For this determination, three conditions must be fulfilled. The first is that the cosmogenic isotope concentration at the beginning of exposure is zero or known (i.e. inheritance). The second is that the system must have been closed with respect to either gain or loss of the respective isotope. The third assumption is that the correct production rates are known and were constant during the exposure time.

CERLING & CRAIG (1994) were the first scientists who discussed the potential application of cosmogenic nuclides to archaeology in their review publication “Geomorphology and Cosmogenic Nuclides” and suggested that the pyramids, Sphinx, Stonehenge or even the

statues on Easter Island could be directly exposure dated. For instance, initial results from the analysis of ^{36}Cl in the bluestones from Stonehenge are controversial (WILLIAMS-THORPE et al. 1995). Furthermore they describe the debate on the construction age of the Sphinx (mainly “*the Final Riddle of Sphinx*”) and discuss a potential solution of this debate with TCN dating suggesting that archaeologists inform their scientific progeny since the time for this study would come. In addition to this exciting review, STUART (2001) summarised the principles (mainly for archaeologists) and utility of TCN methods to address archaeological questions. In his review on in-situ cosmogenic nuclides, the controls of the production of TCN in exposed surfaces are outlined and the factors of relevance for exposure dating of archaeological

material are evaluated (STUART 2001).

Construction of the field context in surface exposure dating of archaeological structures is the most important aspect. This context should be delineated with all available archaeological and geological data and the archaeological question discussed in detail. At this point, input from the archaeologist in the construction of the context may be determinant. Many problems which would arise later during evaluation and interpretation of the results can be omitted with careful planning during this phase. After the selection of the appropriate cosmogenic isotope to be applied (Table 1), samples are collected following the strategies defined in other surface exposure dating studies (GOSSE & PHILLIPS 2001). Not only the construction time of archaeological structures, but also collapse time can be surface exposure dated. For instance, exposure dating of blocks within the ruins of an archaeological structure can give information about the collapse event of this structure.

Several problems may arise during the building up of the field context concerning the main assumptions of surface exposure dating. Among these assumptions, the first is likely the most critical one, since the building stone (*sensu lato*) used for the construction has a possibility of being pre-exposed before quarrying and/or of having TCN concentration produced in depth and thus a significant concentration of inherited TCNs (IVY-OCHS & KOBER 2008). Although the inheritance is uncommon in practice when glacially transported erratic boulders are dated with TCNs (PUTKONEN & SWANSON 2003), the probability for archaeological applications is more serious. This potential presence of inheriting TCN can be assessed with careful observation during sampling, particularly in the case of quarried materials. If this is not possible, the analysis of TCN concentration over a depth profile of several meters may then provide information on the inheritance and erosion rates (see IVY-OCHS & KOBER 2008 for further details). If the source quarry can be determined, the concentration of TCN on both exposed and buried surfaces from this quarry can be analysed. In the first instance, this difficulty looks

discouraging; however it can after be eliminated within the field context and related sampling strategies in the field. Also, multiple TCN measurements may be helpful; for instance, measurements of unstable ^{10}Be and stable ^{21}Ne can be used to determine the exposure history of a given archaeological surface. ^{21}Ne concentration will clarify the total cosmogenic exposure history and, as the unstable ^{10}Be will decay in the case of any interruption in exposure, it will reveal the pattern of exposure history: either simple or complex. Simple exposure will result in ^{10}Be and ^{21}Ne ages, which are consistent within the error limits. However, a complex exposure will result in a younger ^{10}Be than ^{21}Ne age, due to the isotopic decay of ^{10}Be . Although complex exposure histories are successfully applied to glacially abraded bedrock and for surfaces (BIERMAN et al. 1999; FABEL et al. 2002; PHILLIPS et al. 2006), the application to archaeology is more complex. In this case, the exposure episodes will be interrupted by either complete or partial (shielding by other agents such as soil, sediment etc...) burial, or a series of different burial times. In such a complicated situation, the only information that can be deduced is the total exposure time to radiation. The best solution to this complexity is the minimization of unknown parameters, i.e. the careful examination of the field context.

Another problem concerning exposure dating assumptions is inconstant local production rates of respective TCN during exposure time. In the case of archaeology, this inconsistency can occur through rebuilding, restoration, renovation of archaeological structures such as buildings, monuments and standing stones. These kinds of post-exposure displacement activities will directly affect the production rate and thus may result in meaningless exposure ages. As in the case of inheritance, the variable production rate problem can be omitted again by careful observation within a well structured context.

Although the possibility is quite low and uncommon, system closedness concerning the third assumption can also cause some problems. There are two ways this occurs: either some of the TCN is lost during the exposure time, such

as chipping out of the material through physical weathering, which will result in younger exposure ages. The prevention is simple: the surface to be sampled should be selected carefully. On the other hand, a gain of TCN through atmospheric cosmogenic isotopes (see IVY-OCHS & KOBER 2008 for further details) such as meteoric ^{10}Be and ^{36}Cl may result in higher exposure ages. Possible contamination by meteoric isotopes is unlikely and normally removed during sample preparation due to improved chemical sample preparation in the TCN technique.

In the first study to solve an archaeological problem by surface exposure dating, PHILLIPS et al. (1997) used ^{36}Cl to address the question of whether the exposed schist surfaces of the Côa valley (northern Portugal) were available for engraving during Palaeolithic times (from around 2.5 Ma to around 10 ka ago). The age of the animal and other figures engraved on these rock surfaces is controversial and hypotheses fall into two time periods: (1) Palaeolithic, due to the stylistic indications (among others ZILHÃO 1995) and (2) Holocene, suggested by radiocarbon dating (WATCHMAN 1995; 1996) and microerosion dating results (BEDNARIK 1995a; 1995b; 1995c). PHILLIPS et al. (1997) revealed that the rock panels had been exposed long enough to be engraved during the Palaeolithic, and argued for a landscape stable enough to support Palaeolithic art (STUART 2001). Although PHILLIPS et al. (1997) had given a maximum exposure age (i.e. the real exposure age is probably younger) for the schist panels, this first application of TCNs was criticised by WATCHMAN (1998) and BEDNARIK (1998), with criticisms focusing mainly on inheritance (i.e. the TCN concentration present prior to the exposure), erosion rates of the schist panels and the potential contamination of meteoric ^{36}Cl due to the high hydrological and therefore hydrochemical activity in the region, i.e. this system was not closed with respect to gain of the respective isotope.

A prime example of surface exposure dating study is by IVY-OCHS et al. (2001) who analysed two chert artifacts (flakes made during the knapping process) of Late Acheulean or

early Middle Palaeolithic age from the Thebes Mountains near Luxor, Egypt. By measuring the cosmogenic ^{10}Be concentration in the artefacts they were able to calculate 'nominal exposure ages' of 326 and 304 ka respectively.

In a later study, FARLEY et al. (2001) successfully measured ^3He in the fluorapatite of fossil teeth, opening up the possibility of dating fossil remains at archaeological sites. On the one hand the continuous exposure requirement must be satisfied. On the other, the preliminary results of FARLEY and coworkers imply that some of the fossils they studied comprise lag deposits and are much older than the associated sediment.

The main disadvantage of surface exposure dating of archaeological structures seems to be the inheritance as was also experienced in the earlier studies. As mentioned above, the inheritance problem can mainly be omitted during the scientific context construction phase; if not possible, this can still be excluded by applying a multiple TCN approach, but even so this will be not more than a limited approach to the problem according to the present level of experience in such situations.

Surface exposure dating is a direct, absolute dating tool which can be applied to a broader time span within and beyond the ^{14}C limit. Its independence of the organic material and lithology is a key advantage compared to the other dating techniques. Furthermore, surface exposure dating can be used as an 'event' dating tool in archaeology such as construction or destruction of archaeological structures. The precision of surface exposure dating is high enough to date such archaeological events.

In particular, lower detection limit of surface exposure dating, which is around 100 years, would for instance mean that marble statues older than 19th century can be exposure dated. This fact, in principle, would imply that TCN could be potentially used to determine whether an archaeological object is original or not. All these advantages of exposure dating are encouraging for its application to the archaeological questions. Much has been learnt from previous studies; however, there is still more to be learnt and experienced with new applications.

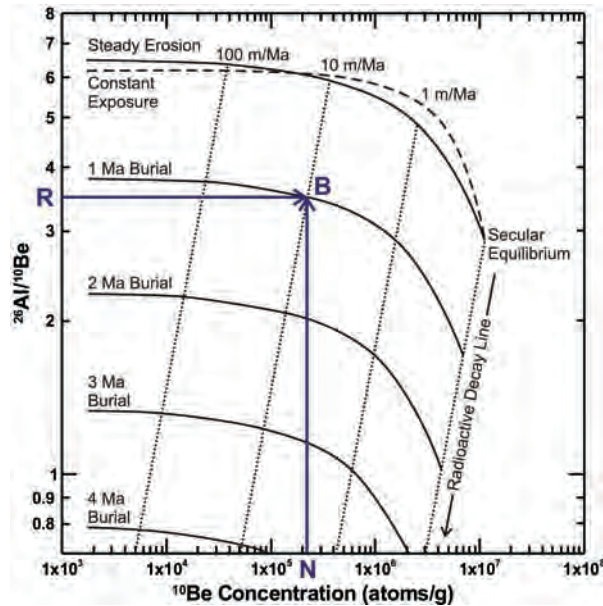


Fig. 3: Plot used in burial dating (modified from GRANGER & MUZIKAR 2001). R is showing the $^{26}\text{Al}/^{10}\text{Be}$ ratio, N the ^{10}Be concentration and B the equivalent burial time.

Abb. 3: Diagramm zu Berechnung von Begrabungsaltern (verändert nach GRANGER & MUZIKAR 2001). R ist das Verhältnis $^{26}\text{Al}/^{10}\text{Be}$, N die Konzentration an ^{10}Be und B die äquivalente Begrabungszeit.

2.2 Burial dating

The application of burial dating to archaeology is a promising tool. Basically, any object of archaeological origin that has been buried can be dated with this technique (Fig. 3). Considering the period of application (from ~100 ka to 5 Ma), burial dating can be mainly applied to the Palaeolithic time (from around 2.5 Ma to around 10 ka ago). Until now, burial dating has been applied using ^{10}Be and ^{26}Al in quartz. The fundamentals are as follows: the cosmic ray cascade causes the production of TCN in quartz crystals (as sediment and/or mineral in the rock) at or near the earth's surface. When quartz grains are eroded, transported and deposited far below the surface (e.g., in a cave), the TCN production rate becomes zero due to the attenuation of the cosmic rays in the rock. The radioactive decay of ^{10}Be and ^{26}Al with time will result in a decrease in concentrations of these radionuclides in the host mineral (mainly

quartz). Since ^{26}Al has a shorter half-life than ^{10}Be (Table 1), ^{26}Al vs. ^{10}Be ratio decreases with time and the time since the sediment was buried can principally be determined when the initial build-up is modelled (for details see DEHNERT AND SCHLÜCHTER 2008). For burial dating application, four requirements must be fulfilled. The first is that the accumulation of TCN in the quartz grains prior to burial must be sufficient enough for a successful AMS measurement. The second is that the transport and the burial of quartz should be contemporaneous with respect to timescales of radioactive decay. The third is that the prospective quartz sample must be buried deep enough to avoid significant TCN production after burial and the fourth requirement is that the TCN production should result from a simple history of exposure prior to burial.

The burial dating technique can be applied, for instance, to the settled caves of Palaeolithic times (e.g. PARTRIDGE et al. 2003). In principle,

one could burial date both the artefact and the enclosing sediment. This is a reliable way to perform a cross-control of the application of TCNs, especially when other dating techniques are not applicable. Application of burial dating to archaeology is not restricted to Palaeolithic caves. Another application can be the strata of different cultures. In this situation, an archaeological stratum can be handled as a geological unit and the deposition time of the layer can be determined within given uncertainties. Burial dating can also be applied to waste disposal in antique quarries. Here, even different phases of waste disposal can be determined in relation to the original quarry phase. Burial dating can also be applied to the special cases where archaeology and natural hazards coincide. For example, a mass movement can destroy a settlement or a part of it and the buried sediment can be dated by TCN analysis.

Again, as in surface exposure dating, construction of the field context is the most important aspect. The context should be constructed in such a way as to minimize uncertainties which can potentially cause problems during consequent phases of burial dating, using all available archaeological and geological data with close cooperation between archaeologists and TCN experts. However, some problems may still appear during archaeological applications of burial dating concerning the four requirements mentioned above. Since the accelerator mass spectrometric measurement technique is able to measure very low concentrations of TCNs, the first requirement (*Measurable TCN concentrations*) will not cause any problem. The second requirement (*Contemporaneous transport and burial*) can easily be fulfilled, since the time between the production and the burial of the archaeological artefacts is considered contemporaneous compared to the radioactive half-lives of ^{26}Al and ^{10}Be . The third requirement (*No TCN production after burial*) is likely the most critical one and affects all burial dating applications. Here, on the other hand the production of TCNs at depth cannot be neglected, since there will still be some amount of TCN concentration produced after deposition and/or burial. This

potential problem can be minimized by the application of an appropriate model for production after deposition. Beside the third requirement, the fourth requirement (*Simple exposure history before burial*) is fulfilled for artefacts by approximations following appropriate models as with other burial dating applications (for details see DEHNERT & SCHLÜCHTER 2008).

During the last decade, burial dating has been applied to archaeological questions. BOARETTO et al. (2000) proposed the application of burial dating to archaeology with a case study from Tabun Cave, Mt. Carmel (Israel), which has a sedimentary sequence representing the type section for about the last 800 kyr in the Levant. Flint tools are also embedded within this quartz rich sequence. In this study, flint tools as well as sediments were sampled for ^{10}Be and ^{26}Al . The flints that they analyzed contained too much stable ^{27}Al and too little ^{26}Al , so that their analyses were complicated. Thus, BOARETTO et al. (2000) did not calculate burial ages for these samples. The key finding of this study is that the internal environment of the flint is well sealed, so that any contamination by meteoric ^{10}Be seems to be unlikely.

Apart from the direct application of burial technique, VERRI et al. (2002, 2004, 2005) studied flints that were embedded within the sedimentary sequence in the Tabun and Qesem caves in Israel. Considering the basic idea that flint mined from deeper than 2 m will have a low ^{10}Be concentration, whereas flint collected from the surface or mined from shallower than 2 m will contain a higher ^{10}Be concentration, they determined that different flint procurement strategies, including deep mining, were used at least as early as the Late Lower Palaeolithic in the Levant. For instance, ^{10}Be concentration of flints from one layer (Lower Layer E, Acheulo – Yabrudian, around 400 – 200 ka) in Tabun cave are very low and consistent compared to the layers below and above (VERRI et al. 2005). Recently, PARTRIDGE et al. (2003) applied burial dating (using ^{10}Be and ^{26}Al) to the fossiliferous breccia in the Jacovec Cavern and Silberberg Grotto at Sterkfontein, South Africa. The results of this application revealed that the ho-

minid fossils embedded within these deposits were accumulated in the Early Pliocene, and also that burial dates obtained from Jacovec Cavern and Silberberg Grotto are indistinguishable. Approximately 4 Ma of burial age for the hominid remains found in these two sites is also consistent with and similar to the findings from East Africa (CLARKE et al. 2003; PARTRIDGE et al. 2003). In a subsequent study, MUZIKAR & GRANGER (2006) applied a combination of cosmogenic, stratigraphic and palaeomagnetic information using a Bayesian approach to refine the burial dating results from Sterkfontein.

The main disadvantage of burial dating of archaeological artefacts seems to be post-burial TCN production and possible complicated TCN production history prior to burial as experienced also in the earlier studies. This problem can be mainly solved –as mentioned above– just during the scientific context construction phase. Especially the complexity vs. simplicity of TCN production (complex or simple exposure) prior to burial can give information about the tool procurement as demonstrated by VERRI et al. (2002, 2004, 2005). As the ^{10}Be and ^{26}Al in quartz have been used in burial dating applications until now, this can look like a disadvantage of lithology dependency, however the use of other TCNs in burial dating is still open and needs to be tested and practiced. Much has been learnt from previous applications: burial dating is applicable to a broader time span, especially to Palaeolithic, i.e. definitely far beyond the radiocarbon dating limit; and it is also a direct, absolute dating tool which is independent of the presence of organic material. Hence, these two advantages are encouraging for its application to archaeological questions, even though there is certainly still more to be learnt and experienced with new applications.

3 Conclusions

During the last decade, TCNs have been successfully applied to a several archaeological problems, although most of these were test applications. TCN methodology has several

advantages over other dating tools: as the radiocarbon technique is widely used by archaeologists, absence of organic material is generally a problem. Moreover, increasing uncertainties with time and calibration issues are sometimes problematic. At this point, TCNs seem to be an outstanding approach in solving archaeological problems. In archaeology, TCNs can be applied (1) to a larger time period beyond the radiocarbon dating limit, (2) to all lithologies, and (3) with considerable precision. Either the production or decay of cosmogenic isotopes is used for the application of TCNs: the accumulation of TCNs will be used for surface exposure dating and their decay for burial dating. The total exposure from 100 a to 5 Ma of a given surface of archaeological origin can be determined by measuring the concentration of the required TCN with accelerator mass spectrometry. Burial time from ~0.1 to 5 Ma is determined by burial dating technique. Although these two types of applications have their own problems, a selective and precise discussion of the scientific and field context before and during sampling will minimize potential complications that may arise during the interpretation phase.

TCN tools, both surface exposure dating and burial dating, have, generally speaking, high potential for applications in the field of archaeology. If we assume that the total number of archaeological sites in the Eastern Mediterranean region is around 10,000 and if < 5% of these archaeological sites are suitable and available for the application of TCN analysis, then total the number of potential study applications will still number in the hundreds.

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The handling of numerical ages and their random uncertainties

MEBUS A. GEYH^{*)}

Abstract: The correct handling of numerical ages and their standard deviations and a proper introduction to error propagation or propagation of uncertainty and statistical evaluation are important to avoid misleading chronological conclusions and statements even though based on properly determined and reliable numerical dates. The conclusions may also be erroneous if dates were taken from databases without sufficient background information on the origin of the dated material and the applied analytical techniques. This paper is an introduction into the field of mathematical handling and testing of numerical ages. The most common and simple calculations and statistical tests that are needed are described and the steps involved are demonstrated on examples. The problems involved in the visualization of numerical dates in the form of normal histograms and dispersion histograms are discussed.

[Der Gebrauch numerischer Alter und ihre Standardabweichungen]

Kurzfassung: Die korrekte Auswertung numerischer Alter mit ihren Plus-Minus-„Fehlerangaben“ oder besser Standardabweichungen und die Anwendung der damit verbundenen statistischen Auswerteverfahren sind für Geowissenschaftler und Geographen oft eine schwierige Aufgabe, weil ihnen dafür die Ausbildung fehlt. Entsprechend finden sich in der Literatur viele nur schwer nachvollziehbare geochronologische Folgerungen. Sie können allerdings auch dann falsch sein, wenn sie sich auf Ergebnisse aus Datenbanken ohne ausreichende Angaben über den Ursprung des datierten Materials und die angewandten analytischen Techniken beziehen. Diese Arbeit führt in die Beurteilung und Handhabung numerischer Altersangaben ein. Mit ihr können elementare Rechnungen und einfache statistische Tests korrekt ausgeführt werden. Entsprechende Beispiele veranschaulichen den Weg dazu. Die mit der Visualisierung von numerischen Altern in Form von einfachen und Dispersions-Histogrammen verbundenen Probleme werden diskutiert.

Keywords: numerical ages, propagation of uncertainty, histogram

1 Introduction

Owing to the growing number of databases open to the public and the increasing production of numerical ages using different dating methods and semi-automated equipment. Quaternary scientists are thus faced with the task of handling large sets of numerical ages with their random uncertainties rather than dealing with single numerical ages. Reliability checks based only on the values themselves are indis-

pensable as in many cases information on the dated material and the analytical techniques is insufficient or not accessible.

Unqualified handling of numerical ages may result in conspicuously erroneous scientific conclusions. An example is a statistically evaluated cycle in the formation of speleothem and calcareous tufa in Europe which appeared to correlate with the 25-ka cycle of the precession of the Earth's axis (BRUNNACKER & HAUSMANN 1987; GEYH 1991). Reliable calculation of error

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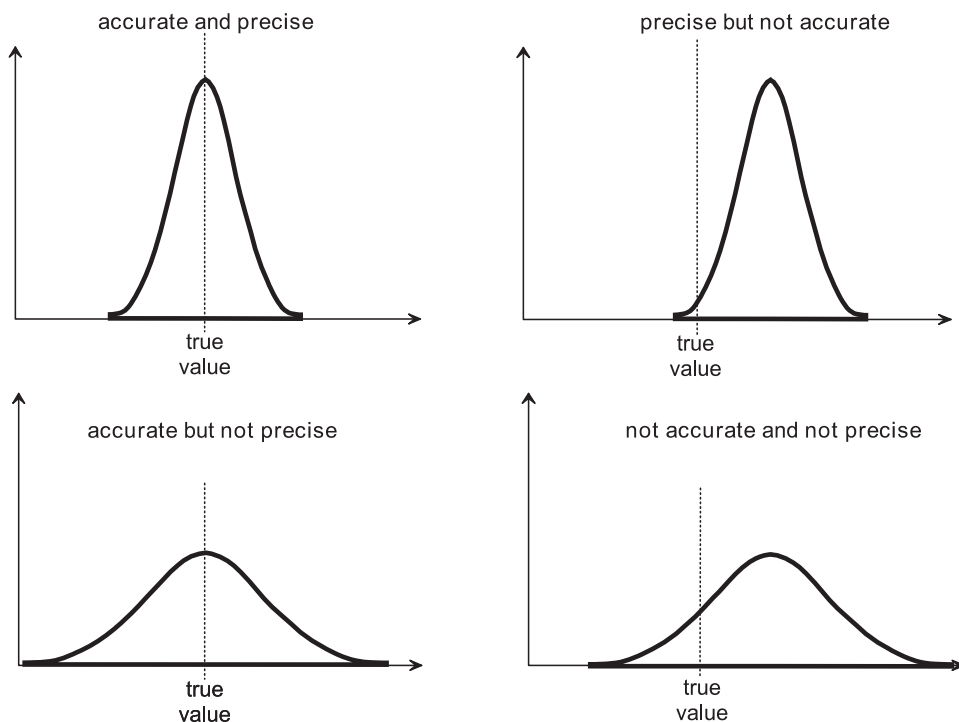


Fig. 1. The meanings of accuracy and precision: Accuracy describes the approximation of a mean to the „true“ value; precision is a measure of the reproducibility of a result.

Abb. 1. Bedeutung von Genauigkeit und Präzision: Genauigkeit beschreibt die Annäherung an den „wahren“ Wert. Die Präzision ist ein Maß für die Reproduzierbarkeit eines Ergebnisses.

propagation avoids such conclusions and also allows manipulated numerical ages to be identified (GEYH 1991). This paper provides the basics necessary for proper mathematical treatment of geochronological and analytical data of any origin with their random uncertainties. Complicated mathematical functions are not discussed. This paper uses the terminology of HUNTLEY (2001) in which „age“ refers to a range of time something has existed, while a „date“ is a particular time on a time scale. The functions given below are valid for any data, not only numerical ages.

2 Precision, accuracy and reporting of data

Precision and accuracy have distinct meanings as illustrated in Figure 1.

1) The *precision* of a result is a measure of the reproducibility of an observation and

corresponds to the *uncertainty* of a result. It is expressed as random error or *random uncertainty* and is mainly determined by the analytical technique and equipment used.

2) The *accuracy* is a measure of the correctness of an observed value and its approximation to the „true“ value. It depends on a large number of often unknown systematic errors, which may be eliminated by carrying out routine measurements on standard materials. Aliquots of the same set of samples of different standard materials are analyzed by laboratories throughout the world and the results compared. Reducing systematic errors increases the accuracy rather than the precision.

The expression $\pm \sigma A$ is termed *random* or *statistical uncertainty* and has to be distinguished from *systematic errors*. A *systematic error* arises from a specific physical or chemical

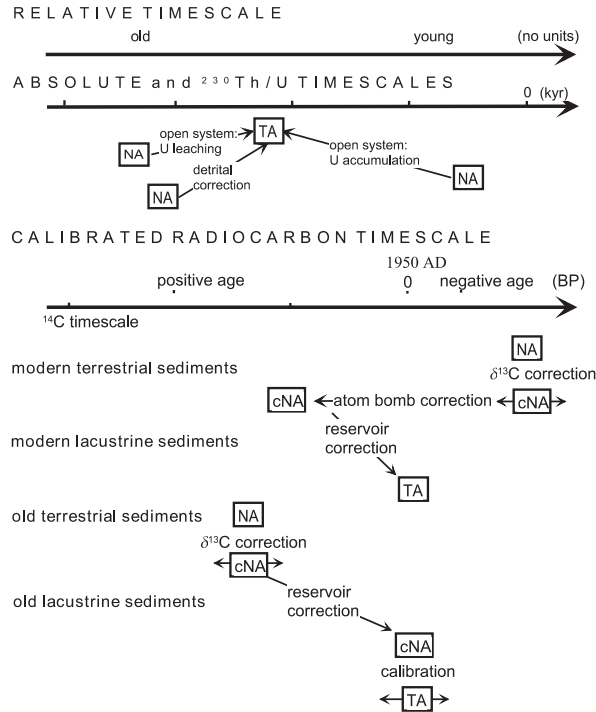


Fig. 2. Scheme for the correction and calibration of radiometric and mass spectrometric ^{14}C and $^{230}\text{Th}/\text{U}$ ages, in order to correlate them with the absolute time scale (GEYH & GROSJEAN 2000). TA – true age; NA – numerical age, cNA – corrected numerical age

Abb. 2. Schematische Darstellung der Korrektur und Kalibration radiometrischer und massenspektrometrischer ^{14}C - und $^{230}\text{Th}/\text{U}$ -Alter zur Korrelation mit der absoluten Zeitskala (GEYH & GROSJEAN 2000). TA – wahres Alter; NA – numerisches Alter, cNA – korrigiertes numerisches Alter

process, only changing the measured values in one direction or the other. If the process is understood and the relevant parameters are known, the result can be corrected. As an example, the relevant processes that affect ^{14}C and $^{230}\text{Th}/\text{U}$ ages are shown in Figure 2. Contamination with material older than the sample to be dated (e.g. with coal, fossil lime or detrital thorium) increases ^{14}C and $^{230}\text{Th}/\text{U}$ ages, respectively. Five percent matter containing no radiocarbon increases the ^{14}C ages by around 400 years. The same amount of uranium of a material in which the ^{238}U series is in radioactive equilibrium will increase the $^{230}\text{Th}/\text{U}$ ages by around 5000 years. A correction of these ages is possible if the proportion of the contaminating material is known. The conventional rules for reporting of quantitative data are often neglected. One rule is that

any quantitative result should be accompanied by a statement of the intrinsic uncertainty. The *precision of the random uncertainty* (the measure of its uncertainty) is usually not better than $\pm 10\%$. An example illustrates this rule. A random uncertainty of ± 213 has an uncertainty of about ± 21 a. Hence, it is reasonable to report the uncertainty as ± 210 a rather than ± 213 a. This means that the uncertainty should be rounded to two digits only (TAYLOR & KUYATT 1994). Another rule is that a set of values should always have the same number of places behind the decimal point. Hence, an age of 2.87 ± 0.20 ka should not be given as 2.87 ± 0.2 ka. This rule is especially important for the compilation of data in a table. Another rule is that only the final result should be rounded off rather than the values used in a calculation.

3 Numerical age, random uncertainty and frequency distribution

Any geochronological framework has to be based on numerical ages determined with suitable physical dating methods (GEYH & SCHLEICHER 1990; WAGNER 1998; GEYH 2005). The resulting ages are often inappropriately termed „absolute“ ages. If an age date is obtained, for example, from stratigraphic or palynological studies it is a relative age. Physical age dating also does not yield absolute ages; the results are better called „numerical ages“. The term absolute age should be reserved only for age dates related to the solar time scale.

The collected samples have to fulfill numerous criteria to be suitable for reliable dating. Different kinds of numerical ages have to be corrected for different sources of systematic error (e.g., ^{14}C ages for contamination, reservoir effect, and ^{13}C isotope fractionation; and $^{230}\text{Th}/\text{U}$ ages for detrital ^{230}Th ; e.g., GEYH & GROSJEAN 2000; Fig. 2)

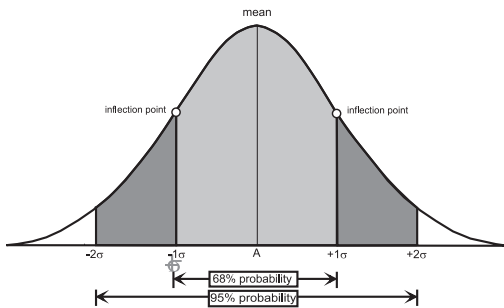


Fig. 3. Gaussian frequency distribution (also bell-shaped curve, normal distribution): The 1-sigma confidence interval lies between the two inflection points of the frequency distribution. The light gray area corresponds to nearly 68 % of the total area, the 2-sigma confidence interval represents a probability of about 95 %.

Abb. 3. Gauß'sche Häufigkeitsverteilung (auch Glockenkurve, Normalverteilung): Das 1-Sigma Vertrauensintervall liegt zwischen den beiden Wendepunkten der Glockenkurve. Die graue Fläche entspricht nahezu 68 % der Gesamtfläche, das 2-Sigma Vertrauensintervall repräsentiert eine Wahrscheinlichkeit von 95 %.

and in the case of dating with cosmogenic isotopes calibration is needed (e.g., ^{14}C dates). This is particularly important when numerical ages obtained from different materials and methods are compared with each other and/or with the absolute time scale (GEYH & SCHLEICHER 1990; WAGNER 1998; GEYH 2005).

When dealing with numerical ages, it is very important to understand the meaning of the common expression $A \pm \sigma A$, e.g., 2000 \pm 50 a before present (BP). *Standard deviation*, 1-sigma interval, random uncertainty, and statistical uncertainty are used as synonyms for $\pm \sigma A$. It defines the precision of the calculated value, and represents a frequency distribution. Theoretically it is assumed that the age of the sample was determined an *infinite* number of times. In practice, however, the standard deviation is precisely determined on the basis of a *finite* number of measurements. The ages determined on the same sample deviate from each other, even under most exacting measurement conditions, and scatter around a mean value \hat{A} , which is assumed to approximate the „true“ age of the sample. The frequency distribution of values is obtained by plotting all these values with the x-axis representing „age“ and the y-axis representing the frequency of occurrence of each value. The resulting curve looks like a vertical cut through a bell and is known as a *Gaussian distribution*, *normal distribution* or *bell-shaped curve* (Fig. 3). It is symmetrical around the *mean* value of all values.

In reality only a finite number of results are available and the frequency distribution is not perfectly symmetrical. In this case the curve peak is given by the median rather than the mean. It divides a frequency distribution into two equal areas. This case will, however, not be discussed here as it is of minor importance in geochronological studies due to the relatively small number of ages obtained from coeval samples.

The area enclosed by the bell-shaped curve, extended to infinity in both directions, represents the total number of values or 100 % probability. The area between the two inflection points, shown in light grey, contains nearly 68 % of all values (68 % probability) (*1-sigma interval* or

1σ confidence interval). The 2-sigma confidence interval contains approximately 95 % of all values (95 % probability), corresponding to a probability of error of 5 %. Hence, one and two sigma means that 2 out of 3 or 19 out of 20 results fit the 1σ and 2σ confidence intervals, respectively, provided a sufficiently large number of ages from coeval samples are available. The 1σ confidence interval for the age $A = 2000 \pm 50$ BP is 1950 BP to 2050 BP; one third of the values should be greater or smaller than these two values ($2050 > A > 1950$ BP).

The convention in science and engineering is to give the 1-sigma confidence interval for the uncertainty of a value. An exception is mass spectrometric ages in geochronology, for which the uncertainty is often given as the 2-sigma confidence interval.

The decision whether the 1-sigma or 2-sigma confidence interval is used depends on the question to be answered. An increase of the width of the confidence interval enlarges the probability that the true age is included, but it decreases the ability to distinguish two ages or to decide whether they belong to the same or different frequency distributions. Thus, it is indispensable to state in each publication whether the given uncertainty is 1σ or 2σ .

An example demonstrates this case. Two ages of 1850 ± 50 a and 2000 ± 50 a do not appear to belong to coeval samples due to the gap of 150 a between the 1σ confidence limits. In contrast, samples with the same ages but twice as large standard deviations – 1850 ± 100 a and 2000 ± 100 a – might have been considered as coeval as their confidence intervals overlap. The next chapter describes how such cases are correctly handled.

4 Arithmetic and weighted means

The *arithmetic mean* age \hat{A} (normally simply called the mean) of a set of n numerical ages (A_1 to A_n) with the same precision is given by the following equation:

$$(1) \quad \bar{A} = \frac{\sum_{i=1}^n A_i}{n}.$$

The *standard deviation* σA of the age values A_i in a set of ages A is calculated as follows:

$$(2) \quad \sigma A = \pm \sqrt{\frac{\sum_{i=1}^n (\bar{A} - A_i)^2}{(n-1)}}.$$

Using the three ages 2000 ± 50 a, 2050 ± 50 a and 2200 ± 100 a as an example, Equations 1 and 2 yield (for which the standard deviations of the individual ages are not used) a mean and standard deviation of 2083 ± 104 a. As explained above, this has to be reported as 2080 ± 100 a. (Contrary to the conventional rule for reporting values given in Chapter 2, the values and their uncertainties in this paper are given as calculated and not as they should be reported. This enables the reader to follow the calculations with the corresponding equations).

The larger the number of ages determined for the same sample, the higher the probability that the calculated mean approximates the „true“ age. This is reflected in the standard deviation of the mean ($\sigma \hat{A}$), which decreases with \sqrt{n} as follows:

$$(3) \quad \sigma \bar{A} = \frac{\sigma A}{\sqrt{n}} = \pm \sqrt{\frac{\sum_{i=1}^n (\bar{A} - A_i)^2}{n \cdot (n-1)}}.$$

Our example yields ± 60 a, while that of a single measurement is ± 104 (Eq. 2).

Numerical age values usually have differing precision, requiring the introduction of a *weighting factor* w . It is calculated from the standard deviation as follows:

$$(4) \quad w_i = \frac{1}{\sigma A_i^2}.$$

The weighting factors of our example given above are 0.0004, 0.0004 and 0.0001, respectively. This means the weighting factor for the two ^{14}C ages with a precision of ± 50 years is four times larger than the weighting factor of the ^{14}C age with a precision of ± 100 years. This is not only a mathematical artifact. The third radiometric ^{14}C measurement required four times the time needed for the first two.

x	y	sy	y-sy	y+sy
0,00	3,0	0,50	2,50	3,50
0,05	3,1	0,48	2,62	3,58
0,10	3,2	0,45	2,75	3,65
0,15	3,3	0,43	2,87	3,73
0,20	3,4	0,40	3,00	3,80
0,25	3,5	0,38	3,12	3,88
0,30	3,6	0,36	3,24	3,96
0,35	3,7	0,33	3,37	4,03
0,40	3,8	0,31	3,49	4,11
0,45	3,9	0,29	3,61	4,19
0,50	4,0	0,27	3,73	4,27
0,55	4,1	0,25	3,85	4,35
0,60	4,2	0,23	3,97	4,43
0,65	4,3	0,22	4,08	4,52
0,70	4,4	0,21	4,19	4,61
0,75	4,5	0,20	4,30	4,70
0,80	4,6	0,19	4,41	4,79
0,85	4,7	0,19	4,51	4,89
0,90	4,8	0,19	4,61	4,99
0,95	4,9	0,19	4,71	5,09
1,00	5,0	0,20	4,80	5,20

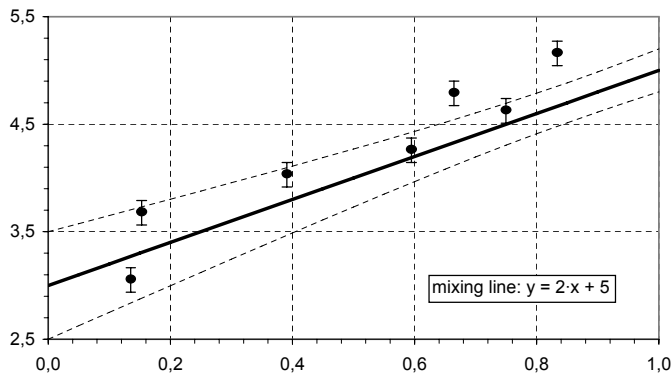


Fig. 4: Linear regression of values to obtain a binary mixing line.

Abb. 4: Lineare Regression von Werten, die einer Zweikomponenten-Mischgerade entsprechen.

The *weighted mean* \hat{A}_w is obtained using the following equation:

$$(5) \quad \bar{A}_w = \frac{\sum_{i=1}^n A_i \cdot w_i}{\sum_{i=1}^n w_i} = \frac{\sum_{i=1}^n \frac{A_i}{\sigma A_i^2}}{\sum_{i=1}^n \frac{1}{\sigma A_i^2}}$$

Equation 5 reduces to Equation 1 if the measurements were done with the same precision: $w_i = \text{constant}$.

The *standard deviation of the weighted mean* $\sigma \hat{A}_w$ is obtained by

$$(6) \quad \sigma \bar{A}_w = \pm \sqrt{\frac{1}{\sum_{i=1}^n w_i}}$$

The weighted mean of our example yields 2044 ± 33 a (to be reported as 2040 ± 30 a), deviating by 35 years from the arithmetic mean of 2083 ± 104 a (to be reported as 2080 ± 100 a) given above. The weighted mean and its standard deviation are shifted towards the more precise results.

Analogous to Equation 3 the *weighted mean standard deviation of a single value* is given by

$$(7) \quad \sigma A_w = \sqrt{n} \cdot \sigma \bar{A}_w.$$

The example yields ± 57 years.

According to Equations 3 and 6 the precision of the mean values $\sigma \hat{A}$ and $\sigma \hat{A}_w$ theoretically approaches zero for an infinite number of ages. In reality the precision has an (often unknown) upper limit resulting from the technical limitations of the method: for example, varying contamination of coeval samples that cannot be removed and slightly deviating measurement conditions (temperature, differing calibration of the equipment in different laboratories, impure chemical reagents, etc.). Ages above the upper limit of the method are given, for example, as $>40,000$ a. They express the minimum age of a sample or the maximum age which can be determined by the method.

5 Chi square test

Calculation of the arithmetic or weighted means \hat{A} or \hat{A}_w of a set of n ages $A_i \pm \sigma A_i$ makes sense only if the σA_i values were determined for coeval samples and therefore belong to the same frequency distribution. To test for this, there are two alternative but equivalent mathematical approaches:

a) The uncertainty $\pm \sigma A$ of the ages is calculated using Equation 2 and compared with the standard deviation σA_w of a single measure-

ment (Eq. 7). The *uncertainty of ages of different precision* is obtained (in analogy to Eq. 2) as follows:

$$(8) \quad \sigma A_g = \pm \sqrt{\frac{\sum_{i=1}^n (\bar{A}_w - A_i)^2 \cdot w_i}{(1 - 1/n) \cdot \sum_{i=1}^n w_i}}$$

It is permissible to calculate the mean \hat{A} only if σA (Eq. 2) or σA_g (Eq. 8) is more or less equal to σA_w (Eq. 7). Our example yields ± 57 a, ± 56 a and ± 104 a, respectively. Hence, the first two standard deviations differ so much from σA_w that the samples cannot be considered to be coeval and the mean should not be calculated.

b) χ^2 test

χ^2 is obtained as follows:

$$(9) \quad \chi^2 = \sum_{i=1}^n (\bar{A} - A_i)^2 \cdot w_i$$

$$\chi_w^2 = \sum_{i=1}^n w_i \cdot A_i^2 - \frac{(\sum_{i=1}^n w_i \cdot A_i)^2}{\sum_{i=1}^n w_i}$$

A mean can be calculated only if χ^2 and $\chi_w^2 \approx n-1$ for $n < 15$. For $n > 15$ the χ^2 value can be taken from any textbook on statistics (e.g., SACHS 1999). Our example yields $\chi^2 = 4.4$ and $\chi_w^2 = 3.2$, confirming the three ages do not belong to the same frequency distribution and thus a mean value would have no validity.

6 Error propagation: difference, rates, mathematical function

There are tasks for which more or less complicated mathematical functions are necessary, e.g., $y = f(x_1, \dots)$. Such functions have a dependent variable y and one or more independent variables $x_1, \dots, x_1, \dots, x_n$. The most simple case is calculation of the difference between two ages which equals the duration of a process. More complicated is, for example, calculation of the sedimentation rate for a certain time interval (Eq. 11). If the uncertainties for the independent variables σx_i are known, the standard deviation of the dependent variable σy can be calculated using Equation 10.

The standard deviation σf of the function $f(x_1,$

$x_2, \dots, x_n)$ is calculated by the law of propagation of uncertainty:

$$(10) \quad \sigma f(x_1, \dots, x_n)^2 = \left(\frac{\partial f}{\partial x_1} \right)^2 \cdot \sigma x_1^2 + \dots + \left(\frac{\partial f}{\partial x_n} \right)^2 \cdot \sigma x_n^2$$

When geological sections are studied, deposition periods and sedimentation rates r between two levels a and b (e.g., at a depth of 100 and 150 cm and ages of 2000 ± 50 and 2200 ± 100 years) are of interest. The function is given by:

$$(11) \quad r = \frac{b - a}{A_2 - A_1}$$

To solve this equation, the difference S between two ages ($A_2 - A_1 = 2200 \pm 100 - 2000 \pm 50$ years) has to be calculated. Neglecting the standard deviation, this example yields a difference of 200 years and a sedimentation rate of 0.25 cm/a. The standard deviation of a *sum* $S = x_1 + x_2$ or a *difference* $\Delta = x_2 - x_1$ is obtained from Equation 12:

$$(12) \quad \sigma S^2 = \sigma \Delta^2 = (\sigma x_1)^2 + (\sigma x_2)^2$$

Our example yields an age difference of 200 ± 112 years. The 1σ and 2σ confidence intervals are 88 to 312 and -24 to +424 years, respectively. The sample ages differ by at least 88 a with a 68 % probability (1σ), but they may be considered as similar with a probability of 95 % (2σ) because a negative age difference is meaningless.

Calculation of the deposition rate r involves a division and two subtraction steps. The corresponding formula involves both multiplication and division:

$$(13) \quad y = \frac{x_1 \cdot x_2}{x_3}$$

The standard deviation of y is obtained as follows:

$$(14) \quad \sigma y = \pm y \cdot \sqrt{\left(\frac{\sigma x_1}{x_1} \right)^2 + \left(\frac{\sigma x_2}{x_2} \right)^2 + \left(\frac{\sigma x_3}{x_3} \right)^2}$$

This corresponding formula for σr is derived from Equations 10 and 12. Assuming depths a and b were determined precisely (i.e., $\sigma a = \sigma b = 0$), the standard deviation of the deposition rate r is obtained by combining Equations 12 and 14 as follows:

$$(15) \quad \sigma r = \pm r \cdot \sqrt{\frac{\sigma A_1^2 + \sigma A_2^2}{(A_2 - A_1)^2}}.$$

According to Equation 15, the standard deviation σr decreases with increasing difference between the ages. The example yields 0.25 ± 0.14 cm/a. The result has 1σ and 2σ confidence intervals (i.e., 68 % and 95 % probabilities) of 0.11 to 0.39 cm/a or 0 to 0.53 cm/a, respectively. Hence, the actual sedimentation rate can be smaller or greater than 0.25 cm/a by a factor of two.

It is worthwhile to note that the formulas for calculating numerical ages usually contain one or more physical parameters that are internationally agreed upon – half lives are an example. Although they were determined with a finite uncertainty, these uncertainties are not usually considered when the law of propagation of uncertainties is applied. Indeed they are not required as long as only numerical ages are compared with each other. But when the ages relate to the absolute time scale, the standard deviation has to include the uncertainties of the internationally agreed parameters and is usually larger than the standard deviation normally reported.

7 Regression analysis and least squares fit

Regression analyses are applied for prediction, testing of hypothesis, and modeling of causal relationships. Such analyses are carried out to determine correlations in numerical data consisting of values of the dependent variable y and the independent variables x_i of a function with one or more parameters, e.g., a and b in Equation (16). These parameters are adjusted to give a best fit of the function to the numerical data. This is usually done using the least squares method. Either the independent variable or the dependent variable is assumed to be a random variable with the uncertainty of the

measurement. The other variable(s) is assumed to be error-free.

The simplest case is a linear function:

$$(16) \quad f(x) = y = b \cdot x + a.$$

Quadratic, exponential and other functions may be transformed into a linear function with a corresponding recalculation of the measured data. Both coefficients a and b are obtained by minimizing the sum of the squares of the deviation of the values y_i from a straight line (the uncertainty in x is assumed to be negligible). The equation is obtained by a least-squares fitting of a straight line to the data:

$$\begin{aligned} \sum \Delta y_i^2 &= \sum [y_i - f(x_i)]^2 = \\ \sum [y_i - a - b \cdot x_i] &= \min. \end{aligned}$$

The values of the parameters a and b are calculated as follows:

$$\begin{aligned} \frac{\partial}{\partial a} \sum w_i \cdot (y_i - a - b \cdot x_i)^2 &= 0 \quad \text{and} \\ \frac{\partial}{\partial b} \sum w_i \cdot (y_i - a - b \cdot x_i)^2 &= 0. \end{aligned}$$

Thus,

$$(17) \quad \begin{aligned} a &= \left(\sum x_i^2 \cdot w_i \sum y_i \cdot w_i - \right. \\ &\quad \left. \sum x_i \cdot w_i \cdot \sum x_i \cdot y_i \cdot w_i \right) / \Delta \\ b &= \left(\sum w_i \cdot \sum x_i \cdot y_i \cdot w_i - \right. \\ &\quad \left. \sum x_i \cdot w_i \cdot \sum y_i \cdot w_i \right) / \Delta \quad \text{where} \\ \Delta &= \sum w_i \cdot \sum x_i^2 \cdot w_i - \left(\sum x_i \cdot w_i \right)^2. \end{aligned}$$

The standard deviations of a and b are given by Equation 18 and shown in Fig. 4:

$$(18) \quad \begin{aligned} \sigma a &= \pm \sqrt{\frac{1}{\Delta} \sum x_i^2 \cdot w_i} \\ \sigma b &= \pm \sqrt{\frac{1}{\Delta} \sum w_i}. \end{aligned}$$

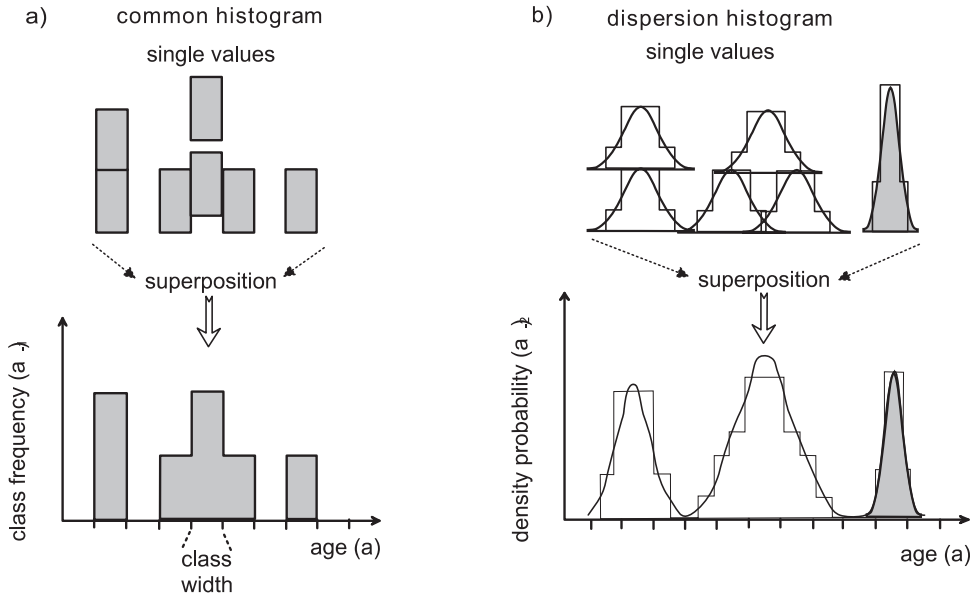


Fig. 5. a) Construction of a normal histogram of five ages based on rectangles, b) Construction of a dispersion histogram of five ages of low (white) and high (gray) precision based on polygons and bell curves, respectively.

Abb. 5. a) Konstruktion eines einfachen Histogramms mit Polygonen für fünf Alter, b) Konstruktion eines Dispersionshistogramms mit Polygonen und Glockenkurven für fünf ungenaue (weiß) und genaue (grau) Alter.

Many computer programs include a least-squares algorithm for fitting a straight line to the data. Often they can also handle composite curves consisting of the superposition of more than one function. A cubic spline can be used to fit a function to a data set. The parameter values of the function are individually chosen to obtain an optimum fit of a curve to all values or to smooth out irregularities in the curve.

8 Graphical presentation: normal and dispersion histograms

Visualization of the frequency distribution of data is often more instructive than a compilation of the data in tables. A *normal histogram* is the most simple way to present ages. The x-axis is assigned an age scale; the y-axis represents the frequency. The x-axis is divided into classes of an arbitrarily chosen width which depends on the age resolution or the average precision

of the available values. Each value is represented by a rectangle with a given area and a class width as base length. All rectangles are superimposed above the x-axis (Fig. 5a). The enveloping curve represents the frequency distribution.

Ages with a mean sigma of ± 50 years (Eq. 7) may be optimally resolved with a class width of 50 years as the base length of the rectangle. If a larger class width is chosen, e.g., 200 years, the optimum age resolution is lost. The advantage of a larger class width is that the number of values per class is increased, which reduces the random scatter of the frequency distribution per class. The uncertainty in a class population is approximated by the square root of the number of values. For instance, four values per class yield $\pm\sqrt{4} = \pm 2$ or $\pm 50\%$ scatter, for nine dates we obtain $\pm\sqrt{9} = \pm 3$ or $\pm 33\%$. As a consequence, the data density per

class governs the random fluctuation of the frequency distribution. If it is small, only minima rather than peaks provide reliable information. The random uncertainty of the histogram peaks may be larger than the actual fluctuations of the frequency of values per class. In contrast, lows always represent periods of missing values, which is why only they allow an unambiguous interpretation of a histogram. They may mirror, for example, arid periods without groundwater recharge, glacial periods without growth of trees, or periods of intensive erosion (see also Section 9.1).

A *dispersion histogram* (GEYH 1969; MICH-CZYNSKA & PAZDUR 2004) provides an alternative visualization of values. A polygon or, even better, a bell-shaped curve with an area representing one value replaces the rectangle of the normal histogram. The width of the area is related to the standard deviation. As the area is constant, its height is determined by the chosen width or standard deviation. Hence, a precise value yields a polygon or bell curve with a short baseline and large height, while a less precise value is represented by a wide baseline and low height (Fig. 5b top).

In a dispersion histogram, the precision of an age represented by its weighting factor w (Eq. 4) is taken into account. Hence, the y-axis represents a density probability function with the dimension of $1/(\text{time unit})^2$ rather than the class frequency with the dimension $1/(\text{time unit})$ (MICH-CZYNSKA & PAZDUR 2004).

There is a special problem with histograms of ^{14}C ages. The time scale is distorted with respect to the solar or absolute time scale. In certain time periods, ^{14}C years are systematically compressed with respect to the solar time scale, in others they are stretched out. This means in the case of a uniform frequency distribution of samples along the absolute time scale that the corresponding ^{14}C histogram may show apparent peaks and lows (GEYH 1980; Fig. 6). In contrast to what is expected, the distortion of the histogram does not disappear if calibrated age intervals are used instead of the ^{14}C ages. Modern calibration programs (e.g., Cal 125 Groningen Radiocarbon Calibration Program)

allow the automated construction of histograms with the calibrated ^{14}C time scale and overcomes the intrinsic problem of calibration of single values.

9 Two case studies

Two case studies are presented which may demonstrate the value of a correct statistical evaluation of ages. It is not the intention of this paper to become involved in the geological aspects of the cited papers or problems inherent in dating methods used. Here, it is mainly considered whether correct handling of the ages gives another numerical result than discussed in the papers.

In the first case study, evidence is provided that increasing the number of numerical age values may not increase the precision of the obtained values. In the second case study, it becomes ob-

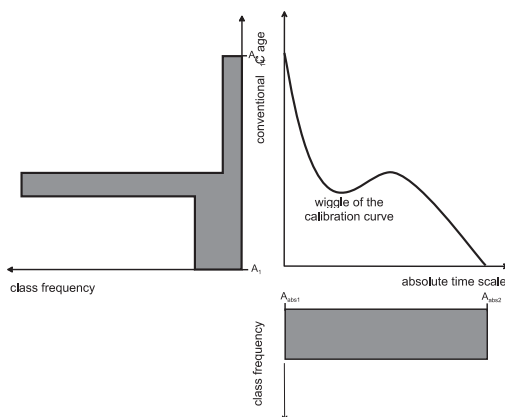


Fig. 6. The histogram between the absolute ages A_{abs1} and A_{abs2} is uniform whereas the histogram of the corresponding ^{14}C dates A_1 and A_2 is distorted. This is the consequence of the wiggles in the ^{14}C calibration curve. Peaks and lows in the ^{14}C histogram may be without actual relevance.

Abb. 6. Das Histogramm der gleich häufigen absoluten Alter A_{abs1} und A_{abs2} entspricht einem Rechteck und liefert für die entsprechenden ^{14}C -Alter ein Polygon. Die Ursache sind die Windungen der ^{14}C -Kalibrationskurve. Gipfel und Täler im ^{14}C -Histogramm zwischen den Altern A_1 und A_2 haben keine wirkliche Bedeutung.

Table 1: ^{14}C ages ordered in size from wood samples dating the beginning of the Younger Dryas in New Zealand (DENTON & HENDY 1994). The standard deviation of the mean (Eq. 7) and the calibrated ^{14}C age of the 2σ confidence interval are given in parentheses.

Tab. 1: Die nach ihrer Größe geordneten ^{14}C -Alter von Holzproben, die den Beginn der Jüngerer Dryas-Zeit in Neuseeland datieren sollen. Die Standardabweichung des mittleren Alters (Gleichung 7) und das 2-Sigma-Konfidenzintervall seines kalibrierten ^{14}C -Alters sind in Klammern angegeben.

^{14}C age (BP) Group I		^{14}C age (BP) Group II	
10,650 ± 100	11,040 ± 90	11,150 ± 160	11,255 ± 95
10,750 ± 100	11,045 ± 85	11,190 ± 60	11,290 ± 150
10,800 ± 90	11,080 ± 60	11,200 ± 50	11,340 ± 110
10,830 ± 110	11,090 ± 70	11,200 ± 120	11,350 ± 90
10,920 ± 90	11,110 ± 110	11,225 ± 60	11,350 ± 60
10,950 ± 100	11,110 ± 90	11,230 ± 130	11,365 ± 60
10,980 ± 90	11,110 ± 130	11,240 ± 100	11,370 ± 190
10,980 ± 100	11,115 ± 90	11,250 ± 70	11,520 ± 170
11,040 ± 70	11,150 ± 60	11,250 ± 50	11,520 ± 140
11,150 ± 14 BP (± 28 a) $\chi^2 = 148$			
13,020 – 13,103 BP (12,965 – 13,117 BP)			
11,011 ± 20 BP (± 32 a) $\chi^2 = 42$		11,265 ± 19 BP (± 13 a) $\chi^2 = 17$	
12,899 – 12,945 BP		13,125 – 13,193 BP	
(12,874 – 12,995 BP)		(13,101 – 13,223 BP)	
10,862 ± 34 BP (± 48 a)	11,097 ± 27 (± 9 a)		
$\chi^2 = 11$	$\chi^2 = 2$		
12,835 – 12,871 BP	12,960 – 13,052 BP		
(12,818 – 12,893 BP)	(12,930 – 13,086 BP)		

vious that the ages were not correctly evaluated in the laboratory and the published standard deviations are meaningless.

9.1 The beginning of the Younger Dryas in New Zealand

DENTON & HENDY (1994) tried to determine the beginning of the Younger Dryas in New Zealand, i.e., the age of a single event. They collected 25 pieces of wood from short-lived trees and covered by Younger Dryas moraines of the Franz-Josef Glacier in the Southern Alps of New Zealand. The holocellulose extract yielded 36 ^{14}C ages determined in three laboratories (Table 1). It was assumed that all samples were coeval and deposited at the beginning of the Younger Dryas.

The weighted mean and its standard deviation of the ^{14}C ages is $11,150 \pm 14$ BP (Eqs. 5 and 6) and the weighted mean standard deviation of a single ^{14}C age is ± 82 a (Eq. 7). DENTON & HENDY (1994) published a mean of $11,170 \pm 14$ BP. The difference between these age values results from the inclusion by the authors of a ^{14}C date which they considered to be older than the target event of the Younger Dryas. The calibrated ^{14}C age interval is $13,020$ – $13,103$ BP ($12,965$ – $13,117$ BP for the 2σ interval). The authors give the mean ^{14}C age as the date for the beginning of the Younger Dryas. The actual standard deviation of the weighted mean, however, is ± 28 a (Eq. 6) and the actual weighted mean standard deviation of the individual ages is ± 169 a (Eq. 8). This finding is not compatible with the assumption that the published ^{14}C

ages belong to a single frequency distribution and, therefore, to a single event. This is supported by the χ^2_w value of 148 for the 36 values. In order to check whether the published ^{14}C ages represent more than one frequency distribution, the ages were ordered according to size and divided into two groups of 18 values each and the first group into two subgroups of nine values (Table 1). Only the weighted means of the second group and the first subgroup belong to a single frequency distribution that represents coeval samples. The other groups represent more than one frequency distribution. The too low χ^2 value for the second subgroup is an artifact of the ordering of the ^{14}C ages according to size.

The present consensus places the beginning of the Younger Dryas between 12,900 and 12,700 BP on the basis of calibrated ^{14}C ages (ALLEY 2000). The calibrated ^{14}C -age range of the weighted mean of the published ^{14}C ages of 12,965–13,117 BP (2σ confidence interval) considerably deviates from that. Only the first group and first subgroup with calibrated ^{14}C age ranges of 12,874–12,995 BP and 12,818–12,893 BP, respectively, agree with this consensus. In both cases several ^{14}C ages have to be discarded as the χ^2 values of 42 and 11 deviate from the target χ^2 values of 17 and 8, respectively.

For the purpose of this paper, it is not of interest to determine the events responsible for this. The wood samples may stem from trees in forests containing trees of different ages that were buried at different times by two or three separate glacier advances. Other causes are possible. Of importance is only that the assumption of the authors the dated samples were coeval is not valid. The precision for the beginning of the studied event was not narrowed to ± 14 years. The actual precision of the date for the beginning of this event is at least twice as large.

The reader may claim that the difference in precision between ± 14 and ± 26 years is small. However, one has to realize that as few as nine ^{14}C ages would have been sufficient to obtain a precision of ± 26 years. This means that up to 75 % of the dating costs could have been saved.

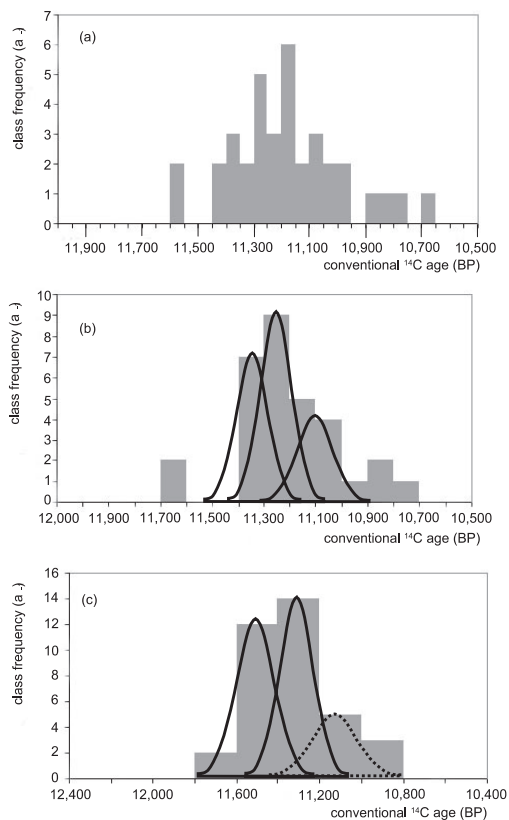


Fig. 7: Normal histogram of 36 ^{14}C ages from 25 wood samples which were assumed to represent the beginning of the Younger Dryas in southern New Zealand (DENTON & HENDY 1994). The arbitrarily chosen class widths of (a) 50, (b) 100 and (c) 200 years shows the influence on the temporal resolution and the scatter of the histogram. The optimum choice may be 100 years (b), which is close to the mean standard deviation of ± 88 years for single ages.

Abb. 7: Einfaches Histogramm von 36 ^{14}C -Altern, die von 25 Holzproben vom südlichen Neuseeland bestimmt worden sind. Sie sollen den Beginn der Jüngeren Dryas-Zeit festlegen (DENTON & HENDY 1994). Die willkürlich gewählten Klassenintervalle von (a) 50, (b) 100 und (c) 200 Jahren zeigen die Auswirkungen auf die altersmäßige Auflösung und auf die Unruhe des Verlaufs des Histogramms. Eine optimale Wahl scheint 100 Jahre zu sein (b), ein Wert, der nahe der mittleren Standardabweichung von Einzeldaten von ± 88 Jahren liegt.

Table 2: ESR ages (ka) of interglacial mollusks from Estonia (MOLODKOV & RAUKAS 1987).

Tab. 2: ESR-Alter (ka) interglazialer Mollusken von Estland (MOLODKOV & RAUKAS 1987).

No	ESR age (ka)	standard deviation (ka)	χ^2 value
1	95±5		0.0
2	90±8	± 3.2	0.3
3	92±7	± 2.5	0.0
4	92±6	± 2.1	0.0
5	92±9	± 2.0	0.0
6	90±8	± 2.0	0.1
7	82±6	± 4.7	2.5
8	81±9	± 5.2	1.1
mean	89.9 ± 2.4 (Eqs. 5 and 6) ± 6.8 (Eq. 7)	88.6 ± 1.8 (Eqs. 3 and 8) ± 5.2 (Eq. 8)	$\chi^2 = 4.0$ target ~ 7

A normal histogram was used for visualization of the ^{14}C ages from New Zealand (Fig. 7). As there are a large number of determined ages, the shape of the normal histogram is very similar to that of a dispersion histogram. The effect of the choice of the class width is demonstrated using 50, 100 and 200 years. The histogram with the smallest class width (Fig. 7, top) shows a large random scatter of up to $\pm 50\%$, which may lead to incorrect statements on the temporal fluctuation of the sample population. The data density per class obviously is too small.

The histogram with a class width of 100 years seems to be the optimum solution. The class width is within the range of 82 to 169 a for the mean standard deviation of the individual ages (Eqs. 7 and 8). The asymmetry of the distribution in the middle histogram suggests the superposition of three frequency distributions with four outliers between 10,700 and 10,900 years and two outliers between 11,600–11,700 years. With the use of the largest class width of 200 years considerable temporal information is lost, but the histogram still clearly shows that the samples represented more than two events.

9.2 ESR ages from mollusks

In the majority of all case studies the scatter of the data corresponds to that expected from the size of the standard deviations or is wider. The-

re are, however, studies in which the opposite case occurs. In these cases, the defined criteria of the expression $A \pm \sigma A$ are not fulfilled and calculations with such dates should be done without the described error algorithms.

MOLODKOV and RAUKAS (1987) published ESR ages from mollusks collected from an interglacial layer on the coast of Estonia. The results are compiled in Table 2.

The actual uncertainty of the ESR ages (Eq. 3 derived from Eq. 8) is considerably smaller than the expected one calculated from the given standard deviations (Eq. 6). Consequently, $\chi^2_w = 4.0$ (Eq. 9), which is also considerably smaller than 7 (the number of ages minus 1). This result is evidence that the propagation of uncertainty of the published ESR ages was not done properly. This is a frequent observation in published ESR, TL and OSL ages. The standard deviations are too large or too small. A discussion of the reasons for this problem would be beyond the scope of this article. One of the main problems is that the statistical uncertainty of several of the parameters involved can only be estimated or only minimum and maximum values are known for the parameters. In addition, systematic errors are often underestimated. In any case, in such cases it is not justified to use weighting factors in the statistical handling of the numerical ages determined with these methods.

10 Conclusion

It is demonstrated that the handling of numerical ages with their random uncertainties requires at least a minimum knowledge of simple statistical methods, especially the algorithms used to determine the propagation of uncertainty. A careless or unqualified use of such dates bears the risk of misleading geoscientific conclusions.

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Quaternary Science Journal

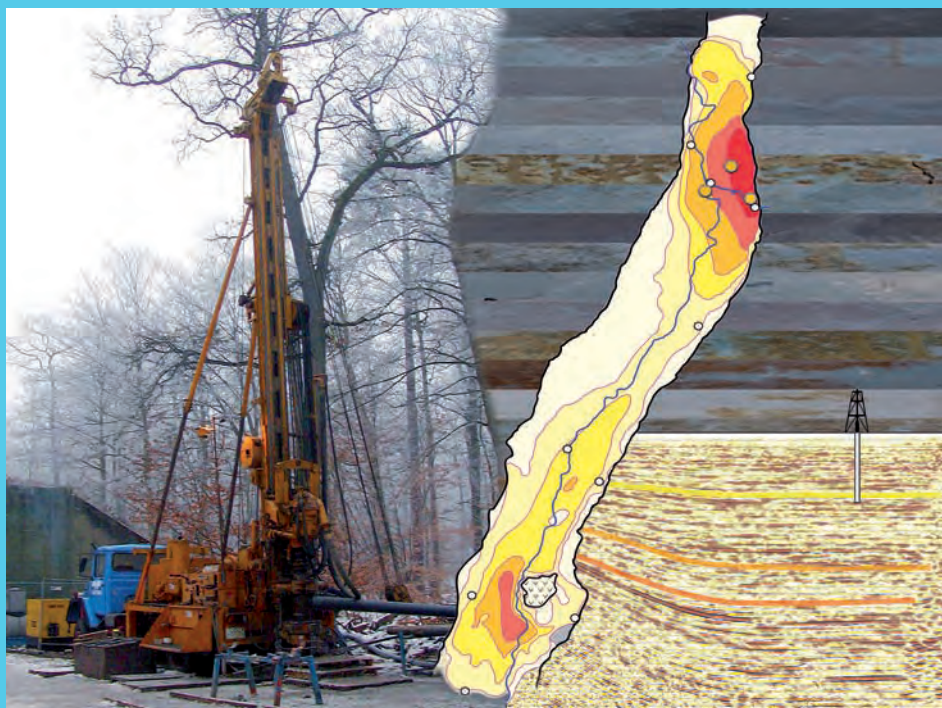
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Quaternary Science Journal



Special issue:

The Heidelberg Basin Drilling Project

Guest editors: Gerald Gabriel, Dietrich Ellwanger,
Christian Hoselmann and Michael Weidenfeller



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Titelbild: Collage „Bohrprojekt Heidelberger Becken“ (Gestaltung: Juliane Herrmann, Leibniz-Institut für Angewandte Geophysik) Bohranlage an der Lokation Viernheim (Foto: Christian Hoselmann, Hessisches Landesamt für Umwelt und Geologie) – Mächtigkeitkarte der quartären Sedimente im Oberrheingraben (Hessisches Landesamt für Umwelt und Geologie) – reflexionsseismisches Profil in der Umgebung der Forschungsbohrungen Heidelberg UniNord (Hermann Bunes, Leibniz-Institut für Angewandte Geophysik) – Bohrkerndetails (Fotos: Christian Hoselmann, Hessisches Landesamt für Umwelt und Geologie)

Front cover: collage „Heidelberg Basin Drilling Project“ (design: Juliane Herrmann, Leibniz Institute for Applied Geophysics) drilling rig at the Viernheim location (photo: Christian Hoselmann, Geological Survey of Hessen) – thickness map of Quaternary sediments in the Upper Rhine Graben (Geological Survey of Hessen) – reflection seismic profile in the vicinity of the research boreholes Heidelberg UniNord (Hermann Bunes, Leibniz Institute for Applied Geophysics) – core details (photos: Christian Hoselmann, Geological Survey of Hessen)

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Preface: The Heidelberg Basin Drilling Project

GERALD GABRIEL, DIETRICH ELLWANGER, CHRISTIAN HOSELMANN & MICHAEL WEIDENFELLER

Since Late Pliocene / Early Pleistocene, the River Rhine, as one of the largest European rivers, has acted as the only drainage system that connected the Alps with Northern Europe, especially the North Sea. Along its course from the Alps to the English Channel the river passes several geomorphological and geological units, of which the Upper Rhine Graben acts as the major sediment trap (Fig. 1). Whereas the potential of sediment preservation of the alpine foreland basins is low due to the high dynamics of the system, and the area of deposition close to the North Sea was significantly affected several times by Pleistocene sea level changes, the ongoing subsidence of the Upper Rhine Graben offers a unique potential for a continuous sediment accumulation and preservation. The two major sediment traps are the Geiswasser Basin to the south and the Heidelberg Basin farther to the north. Generally the mean grain size of the deposited alpine sediments in the Upper Rhine Graben decreases from south to north. In the Upper Rhine Graben, the Heidelberg Basin acts as the distal sediment trap for alpine sediments transported northwards by the River Rhine. Here, continuous sediment deposition is less disturbed by significant unconformities than in the south. Therefore, the Heidelberg Basin defines a key location to understand the glacial evolution of the Alps since Late Pliocene, and moreover to compare it with that of Northern Europe (ELLWANGER et al. 2005).

The Heidelberg Basin hosts one of the thickest successions of Plio-/Pleistocene sediments in continental Mid-Europe. The Radium Sol Therme borecore drilled during 1913-1918, is located in the centre of the city of Heidelberg, close to the River Neckar. It is controversially discussed in literature. „Base Quaternary“ was interpreted to be at depths of 382 m (BARTZ 1951), 400 m (SALOMON 1927), or even 650 m

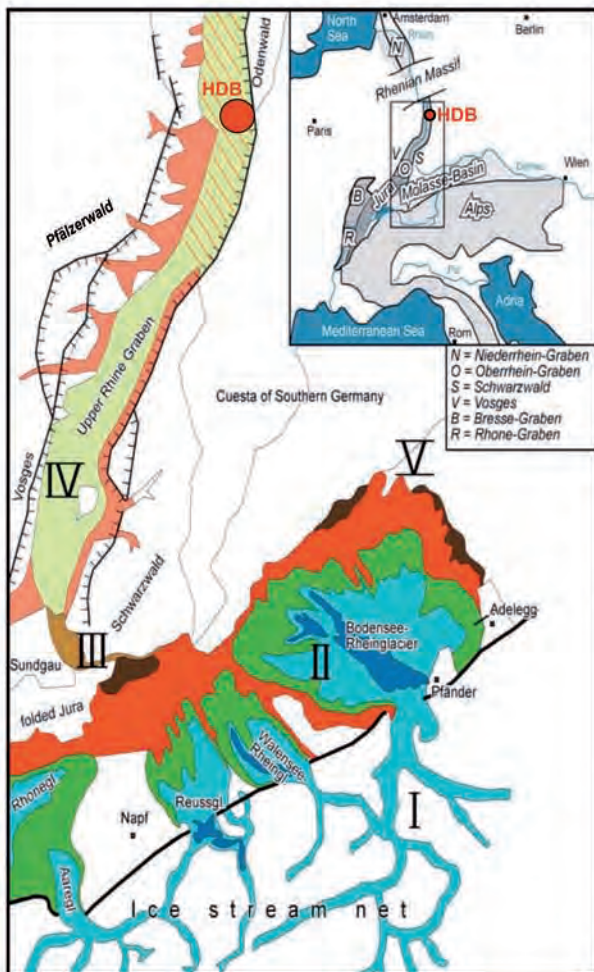
(FEZER 1997). Nowadays, from extensive reflection seismics, it is well known that Quaternary deposits are thicker than 300 m. Nevertheless, because reflection seismic surveys were only conducted in the vicinity of Heidelberg but not within the city itself, no detailed information about Quaternary structures has been available for this specific area up to now.

Aiming to better understand the geological evolution of the Heidelberg Basin, its control by climate changes and tectonics, and the correlation of alpine and north European glacial evolution, the Heidelberg Basin Drilling Project was promoted by the Leibniz Institute for Applied Geophysics (LIAG – formerly Leibniz Institute for Applied Geosciences, GGA-Institute) and the three Geological Surveys of Baden-Württemberg, Hessen, and Rheinland-Pfalz. The project focuses on the evolution of the basin since Late Pliocene. Investigations are mainly based on newly cored boreholes at three different locations within the Heidelberg Basin, which represent different types of the basin facies (Fig. 2). Two 300 m deep, cored boreholes in the city of Ludwigshafen – finished in 2002 and 2006 – represent the western margin of the basin. These boreholes were carried out within the framework of groundwater exploration and then left to the Geological Survey of Rheinland-Pfalz. The 350 m deep, cored borehole close to the village of Viernheim should reveal information about the central basin facies. This borehole was carried out and sponsored by the Geological Survey of Hessen and finished in summer 2006. The last of the cored boreholes, the borehole Heidelberg UniNord, is located above the depocentre of the basin, on the eastern margin of the Rhine Graben. This borehole reached its final depth of 500 m in July 2008 and obtained financial support from the Leibniz Institute for Ap-

Fig. 1: The Heidelberg Basin (HDB) within the Geosystem „Rhine“ (modified after ELLWANGER et al. 2003, 2005).

- Small map: The Upper Rhine Graben as main sediment trap of the Rhine between the Alps and the North Sea.
- Large map: Various landsystems along the Rhine from the Alps to the Upper Rhine Graben.

- I – Inneralpine (overdeepened) valleys and the ice stream net of the last ice advance.
- II – The landsystem of glacial basins and lakes at the alpine margin, including various ice margins and the ice cover of the last ice advance.
- III – Hochrhein valley, terraces and buried valleys.
- IV – Upper Rhine Graben and the Upper Rhine lowlands, in the southern part mainly sediments of alpine provenance, in the northern part (hatched) mainly sediments of local provenance (Black Forest, Vosges).
- V – The classical pre-alpine meltwater land-system, type region of the classical glacial units of the Würmian, the Rißian, the Mindelian and the Günzian.



- I – inneralpine ice stream net during last ice advance

- II – landsystem of glacial basins at the alpine margin
 - covered by ice during the last advance
 - Würm-Maximum
 - Riss-Maximum
 - most extensive ice advance

- III – Hochrhein Valley
 -

- IV – Upper Rhine Graben resp. lowlands
 - alpine input
 - local input
 - (hatched) mainly local, but with alpine signals

- V – classical alpine type region of the Quaternary

Tab. 1: Lithostratigraphic units as introduced by the three Geological Surveys working on the Heidelberg Basin.

Baden-Württemberg <i>Symbolschlüssel Geologie Baden-Württemberg</i>		Rheinland-Pfalz <i>Weidenfeller & Kärcher 2008 Weidenfeller & Knipping 2008</i>	Hessen	Bartz 1982
			<i>Aeolian sand (Pleistocene to Holocene)</i>	
Mannheim-Formation		Upper gravel layer (OKL)	Sand-gravel layers <i>Neckar dominated, less alpine</i>	Oberes Kieslager (OKL) <i>contains coarse material (alpine and local)</i>
Kurpfalz-Formation	Ladenburg Horizon	Upper interlayer (OZH)	Interlayer <i>predominant fine-clastic sediments</i>	Obere Zwischenschicht (OZ) <i>only fine material</i>
	Weinheim Beds	Middle sand-gravel layer	Alternation of sand-(gravel) layers frequently in "Rhenish Facies" and	Mittleres Kieslager (MKL) <i>contains coarse material</i>
		Lower interlayer (UZH)		Untere Zwischenschicht (UZ) <i>only fine material</i>
		Lower sand-silt layer	fine-clastic interlayers <i>Rhine (alpine) material, less Neckar</i>	Unteres Kieslager (UKL) <i>contains coarse material</i>
major change of provenance				
Iffezheim-Formation		Clay-silt-sand-layer	Clay-silt- and sand-layers <i>material of local provenance</i>	Pliocene I-III * <i>material of local provenance</i>

* rather an indication of a sediment type than a strong geochronological classification

plied Geophysics and the Geological Survey of Baden-Württemberg. Combining all new cored boreholes, 1450 m of core material is available now for a detailed investigation program. At present, no uniform classification for the Plio-/Pleistocene deposits is available for the three federal states, that are working on the Heidelberg Basin, namely Baden-Württemberg, Hessen, and Rheinland-Pfalz. Within each Geological Survey some mandatory guidelines exist that match the specific requirements derived from the local geology. The geology of the southern Upper Rhine Graben on the territory of Baden-Württemberg is significantly affected by the deposition of coarse-grained sediments eroded in the Alps. Therefore, a classification was introduced that considers major unconformities and therefore the dynamic of the sedimentary system in the southern Upper Rhine Graben (SYMBOLSCHLÜSSEL GEOLOGIE BADEN-WÜRTTEMBERG 2007). In contrast, the terminology used in Hessen and Rheinland-Pfalz is built on the systems introduced by

BARTZ (1982) and HGK (1999), which distinguish between hydrogeological units. At this early stage of the Heidelberg Basin project, it is impossible to set up a uniform nomenclature. Table 1 summarizes the different classifications used in this special issue. To overcome these discrepancies, investigations in the Heidelberg Basin project must preliminary focus on the establishment of a reference profile of the region north of the Alps, including petrographic, sequence stratigraphic, biostratigraphic, and magnetostratigraphic approaches, complemented by geochronological and geophysical data. Beyond these local to regional aspects, these boreholes offer a sediment archive of supra-regional relevance, particularly regarding the correlation between the glacial evolution in the alpine region and that of Northern Europe. In this context, proxies of past environmental / climate change will be also derived. These aims can only be achieved because the drilled cores reveal a high temporal resolution without significant disconformities

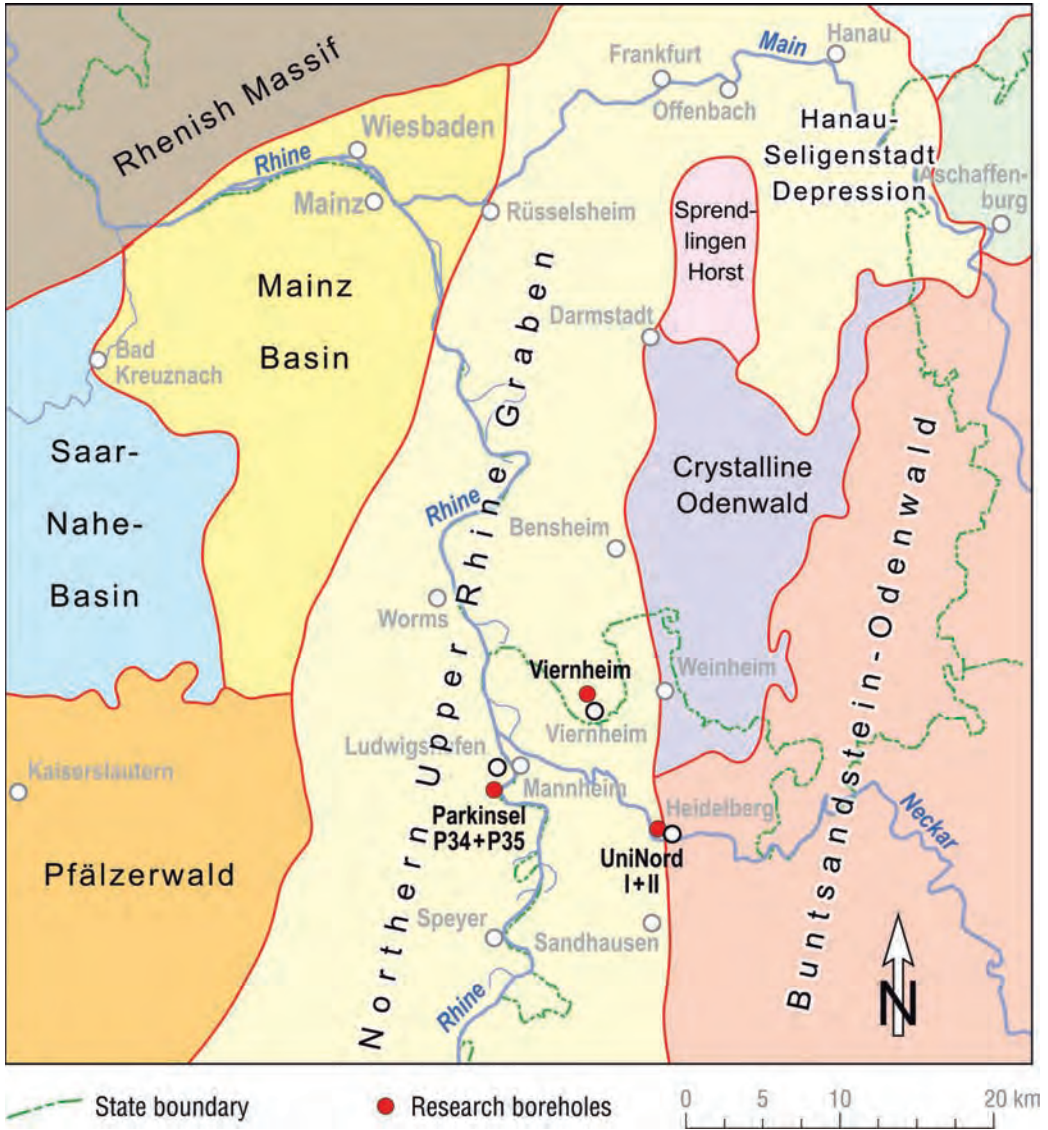


Fig. 2: Geological structural zones with the three borehole locations Ludwigshafen-Parkinsel (cored boreholes P34 and P35, both 300 m deep), Viernheim (350 m deep), and Heidelberg UniNord (research boreholes Heidelberg UniNord 1, 190 m deep, and Heidelberg UniNord 2, 500 m deep) in the Heidelberg Basin.

(HOSELMANN et al. 2008). The papers compiled in this special volume of the Quaternary Science Journal (*Eiszeitalter und Gegenwart*) prove that the drilled cores can be considered as nearly ideal with respect to these aspects.

Due to the availability of the two boreholes in Ludwigshafen at an early stage in this joint project, a comprehensive dataset already exists for this location. WEIDENFELLER & KNIPPING (2008) present complementary and consistent datasets as heavy mineral analysis, pollen assemblages and the core description itself. Somewhat surprising the results of the two Ludwigshafen-Parkinsel boreholes differ in some aspects significantly, although the locations lie only 500 m apart. Whereas the upper sections of both boreholes correlate well, thicknesses of individual layers in the deeper part are quite variable. Even the transition between Pliocene and Pleistocene deposits was found at different depths. Consequently this region seems to be affected by tectonics during Upper Pliocene/Lower Pleistocene. These results emphasize that the interpretation of the climatic and fluvial history of an area on the basis of the analysis of only a few boreholes becomes very difficult, not only because the sediments were affected by fluvial dynamics, but also by small-scale tectonics. The distinct characterisation and stratigraphic identification based on several independent methods, and on pollen analysis in particular, are basic requirements for a reliable interpretation of the sedimentation history controlled both by climate and tectonics.

Considering the data available so far, a pattern similar to that in Ludwigshafen is found in the Viernheim borehole (HOSELMANN 2008). Based on heavy mineral data, carbonate content, and petrography, the transition Plio/Pleistocene is seen at 225 m depth. However, this boundary is not sharp, but imaged as a transition zone consisting of a reworked horizon. The Pleistocene sediment succession is dominated by alpine sediments, local deposits from the Neckar occur only occasionally. The main part of the Pleistocene section is made up of ten repeated suites that begin with gravely sand layers and end with silty clay sediments or peat. Due to miss-

ing pollen data, a detailed correlation with the Ludwigshafen boreholes has not been possible so far. However, HOSELMANN (2008) presents a first correlation with other boreholes of the Viernheim-Bensheim region, based on reference markers, e.g. the occurrence of the „Rhenish Facies“. Although a general trend of increasing thickness of the Rhenish Facies towards the centre of the Heidelberg Basin in the south is depicted, the fundamental problem of hampered correlation due to the absence of particular beds at some borehole locations is also addressed.

The last of the boreholes to finish was the Heidelberg UniNord at the end of July 2008 (ELLWANGER et al. 2008). Based on the information from the reflection seismic surveys and the old Radium Sol Therme borehole, this location was expected to represent the depocentre of the Heidelberg Basin. With respect to the first palynological data, this assumption seems to be confirmed. Even at 420 m depth Quaternary pollen assemblages are found. If this can be confirmed by more detailed studies in future at this location, the thickness of Pleistocene sediments would be about three times more than in Ludwigshafen; therefore the temporal resolution will be increased by the same amount. Furthermore, the results of the reflection seismic surveys and the pollen data are promising, regarding the completeness of the profile. The interglacials – Cromerian, Bavelian, Waalian, and Tiglian – could already be identified by a first scan of the pollen assemblages. To some extent the cores reveal strong and fast changes of the accommodation space and sediment supply ratio. The mostly-fluviatile character of the depositional environment was temporarily interrupted by lacustrine periods.

To close the gap of seismic information in the centre of the Heidelberg Basin and to ensure that the boreholes reveal thick and more or less complete sediments, geophysical pre-site surveys were conducted at the Viernheim and the Heidelberg borehole locations (BUNESS, GABRIEL & ELLWANGER 2008). In close relation to this project, the first gravity map of the Upper Rhine Graben that comprises all available French and German data was com-

piled (ROTSTEIN et al. 2006). This map clearly images the course of the Upper Rhine Graben by the occurrence of negative gravity anomalies, of which the strongest is observed in the area around Heidelberg. This could be an indicator for a sediment succession of unusual large thickness. Furthermore, in the vicinity of the borehole Heidelberg UniNord reflection seismic profiles were recorded of which one N-S profile contributed significantly to the decision about the location for the borehole (BUNESS, GABRIEL & ELLWANGER 2008). This profile imaged for the first time a sub basin within the Heidelberg Basin. The usefulness of a seismic pre-site survey was also well demonstrated by the investigations around the borehole in Viernheim. Without the seismic information the borehole would have penetrated a fault at only 200 m depth – something that should be avoided with regard to the goals of this project. Based on the results of the seismic surveys, the borehole location could be relocated by several hundred meters.

One of the challenges of this project is the correlation between the three locations of the new boreholes, and beyond this, the correlation with additional, shallower boreholes in the area of the Heidelberg Basin. In addition to lithostratigraphic or biostratigraphic approaches results from geophysical downhole logging can also reveal additional insight into the sedimentary system. Downhole logging data is of particular value, wherever core quality is low or – even worse – core is not recovered, because it provides in-situ information about the sediment successions. HUNZE & WONIK (2008) discuss the physical properties of the different lithologies on the basis of logging data and suggest a hole-to-hole correlation between several locations in the Heidelberg Basin. Furthermore, by statistical analysis of specific data sets additional information about sediment provenance could be derived. With sediments deposited by the rivers Rhine and Neckar, two main sediment provenances could be distinguished.

Facing the challenge of stratigraphic correlation, but also that of climate reconstruction, WEDEL (2008) analysed core material from both the Viernheim and Ludwigshafen locations with

regard to their fossil content, and particularly the remains of molluscs. The potential for the preservation of molluscs is highly variable. Fossils are often found in the argillaceous layers of the fine-grained horizons (Zwischenhorizonte), and to a minor amount also in the fine and medium-grained sand fraction of the coarse-grained horizons (Kieslager). The Pliocene beds do not contain any significant remnants of molluscs. Especially from a Mid-Pleistocene horizon, distinct information about several interglacials can be revealed that are suitable for correlation with pollen data and a reconstruction of the palaeo-environment. As a highlight, two mollusc species and one rodent species from the Lower Pleistocene (Lower Biharium) were identified in the northern Upper Rhine Graben for the first time.

First pollen spectra from the research borehole Heidelberg UniNord 1 are discussed by HAHNE, ELLWANGER & STRITZKE (2008). Although only four short sections with well-preserved pollen were found, with the evidence for a Waalian thermomer, a first biostratigraphic marker of regional relevance was found. The pollen spectrum derived from the peaty sequence between 180 and 181.7 m depth is very similar to the flora of the type-locality for the Waalian (the Leerdam section in the Netherlands), both in pollen genera and quantities. Especially the occurrence of *Tsuga* strengthens the interpretation as Waalian. From these first findings a uniform forest vegetation in Mid Europe for the early Pleistocene is concluded, representing a climate that was not warmer than today.

In future, correlation between all the available boreholes must be based on a combination of several independent observations. Beside geophysical data and palynological data, absolute geochronological data is required especially. Unfortunately, no dating method is available that can be applied to the entire Quaternary age spectrum. Young sediments (up to ~150 ka) can be dated by applying luminescence dating techniques. Although in the meantime some few and unpublished results are available for the new cored-boreholes in the Heidelberg Basin (pers. communication Lauer), the method has been tested before with fluvial sediments of

the Bremgarten section in the southern part of the Upper Rhine Graben (FRECHEN et al. 2008). The results prove that OSL dating is a suitable method for fluvial sediments from large river systems. Insufficient bleaching of the sediments prior to deposition seems to be not as dramatic as previously thought. The most important finding for the Bremgarten section is a short period of major erosion and re-sedimentation of fluvial sediments from the „Tiefgestade“ at the Bremgarten section between 500 and 600 years before present, which correlates with the begin of the Little Ice Age ca. AD 1450. Likewise interesting results are expected for the Heidelberg Basin Drilling Project in the future.

This special issue on the Heidelberg Basin Drilling Project is published at an early stage of a just-starting joint research project that comprises aspects from many geoscientific disciplines. The intention of this issue is to make some main results already available to the geoscientific community, e.g. the reflection seismic data or the lithologs of the new-cored boreholes. In fact, detailed analytic investigation of the core material has not begun. Therefore, a uniform interpretation of the three borehole locations cannot be presented, which becomes especially obvious with the use of the terms „Base Quaternary“ or „transition Plio-Pleistocene“. But the cored boreholes should provide unique material to solve these challenges during the next years. Some aspects of this study have been submitted as a proposal to the German Research Foundation in 2008.

These eight contributions to this special issue of the Quaternary Science Journal (Eiszeitalter und Gegenwart) present the most recent findings from ongoing research on the Heidelberg Basin. Thereby, they complement some papers dealing with „The Rhine – a major fluvial record“, which is the title of a special issue of the Netherlands Journal of Geosciences published in 2008, edited by Wim Westerhoff. This special issue addresses, for instance, issues like the tectonic influence on the preservation of fluvial sediments (WEIDENFELLER & KÄRCHER 2008), palynological investigations (KNIPPING 2008), heavy mineral analysis (HAGEDORN & BOENIGK

2008), and rock and paleomagnetic studies (ROLF, HAMBACH & WEIDENFELLER 2008).

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Prerequisite for this special issue about the Heidelberg Basin Drilling Project was the offer from Holger Freund as editor of Quaternary Science Journal (Eiszeitalter und Gegenwart) to publish a special issue. He organised the review process with great patience. But the most important work was done by the many authors who contributed with their papers to this issue. The corresponding comments of many reviewers helped to improve these papers. Many thanks to all colleagues involved!

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Vorwort: Das Bohrprojekt „Heidelberger Becken“

GERALD GABRIEL, DIETRICH ELLWANGER, CHRISTIAN HOSELMANN & MICHAEL WEIDENFELLER

Seit dem späten Pliozän / frühen Pleistozän stellt der Rhein als einer der größten europäischen Flüsse das einzige Entwässerungssystem dar, welches die Alpen mit der Nordsee verbindet. Auf seinem Verlauf von den Alpen bis hin zum Ärmelkanal passiert er unterschiedliche geomorphologische und geologische Einheiten, von denen der Oberrheingraben die Hauptsedimentfalle bildet (Abb. 1). Während die alpinen Vorlandbecken aufgrund der hohen Dynamik des Gesamtsystems nur ein geringes Erhaltungspotenzial hinsichtlich der Sedimentablagerung aufweisen, und das Ablagerungsgebiet unmittelbar an der Nordseeküste mehrfach signifikant durch pleistozäne Meeresspiegelschwankungen beeinflusst wurde, bietet die andauernde Subsidenz des Oberrheingrabens einmalige Bedingungen für die kontinuierliche Akkumulation von Sedimenten. Die beiden größten Sedimentfällen sind dabei das Geiswasser Becken im südlichen Teil sowie das Heidelberger Becken im Nordosten. Generell nimmt die mittlere Korngröße der im Oberrheingraben abgelagerten alpinen Sedimente von Süden nach Norden ab. Das Heidelberger Becken fungiert als distale Falle im Oberrheingraben für alpine Sedimente, die durch den Rhein Richtung Norden transportiert werden. Hier ist die kontinuierliche Ablagerung von Sedimenten weniger stark durch Diskontinuitäten gestört als im südlichen Teil des Rheingrabens. Daher stellt das Heidelberger Becken eine Schlüsselposition für das Verständnis der glazialen Entwicklung der Alpen seit dem späten Pliozän und darüber hinaus für einen Vergleich mit der glazialen Entwicklung Nordeuropas dar (ELLWANGER et al. 2005).

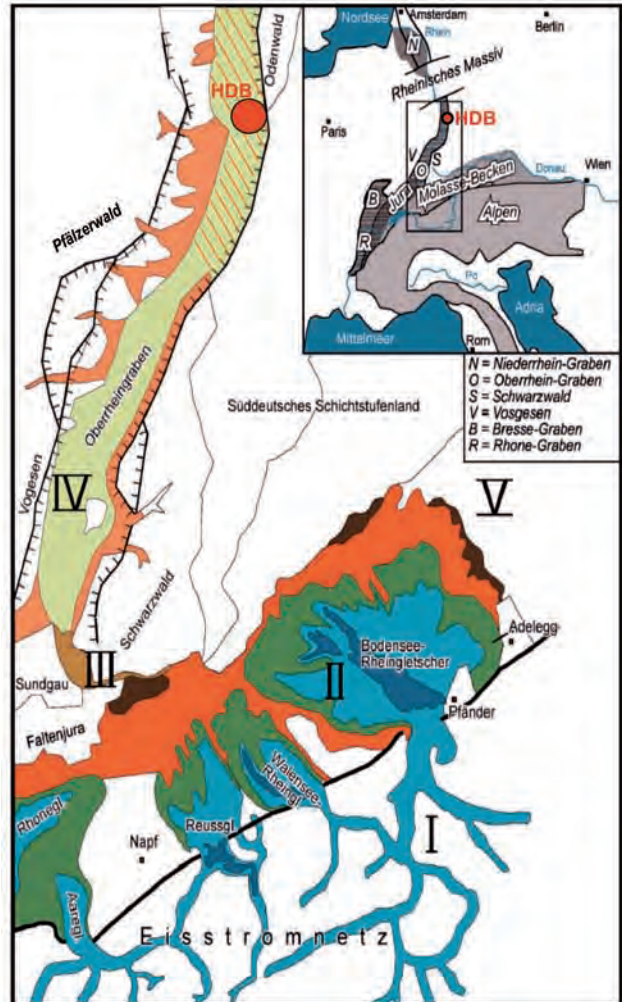
Das Heidelberger Becken enthält eine der mächtigsten Abfolgen plio-/pleistozäner Sedimente im kontinentalen Mitteleuropa. Die Ra-

dium Sol Therme Bohrung, welche zwischen 1913 und 1918 im Zentrum von Heidelberg abgeteuft wurde, wird in der Literatur bis heute kontrovers diskutiert. Die „Quartärbasis“ wird in Tiefen von 382 m (BARTZ 1951), 400 m (SALOMON 1927) oder sogar 650 m (FEZER 1997) gesehen. Durch umfassende reflexionsseismische Messungen ist heute belegt, dass die quartären Ablagerungen Mächtigkeiten größer 300 m erreichen. Da reflexionsseismische Untersuchungen jedoch nur in der Umgebung von Heidelberg durchgeführt wurden, nicht jedoch im Stadtgebiet selbst, waren bislang keine detaillierten Informationen über quartäre Strukturen in diesem Bereich verfügbar.

Mit dem Ziel, zu einem tieferen Verständnis hinsichtlich der geologischen Entwicklung des Heidelberger Beckens, insbesondere der Steuerung durch Klimaveränderungen und Tektonik, sowie der Korrelation der alpinen und nordeuropäischen Vereisungsgeschichte zu gelangen, wurde das Bohrprojekt „Heidelberger Becken“ durch das Leibniz-Institut für Angewandte Geophysik (LIAG – vormals Institut für Geowissenschaftliche Gemeinschaftsaufgaben, GGA-Institut) und die drei geologischen Dienste von Baden-Württemberg, Hessen und Rheinland-Pfalz initiiert. Das Projekt fokussiert auf die Beckenentwicklung seit dem späten Pliozän. Die Untersuchungen basieren wesentlich auf neuen Kernbohrungen an drei verschiedenen Lokationen innerhalb des Heidelberger Beckens, welche unterschiedliche Faziesräume abbilden (Abb. 2). Zwei 300 m tiefe Kernbohrungen in Ludwigshafen – abgeteuft 2002 und 2006 – repräsentieren den westlichen Rand des Beckens. Diese Bohrungen wurden im Rahmen der Grundwassererkundung realisiert und dem Geologischen Dienst von Rheinland-Pfalz zur Verfügung gestellt. Die 350 m tiefe Kernboh-

Abb. 1: Das Heidelberger Becken (HDB) im Geosystem „Rhein“ (verändert nach ELLWANGER et al. 2003, 2005).

- Kleine Karte: Der Oberrhein-graben als Hauptsedimentfalle des Rheins zwischen Alpen und Nordsee.
 - Große Karte: Verschiedene Landschaftstypen entlang des Rheins von den Alpen bis zum Oberrheingraben.
- I – Inneralpine (übertiefe) Täler und die Eisströme beim letzten Vorstoß.
 - II – Randalpine Becken- und Seenlandschaft mit verschiedenen Eisrandlagen und der Eisbedeckung beim letzten Vorstoß.
 - III – Hochrheintal, Terrassenstufen und verschüttete Täler.
 - IV – Oberrheingraben bzw. oberrheinische Tiefebene, Lockersedimente im Südtteil überwiegend alpiner Herkunft, im Nordteil (schräg schraffiert) überwiegend lokaler Herkunft (Schwarzwald, Vogesen).
 - V – Die klassische oberschwäbische Schmelzwasserterrassenlandschaft (Typregion für Würm, Riss, Mindel und Günz).



- I – inneralpine Eisströme beim letzten Vorstoß
- II – randalpine Beckenlandschaft
 - Eisbedeckung beim letzten Vorstoß
 - Würm-Maximum
 - Riss-Maximum
 - weitester Vorstoß
- III – Hochrheintal
- IV – Oberrheingraben bzw. oberrheinische Tiefebene
 - alpine Schüttungen
 - lokale Schüttungen
 - (schräg schraffiert) überwiegend lokal
- V – klassische alpine Quartär-Typusregion

Tab. 1: Lithostratigraphische Einheiten, wie sie durch die drei Geologischen Dienste verwendet werden, welche Anteile am Heidelberger Becken haben.

Baden-Württemberg <i>Symbolschlüssel Geologie Baden-Württemberg</i>		Rheinland-Pfalz <i>Weidenfeller & Kärcher 2008 Weidenfeller & Knipping 2008</i>	Hessen	Bartz 1982
			Äolische Sande (Pleistozän bis Holozän)	
Mannheim-Formation		Oberes Kieslager (OKL)	Sand-Kies Lagen <i>Neckar-dominiert, wenig alpin</i>	Oberes Kieslager (OKL) <i>Grobsedimente (alpin und lokal)</i>
Kurpfalz-Formation	Ladenburg Horizont	Oberer Zwischenhorizont (OZH)	Zwischenhorizont <i>vorwiegend feinklastische Sedimente</i>	Obere Zwischenschicht (OZ) <i>nur Feinsedimente</i>
	Weinheim Schichten	Mittlere sandig-kiesige Folge	Wechsel von Sand-(Kies) Lagen häufig in "Rheinischer Fazies" und feinklastischen Zwischenhorizonten <i>(alpine) Rheinsedimente, wenig Neckar</i>	Mittleres Kieslager (MKL) <i>Grobsedimente (alpin und lokal)</i>
		Unterer Zwischenhorizont (UZH)		Untere Zwischenschicht (UZ) <i>nur Feinsedimente</i>
		Untere sandig-siltige Folge		Unteres Kieslager (UKL) <i>Grobsedimente (alpin und lokal)</i>
				Altquartär 1 und 2 (AQ1 und AQ2)* <i>Übergangsbereich mit ersten alpinen Sedimenten</i>
	Wechsel des Hauptliefergebietes			
Iffezheim-Formation		Ton-Sand-Silt Folgen	Ton-Silt und Sand Lagen <i>Sedimente lokalen Ursprungs</i>	Pliozän I-III * <i>Sedimente lokalen Ursprungs</i>

* eher die Bezeichnung eines Sedimenttyps, denn eine strenge geochronologische Einordnung

rung bei Viernheim soll Informationen über die zentrale Beckenfazies liefern. Diese im Sommer 2006 abgeschlossene Bohrung wurde durch den Geologischen Dienst von Hessen betreut und finanziert. Die letzte Kernbohrung, Heidelberg UniNord, wurde im Subsidenzzentrum des Beckens am östlichen Rand des Oberrheingrabens angesetzt. Diese durch das Leibniz-Institut für Angewandte Geophysik und den Geologischen Dienst von Baden-Württemberg finanzierte Bohrung hat ihre Endteufe von 500 m im Juli 2008 erreicht. Alle neuen Kernbohrungen zusammen liefern 1450 m Kernmaterial, das nun für ein detailliertes Untersuchungsprogramm zur Verfügung steht. Bislang existiert in den Bundesländern mit Anteilen am Heidelberger Becken (Baden-Württemberg, Hessen und Rheinland-Pfalz) keine einheitliche Gliederung der plio-/pleistozänen Ablagerungen. In jedem geologischen Dienst gibt es verbindliche Vorgaben, welche den spezifischen Gegebenheiten

der lokalen Geologie Rechnung tragen. Die Geologie des südlichen Oberrheingrabens in Baden-Württemberg wird maßgeblich durch die Ablagerung grobklastischer Sedimente bestimmt, die in den Alpen abgetragen wurden. Daher wird eine Formationsgliederung angewendet, die im südlichen Graben die Hauptdiskontinuitäten und somit die Dynamik des Systems berücksichtigt (SYMBOLSCHLÜSSEL GEOLOGIE BADEN-WÜRTEMBERG 2007). Dagegen basiert die in Hessen und Rheinland-Pfalz verwendete Terminologie zum Teil auf den von BARTZ (1982) und durch die HGK (1999) eingeführten Systemen, die zwischen hydrogeologischen Einheiten unterscheiden. In diesem frühen Stadium des Projekts „Heidelberger Becken“ ist die Definition einer einheitlichen Nomenklatur noch nicht möglich. Tabelle 1 stellt die in diesem Themenheft verwendeten unterschiedlichen Gliederungen gegenüber. Um diese Diskrepanzen zu überwinden, müssen die Untersuchungen im Projekt „Heidelberger

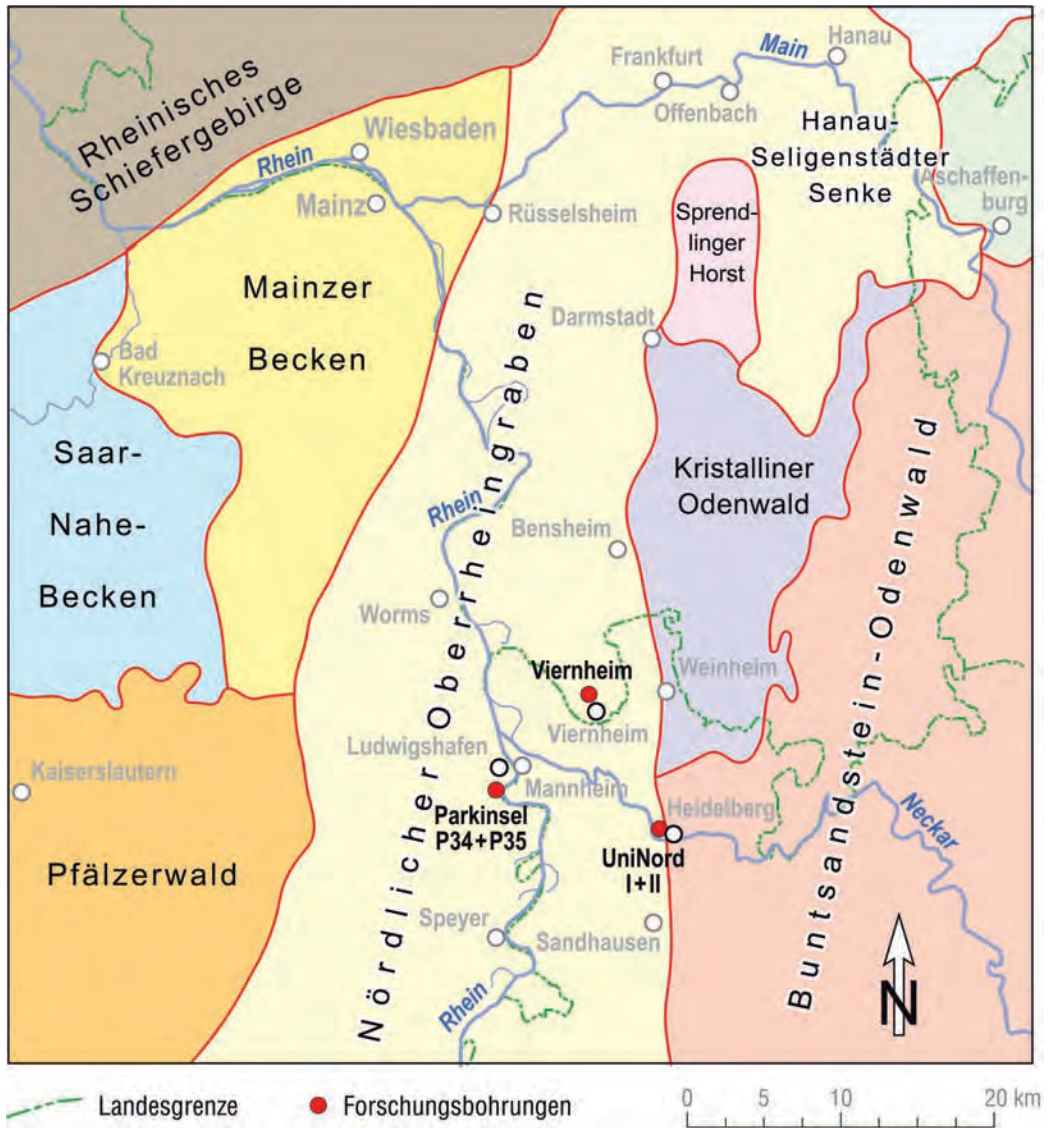


Abb. 2: Geologische Strukturräume mit den drei Bohrlokalationen Ludwigshafen-Parkinsel (Kernbohrungen P34 und P35, jeweils 300 m tief), Viernheim (350 m tief) und Heidelberg UniNord (Forschungsbohrungen Heidelberg UniNord 1, 190 m tief und Heidelberg UniNord 2, 500 m tief) im Heidelberger Becken.

Becken“ zunächst auf die Erstellung eines Referenzprofils für das Gebiet nördlich der Alpen ausgerichtet sein. Dieses beinhaltet petrographische, sequenzstratigraphische, biostratigraphische und magnetostratigraphische Ansätze, die durch geochronologische und geophysikalische Daten ergänzt werden. Über diese lokalen bis regionalen Aspekte hinaus ist mit den neuen Bohrungen im Hinblick auf die Korrelation der glazialen Entwicklungen im Alpenraum und Nordeuropa ein Sedimentarchiv von überregionaler Bedeutung erschlossen worden. In diesem Zusammenhang sollen auch Proxy für Umweltveränderungen/Klimaveränderungen in der Vergangenheit abgeleitet werden. Diese Ziele können nur erreicht werden, weil die gebohrten Kerne eine hohe zeitliche Auflösung und nur wenige Schichtlücken aufweisen (HOSELMANN et al. 2008). Die in diesem Themenheft vom Quaternary Science Journal (Eiszeitalter und Gegenwart) zusammengestellten Artikel zeigen, dass die gebohrten Kerne unter diesen Aspekten als nahezu ideal angesehen werden können.

Aufgrund der Verfügbarkeit der beiden Kernbohrungen in Ludwigshafen zu einem sehr frühen Zeitpunkt in diesem Projekt, liegt für diese Lokation aktuell der umfangreichste Datensatz vor. WEIDENFELLER & KNIPPING (2008) präsentieren sich ergänzende konsistente Datensätze von Schwermineralanalysen, Pollenanalysen und der Kernbeschreibung selbst. Etwas überraschend unterscheiden sich die Ergebnisse der beiden Bohrungen Ludwigshafen-Parkinsel P34 und P35 in einzelnen Aspekten signifikant, obwohl beide Lokationen nur etwa 500 m auseinander liegen. Während die oberen Abschnitte beider Bohrungen gut korrelieren, sind die Mächtigkeiten einzelner Einheiten des unteren Teils lateral sehr variabel. Selbst der Übergang Plio-/Pleistozän wurde in unterschiedlichen Teufen angetroffen. Folglich erscheint diese Region während des späten Pliozäns/frühen Pleistozäns maßgeblich durch Tektonik beansprucht worden zu sein. Diese Ergebnisse belegen, dass die Ableitung der klimatischen und fluviatilen Entwicklung einer Region un-

sicher ist, sofern diese nur auf einer Bohrung beruht. Die Sedimente werden nicht nur durch die fluviatile Dynamik beeinflusst, sondern auch durch kleinmaßstäbliche tektonische Ereignisse. Die auf verschiedenen unabhängigen Untersuchungen, insbesondere auch Pollenanalysen, basierende Charakterisierung und stratigraphische Einordnung der Sedimente ist grundlegende Voraussetzung für eine verlässliche Interpretation der durch Klima und Tektonik gesteuerten Ablagerungsgeschichte. Unter Berücksichtigung des bislang vorliegenden Datensatzes ergibt sich für die Bohrung Viernheim ein Bild, das dem in Ludwigshafen sehr ähnlich ist (HOSELMANN 2008). Auf Schwermineralanalysen, Karbonatgehalten und der Petrographie basierend, wird der Übergang Plio-/Pleistozän bei 225 m gesehen. Allerdings handelt es sich dabei um keinen scharfen Übergang, sondern um einen Bereich aufgearbeiteter Horizonte. Die pleistozäne Sedimentabfolge wird durch alpines Material dominiert, lokale Ablagerungen des Neckars treten nur untergeordnet auf. Der wesentliche Teil des pleistozänen Abschnitts wird aus zehn sich wiederholenden Abfolgen aufgebaut, welche mit kiesig-sandigen Lagen beginnen und mit schluffig-tonigen Sedimenten enden. Aufgrund noch fehlender Pollenanalysen kann bislang keine detaillierte Korrelation mit den Bohrungen in Ludwigshafen vorgenommen werden. Jedoch stellt HOSELMANN (2008) basierend auf Leithorizonten, wie dem ersten Auftreten der „Rheinischen Fazies“, erste Korrelationen mit weiteren Bohrungen aus der Region Viernheim-Bensheim vor. Wenngleich der generelle Trend einer Zunahme der Mächtigkeit der rheinischen Fazies zum Zentrum des Heidelberger Beckens hin aufgezeigt werden kann, wird auch das fundamentale Problem der erschwerten Korrelation infolge an bestimmten Lokationen fehlender einzelner Schichtpakete angesprochen.

Die Heidelberg UniNord Bohrung wurde erst Ende Juli 2008 fertig gestellt (ELLWANGER et al. 2008). Basierend auf den Ergebnissen der reflexionsseismischen Erkundung und der alten Bohrung Radium Sol Therme wird davon ausgegangen, dass diese Lokation das Depozent-

rum des Heidelberger Beckens repräsentiert. Diese Annahme scheint durch die ersten palynologischen Daten bestätigt zu werden. Selbst in 420 m Teufe ist das Pollenspektrum noch quartärzeitlich. Sollte sich dieses Ergebnis zukünftig durch detaillierte Untersuchungen erhärten, wäre die Mächtigkeit der pleistozänen Sedimente an dieser Lokation etwa dreifach so groß wie in Ludwigshafen und es würde sich die erwartete hohe zeitliche Auflösung bestätigen. Darüber hinaus werden die Ergebnisse der reflexionsseismischen Messungen und der Pollendaten als viel versprechend im Hinblick auf die Vollständigkeit des Profils angesehen. Bereits bei der ersten Durchsicht der Pollenspektren konnten die Interglaziale Cromer, Bavel, Waal und Tigium identifiziert werden. Die Bohrkerne zeigen teilweise starke und auch schnelle Änderungen im Verhältnis von Akkommodationsraum zu Sedimentangebot. Der meist fluviatil geprägte Ablagerungsraum wird zeitweise von lakustrinen Perioden abgelöst.

Um die Lücke an seismischen Informationen im Zentrum des Heidelberger Beckens zu schließen und sicher zu stellen, dass die Bohrungen mächtige und vergleichsweise vollständige Sedimente erschließen, wurden an den Bohrlokationen Viernheim und Heidelberg UniNord geophysikalische Vorerkundungen durchgeführt (BUNESS, GABRIEL & ELLWANGER 2008). In enger Verknüpfung mit diesem Projekt wurde die erste Schwerekarte des Oberrheingraben erstellt, die sämtliche verfügbaren Daten von französischer und deutscher Seite berücksichtigt (ROTSTEIN et al. 2006). Diese Karte bildet durch das Auftreten negativer Schwereanomalien deutlich den Verlauf des Oberrheingraben ab, von denen die größte im Bereich um Heidelberg zu beobachten ist. Dies kann als Hinweis auf Sedimentabfolgen von ungewöhnlich großer Mächtigkeit angesehen werden. Ferner wurden im Umfeld der Bohrung Heidelberg UniNord reflexionsseismische Profile aufgenommen, von denen ein N-S Profil maßgeblich zur Festlegung des Bohrpunktes beigetragen hat (BUNESS, GABRIEL & ELLWANGER 2008). Dieses Profil bildet zum ersten Mal ein Subbecken im Heidelberger Becken ab. Der Mehr-

wert einer seismischen Vorerkundung wurde ebenfalls durch die Untersuchungen rund um die Bohrlokation bei Viernheim demonstriert. Ohne die seismischen Informationen hätte die Bohrung bereits in einer Tiefe von 200 m eine Störung durchteuft – ein Sachverhalt, der im Hinblick auf die Ziele dieses Projekts unbedingt vermieden werden sollte. Die Ergebnisse der seismischen Messungen berücksichtigend konnte der Bohransatzpunkt somit um einige hundert Meter verschoben werden.

Eine der Herausforderungen in diesem Projekt liegt sicherlich in der Korrelation zwischen den drei Lokationen der neuen Bohrungen sowie darüber hinaus in der Korrelation mit weiteren, weniger tiefen Bohrungen im Bereich des Heidelberger Beckens. In Ergänzung zu lithostratigraphischen oder biostratigraphischen Ansätzen können auch bohrlochgeophysikalische Messungen Einblicke in das sedimentäre System liefern. Bohrlochgeophysikalische Daten sind dort von besonderem Wert, wo die Kernqualität schlecht ist, oder – noch ungünstiger – keine Kerne gewonnen werden konnten, da sie in-situ Informationen über die Sedimentabfolgen zur Verfügung stellen. HUNZE & WONIK (2008) diskutieren die physikalischen Eigenschaften der verschiedenen Lithologien auf der Basis von Bohrlochmessungen und schlagen eine Korrelation zwischen verschiedenen Bohrpunkten im Heidelberger Becken vor. Darüber hinaus werden durch die Anwendung statistischer Verfahren auf spezifische Datensätze Informationen bezüglich der Liefergebiete der Sedimente abgeleitet. Mit Sedimentablagerungen durch den Rhein bzw. den Neckar können zwei unterschiedliche Liefergebiete unterschieden werden.

Ebenfalls die stratigraphische Korrelation betreffend, aber auch Aspekte der Paläo-Klimarekonstruktion, analysiert WEDEL (2008) Kernmaterial der Bohrungen Viernheim und Ludwigshafen unter paläontologischen Aspekten, insbesondere im Hinblick auf Molluskenreste. Das Erhaltungspotenzial für Mollusken erscheint hochgradig variabel. Fossilien werden oftmals in den lehmig/tonigen Schichten der feinkörnigen Lagen (Zwischenhorizonte)

gefunden, untergeordnet auch in den feinen und mittelfeinen Sanden der grobkörnigen Ablagerungen (Kieslager). In den pliozänen Ablagerungen finden sich keinerlei Molluskenreste. Eindeutige Informationen über mehrere Interglaziale können insbesondere aus einem Horizont entnommen werden, welcher dem mittleren Pleistozän zugeordnet wird. Diese Daten sind sowohl für die Korrelation mit Pollenanalysen als auch für die Rekonstruktion der Paläo-Umweltbedingungen geeignet. Ein „Highlight“ stellt der erstmalige Fund von zwei Molluskenarten sowie einer Nagetierart im nördlichen Oberrheingraben aus dem unteren Pleistozän (Unteres Biharium) dar.

HAHNE, ELLWANGER & STRITZKE (2008) diskutieren erste Pollenanalysen der Forschungsbohrung Heidelberg UniNord 1. Wenngleich gut erhaltene Pollen lediglich in vier kurzen Abschnitten aufgefunden wurden, konnte mit der Identifikation der Waal-Warmzeit ein biostratigraphischer Marker bestimmt werden, der auch überregional von Bedeutung ist. Das der torfigen Sequenz zwischen 180,0 und 181,7 m zugehörige Pollenspektrum ähnelt sowohl hinsichtlich der Pollentypen als auch der Pollenhäufigkeit stark der Flora des Typus-Profiles für das Waalium (Profil Leerdam, Niederlande). Insbesondere das Auftreten von *Tsuga* bedingt die Einordnung in die Waal-Warmzeit. Basierend auf diesen ersten Ergebnissen wird eine einheitliche Bewaldung Mitteleuropas im Zeitraum des frühen Pleistozäns postuliert, die ein Klima repräsentiert, das nicht wärmer war als das heutige.

Korrelationen zwischen den verschiedenen Bohrungen müssen zukünftig auf der Kombination der verschiedenen unabhängigen Verfahren aufbauen. Neben geophysikalischen und palynologischen Daten sind vor allem absolute Altersdatierungen notwendig. Leider steht kein Datierungsverfahren zur Verfügung, welches auf das gesamte Quartär angewendet werden kann. Junge Sedimente (bis zu ~150 ka) können mit Lumineszenztechniken datiert werden. Obwohl inzwischen auch erste unpublizierte Ergebnisse für die neuen Kernbohrungen im Heidelberger Becken vorliegen (Lauer, pers.

Mitteilung), wurde die Methode zunächst an fluviatilen Sedimenten des Profils Bremgarten aus dem südlichen Teil des Oberrheingrabens getestet (FRECHEN et al. 2008). Die Ergebnisse zeigen, dass OSL-Techniken für die Datierung fluviatiler Sedimente großer Flusssysteme geeignet sind. Eine ungenügende Bleichung der Sedimente vor der Ablagerung scheint weniger großen Einfluss auf die Ergebnisse zu haben, wie bislang angenommen. Das wesentliche Ergebnis hinsichtlich des Profils Bremgarten besteht in der Ermittlung eines kurzen Abschnitts starker Erosion und Resedimentation fluviatiler Sedimente des Tiefegestades vor 500 bis 600 Jahren. Dieses Ereignis korreliert mit dem Beginn der kleinen Vereisung etwa 1450 AD. Ähnlich interessante Datierungsergebnisse sind zukünftig im Rahmen des Bohrprojektes „Heidelberger Becken“ zu erwarten.

Dieses Themenheft über das Heidelberger Becken erscheint zu einem frühen Zeitpunkt eines gerade beginnenden Forschungsvorhabens, welches Aspekte diverser geowissenschaftlicher Disziplinen vereint. Die Intention besteht darin, verschiedene grundlegende Ergebnisse, wie die Daten der Reflexionsseismik oder die Lithologs der neuen Kernbohrungen, der geowissenschaftlich arbeitenden Gemeinschaft verfügbar zu machen. Tatsächlich fehlen noch verschiedene detaillierte Untersuchungen am Kernmaterial. Daher kann zum jetzigen Zeitpunkt keine gemeinsame Interpretation des gesamten Kernmaterials vorgestellt werden, was sich insbesondere in der unterschiedlichen Verwendung der Begriffe „Quartärbasis“ oder „Übergang Plio-/Pleistozän“ niederschlägt. Mit den neuen Bohrkernen sollte aber einzigartiges Material zur Verfügung stehen, um diese Aufgabe in den kommenden Jahren lösen zu können. Ein Teil der Untersuchungen soll im Rahmen eines Antragspakets durchgeführt werden, welches 2008 bei der Deutschen Forschungsgemeinschaft eingereicht wurde.

Die acht Beiträge zu diesem Themenheft des Quaternary Science Journal (Eiszeitalter und Gegenwart) präsentieren die aktuellsten Ergebnisse andauernder Forschungsarbeiten zum Heidelberger Becken. Damit komplettieren

sie verschiedene Artikel, welche sich mit dem „Rhein – ein bedeutendes fluviatiles Sedimentarchiv“ befassen. Dies ist der frei übersetzte Titel eines Sonderbands des *Netherlands Journal of Geosciences*, der 2008 unter der Editorenschaft von Wim Westerhoff publiziert wurde. Dieser Sonderband spricht ergänzende Themen, wie den Einfluss der Tektonik auf die Erhaltung fluviatiler Sedimente (WEIDENFELLER & KÄRCHER 2008), palynologische Untersuchungen (KNIPPING 2008), Schwermineralanalysen (HAGEDORN & BOENIGK 2008) sowie gesteins- und paläomagnetische Studien (ROLF, HAMBACH & WEIDENFELLER 2008) im Oberrheingraben an.

Dank

Bohrprojekte stellen stets eine herausfordernde Aufgabe dar, die Unterstützung vieler Personen und Institutionen verlangt. Das Bohrprojekt „Heidelberger Becken“ hat wesentlich von der Unterstützung der derzeitigen und früheren Leiter der beteiligten Einrichtungen profitiert, namentlich Prof. Dr. U. Yaramanci, Prof. Dr. H.-J. Kämpel, Prof. Dr. R. Watzel, Ltd. Bergdirektor V. Dennert, Prof. Dr. B. Stribny, Dr. R. Becker und Prof. Dr. H. Ehses. Die Kernbohrungen Ludwigshafen-Parkinsel wurden durch die Technischen Werke Ludwigshafen zur Verfügung gestellt. Logistische Unterstützung gaben die Universität Heidelberg in Form der Bereitstellung des Grundstücks für die Bohrung Heidelberg UniNord 1, Helmut Huber sowie das Amt für Vermögen und Bau Baden-Württemberg mit der Bereitstellung des Geländes für die Bohrung Heidelberg UniNord 2 und der Springer-Verlag. Dr. E. Würzner, Oberbürgermeister der Stadt Heidelberg, und Dr. R. Franke, Stadtwerke Viernheim, vermittelten Kontakte, wo immer notwendig. Die beteiligten Mitarbeiter des Amtes für Umweltschutz der Stadt Heidelberg und der Universität Heidelberg waren stets am Fortgang des Projekts interessiert und gaben jegliche Hilfe, die notwendig war. Für die gesamte Unterstützung herzlichen Dank!

Voraussetzung für dieses Themenheft über das Bohrprojekt „Heidelberger Becken“ war

das Angebot von PD Dr. H. Freund als Editor von *Quaternary Science Journal* (Eiszeitalter und Gegenwart) einen Sonderband zu publizieren. Er organisierte mit großer Geduld das Begutachtungsverfahren. Die wichtigsten Arbeiten wurden jedoch durch die vielen Autoren durchgeführt, die mit ihren Artikeln zu diesem Themenheft beitragen. Die Anmerkungen der Gutachter gaben wesentliche Hinweise für die Verbesserung einzelner Manuskripte. Vielen Dank an alle Kollegen, die sich beteiligt haben!

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Correlation of Pleistocene sediments from boreholes in the Ludwigshafen area, western Heidelberg Basin

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Abstract: Cores from several boreholes in the Ludwigshafen area were analysed to investigate their sedimentology, palynology, palaeomagnetism, rock magnetism and heavy mineral composition. The preliminary results are presented from the new Ludwigshafen-Parkinsel borehole P35, which was drilled 500 m WSW of borehole P34, to a total depth of 300 m. Correlation between the two boreholes reveals similarities and dissimilarities in stratigraphy, structure and the thickness of the sediments. As a result of core documentation and the preliminary evaluation of the investigation results, a good correlation is established between the coarse and fine-grained sequences in both boreholes down to a depth of 122 m. However, the Plio-Pleistocene boundary in borehole P35 is much deeper than in P34. A fault throw of 42 m is assumed, attributable to young tectonics. The poor correlation between the thicknesses of the sediments in the lower sections of the two boreholes suggests that tectonism was particularly active in the Pliocene and Lower Pleistocene. The different occurrence of interglacial sequences in the two Ludwigshafen boreholes can be attributed to fluvial dynamics and neotectonic events. Further palynological analysis is required to determine whether the alternation of at least five interglacial periods determined in the Ludwigshafen-Parkinsel P34 borehole, can also be confirmed in the P35 borehole. The information gained so far from the correlation of the already analysed Middle Pleistocene interglacials in the Ludwigshafen/Mannheim area, as well as the links with the primarily Lower Pleistocene sections in Schifferstadt, already suggest that this would allow a much better understanding of the changes in vegetation and climate during the Pleistocene.

[Korrelation pleistozäner Sedimente aus Bohrungen im Raum Ludwigshafen, westliches Heidelberger Becken]

Kurzfassung: Im Raum Ludwigshafen wurden mehrere Kernbohrungen sedimentologisch, palynologisch, paläomagnetisch, gesteinsmagnetisch und schwermineralogisch untersucht. Erste Ergebnisse der neuen Bohrung Ludwigshafen-Parkinsel P35 werden vorgestellt, die 500 m WSW der Bohrung P34 bis 300 m abgeteufelt wurde. Die Gegenüberstellung beider Bohrungen zeigt Übereinstimmungen, aber auch Unterschiede im Aufbau, Struktur und Mächtigkeit der Sedimente. Nach der Bohrkerndokumentation und ersten Auswertungen von Untersuchungsergebnissen lassen sich die grob- und feinkörnigen Sequenzen aus beiden Bohrungen bis in eine Teufe von 122 m gut miteinander korrelieren. Allerdings liegt die Plio-/Pleistozängrenze in der Bohrung P35 deutlich tiefer. Wahrscheinlich ist ein Versatzbetrag von 42 m anzunehmen, der auf junge Tektonik zurückzuführen ist. Die geringe Übereinstimmung der Mächtigkeiten in den tieferen Abschnitten der Bohrungen lässt vermuten, dass die Tektonik besonders im Pliozän und Unterpleistozän aktiv war. Die unterschiedliche Präsenz von warmzeitlichen Sequenzen in den beiden Ludwigshafener Bohrungen kann auf fluviale Dynamik und neotektonische Ereignisse zurückgeführt werden. Ob die in der Bohrung Ludwigshafen Parkinsel P34 erfassten Wechsel von mindestens 5 Warmzeiten auch in der Bohrung P35 bestätigt werden können, bleibt weiteren palynologischen Untersuchungen vorbehalten. Schon jetzt lässt die Korrelation zwischen den bereits bearbeiteten mittelpleistozänen Warmzeiten im Raum Ludwigshafen/Mannheim sowie die Verknüpfung mit den überwiegend altpleistozänen Abschnitten von Schifferstadt eine deutliche Kenntniserweiterung der pleistozänen Vegetations- und Klimaentwicklung erwarten.

Keywords: Upper Rhine Graben, Heidelberg Basin, Pleistocene, fluvial sediments, pollen analysis, neotectonics

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1 Introduction

To investigate hydrogeological aspects, numerous boreholes were cored in the Ludwigshafen/Mannheim area in recent years. These cores were analysed using a wide range of methods (Fig. 1, Table 1). The results of the Ludwigshafen-Parkinsel P34 borehole in particular – which was drilled to a TD (total depth) of 300 m in 2002 with 95 % core recovery – provided a great deal of new information on the history of the river Rhine and the changes in vegetation and climate in the northern Upper Rhine Graben during the Pleistocene (WEIDENFELLER & KÄRCHER 2008).

Of particular value were the palynological analyses (KNIPPING 2008), heavy mineral analyses (HAGEDORN & BOENIGK 2008) and the palaeomagnetic and rock-magnetic analyses (ROLF et al. 2008). This made it possible for the first time to reliably detect the Plio-Pleistocene boundary in the northern Upper Rhine Graben in a core, confirmed by various independent methods. Another core of 300 m (P35) was drilled approximately 500 m WSW from borehole P34 in 2006. P35 was analysed using a very similar range of methods. The results are presented in this paper, including a preliminary correlation between the two boreholes.

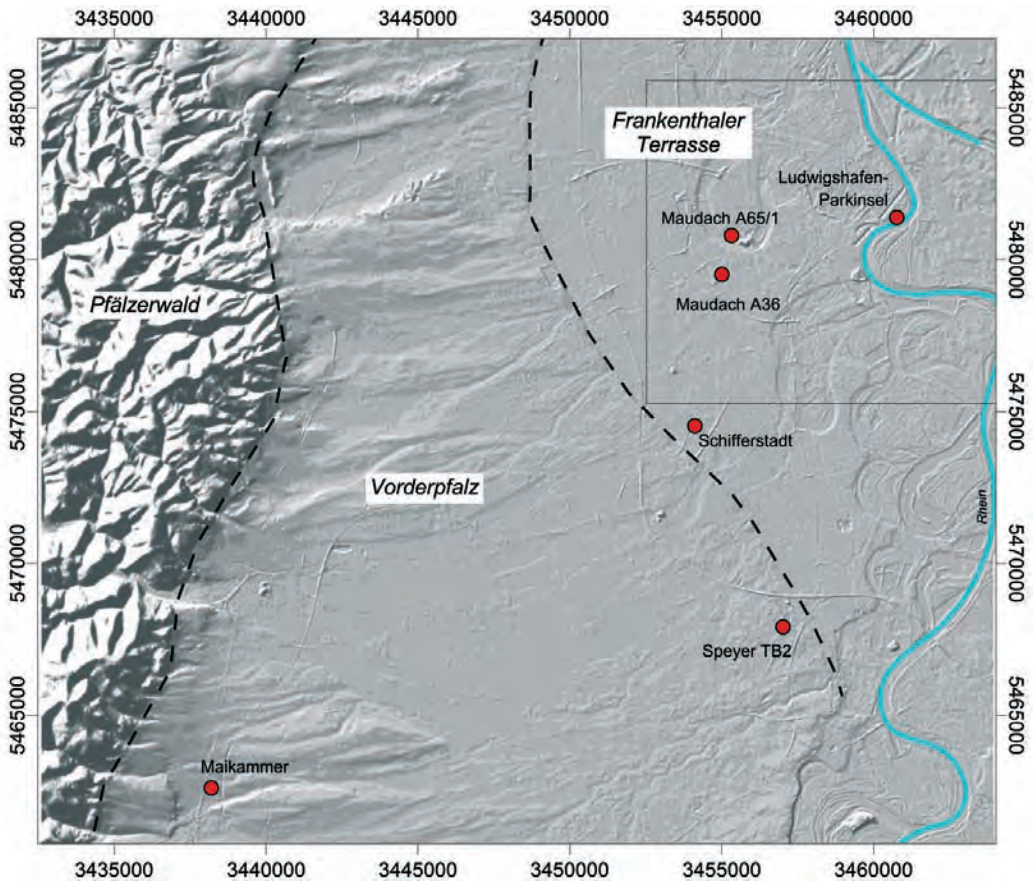


Fig. 1: Position of the investigated boreholes in the Vorderpfalz, on the Frankenthaler Terrasse and in the Ludwigshafen area.

Abb. 1: Lage untersuchter Bohrungen in der Vorderpfalz, auf der Frankenthaler Terrasse und im Raum Ludwigshafen.

The results of the P34 and P35 cores are the key for the interpretation of the unconsolidated sedimentary sequences in the Ludwigshafen/Mannheim area. The sequences in both cores are characterised by alternating coarse-grained and fine-grained horizons. They are stratigraphically subdivided using a hydrogeological terminology (BARTZ 1959, 1974, KÄRCHER 1987) into aquifers (Oberes Kieslager "OKL" (Upper Gravels), Mittlere sandig-kiesige Folge (Middle Sandy-Gravelly Series), Untere sandig-siltige Folge (Lower Sandy-Silty Series)) and Zwischenhorizonte (intermediate horizons)

(Oberer Zwischenhorizont "OZH" (Upper Intermediate Horizon), Unterer Zwischenhorizont "UZH" (Lower Intermediate Horizon)). Reddish-brown sandy sediments were encountered in some sections and interpreted on the basis of heavy mineralogical analysis as fans deriving from the Pfälzerwald (HAGEDORN 2004, HAGEDORN & BOENIGK 2008).

Whereas the stratigraphic position of the Lower Pleistocene sediments is uncertain, the upper gravels ("OKL") are conventionally attributed to a Weichselian age and referred to in the terminology of terrace division as a Lower

Table 1: Investigations of cores from boreholes Ludwigshafen-Parkinsel P34, P35 and Ludwigshafen-Maudach A36, TK 25 6516 Mannheim-Südwest

Tab. 1 : Untersuchungen an Kernen der Bohrungen Ludwigshafen-Parkinsel P34, P35 und Ludwigshafen-Maudach A36, TK 25 6516 Mannheim-Südwest

	Lu-Parkinsel P34	Lu-Parkinsel P35	Lu-Maudach A36
Coordinate (R-Wert)	34 60 666	34 60 230	34 54 850
Coordinate (H-Wert)	54 81 069	54 80 990	54 79 490
Depth	300 m	300 m	50.6 m
Surface	92 m NN	92 m NN	97 m NN
Description of the borehole sections	WEIDENFELLER (unpublished)	WEIDENFELLER (unpublished)	WEIDENFELLER (unpublished)
Lithofacies	HAGEDORN (2004), WEIDENFELLER & KÄRCHER (2008)	this volume	-
Sedimentology	KONTNY & SCHULTE (unpublished)	SIROCCO & SCHABER (unpublished)	-
Gamma Log	BLM, München (unpublished)	BLM, München (unpublished)	-
Thermal conductivity	-	WERNER (2006)	-
Thin-section investigations	MENZIES (2006)	-	-
Pollen analysis	KNIPPING (2008, in progress)	KNIPPING (in progress)	KNIPPING (in progress)
Heavy minerals	HAGEDORN (2004), HAGEDORN & BOENIGK (2008)	HOSELMANN (in progress)	-
Palaeomagnetic investigations/ Susceptibility/ rock magnetic investigations	ROLF et al. (2008)	ROLF & HAMBACH (in progress)	-
Clay mineralogy	-	ELSASS et al. (in progress)	-
Molluscs	WEDEL (2008)	WEDEL (2008)	WEDEL (2008)

terrace (KÄRCHER 1987). Accordingly, the interpretation of the upper aquitard (Oberer Zwischenhorizont "OZH") as an Eemian formation seems doubtful, which is confirmed by the research of ENGESSER & MÜNZING (1991), RÄHLE (2005) and WEDEL (2008). Pollen analytical research by KNIPPING (2004, 2008) in the Mannheim area and on drillings at Schifferstadt (Fig. 1) and P34 indicates a Middle Pleistocene age (most probably Cromerian) for the OZH. Such positioning is further supported by palaeomagnetism measurements (ROLF et al. 2008), that indicate first reversal magnetism (780 ka, Matuyama-Brunhes boundary) to be positioned below the upper aquitard. The base of the Quaternary (Gauss-Matuyama boundary) at 177 m below surface in P34 is clearly deeper situated than in Schifferstadt and Speyer TB2 (Fig.1). This prompted HAGEDORN (2004) to assume that the northeastern part of the central Graben block is being downthrown by active tectonism.

The rock magnetic investigations (ROLF et al. 2008) together with results of heavy mineral analyses (HAGEDORN & BOENIGK 2008) show a clearly structured sediment profile in P34. It was possible to identify the change from mainly locally controlled sedimentation of the graben margins to a more distinct alpine controlled sedimentation at a depth of 177 m (G/M-boundary) by magnetic data. Based on lithostratigraphic correlation with other sedimentary records from the URG and also based on palynological evidence, this event happened at the end of Late Pliocene during a time of normal polarity of the Earth's magnetic field (Gauss Chron?). The well-documented characteristic change in magneto-mineralogy from goethite to greigite almost at the same stratigraphic level, was interpreted solely as a climatic signal (ROLF et al. 2008) which can be correlated with the global climate change at ~2.5 Ma that is well documented in deep-sea sediments (SHACKLETON et al. 1984), loess deposits (HELLER & LIU 1982) and fluvio-lacustrine sediments (HAN et al. 1997).

The heavy mineral spectrum in the Pliocene sediments confirms that the only erosional ma-

terial entering the Upper Rhine Graben came from the graben margins. Whether or not it is possible to subdivide the Pliocene sequences further depends on the results of additional investigations. In the uppermost Pliocene, the connection of the Palaeo-Rhine to the Alpine drainage system changed the heavy mineral spectrum of the Rhine sediments and led to a dominance of Alpine minerals (HAGEDORN & BOENIGK 2008). The boreholes drilled in the Ludwigshafen area revealed changes in the position of the river bed. Whilst the Rhine deposited sediments on the western margin of the northern graben during the Pliocene and at the beginning of the Quaternary, the river bed moved to further east during the subsequent period because the western margin is primarily dominated by sediments deriving from the Pfälzerwald. The situation further to the east is different because the sediments are dominated here by the Alpine heavy mineral spectrum. Because of the rapid subsidence of the eastern margin of the graben (Heidelberg Basin) it is conceivable that the bed of the river Rhine was located in this area of tectonic subsidence (ELLWANGER et al. 2005). Renewed sedimentation by the Rhine in the western part of the northern Upper Rhine Graben did not start again before the deposition of the Upper Gravels during the Weichsellian glaciations, as seen on the Frankenthal Terrace (WEIDENFELLER & KÄRCHER 2008).

2 Results

The Upper Gravels (OKL) consist of arenaceous medium gravel to coarse gravel of up to 20 m in both cores. The OKL can be divided into two sections in both boreholes. The upper section consists of coarse gravel, extends in each case to a depth of 13 m, and is characterised by fining upwards from 10 m to 1 m depth. This is underlain by interbedded coarse sands and fine to medium gravels down to a depth of 19.5 m. A coarse-grained facies is only present in the Upper Gravels. The underlying sediments are distinctly finer grained (sandy-gravelly in the middle aquifer, and sandy-silty

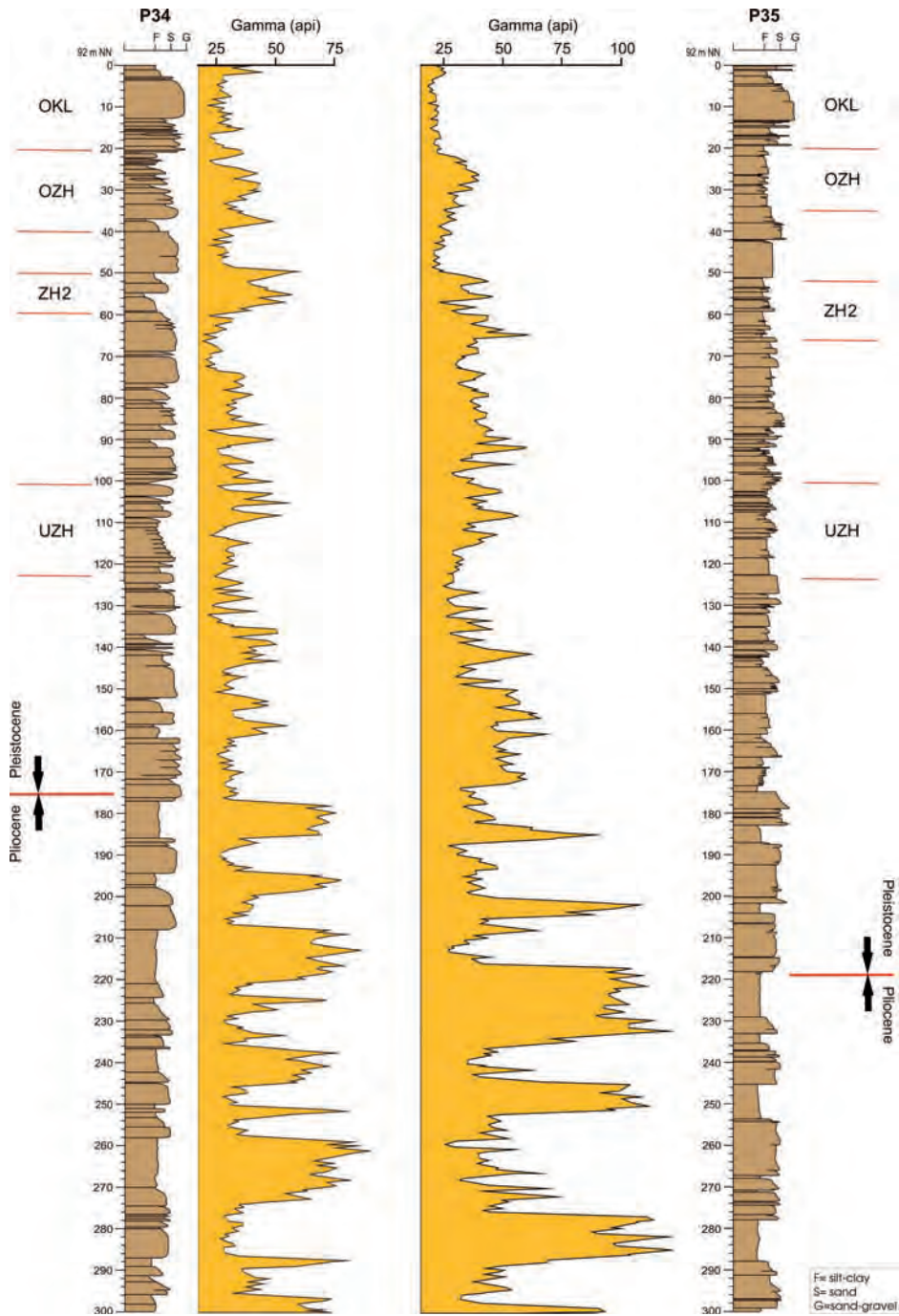


Fig. 2: Correlation of the lithofacies sections and Gamma Ray logs in the Ludwigshafen P34 and P35 bore-holes.

Abb. 2: Gegenüberstellung der Lithofaziesprofile und Gamma-Logs der Bohrungen Ludwigshafen P34 und P35.

in the lower aquifer). The OKL is underlain by the Upper Intermediate Horizon (OZH) which is widely distributed in the northern Upper Rhine Graben, in the same way as the OKL. The OZH is located in borehole P34 between 21 m to 40 m, and in borehole P35 between 19.5 m to approximately 34 m depth (Fig. 2). The interbeds of the aquitard are clayey-silty and frequently contain organic intercalations of peat or turfy moulder.

The OZH in both boreholes is underlain to a depth of 101 m by a medium to coarse sandy sequence containing only subordinate and thin horizons of fine gravel. A silty-sandy intermediate horizon with interbedded organic horizons (ZH2) occurs in borehole P34 between 50 m and 60 m depth, and between 51 m and 66 m depth in borehole P35. This is followed by a medium to coarse sandy section which reaches a depth of approximately 87 m in both boreholes, underlain in P34 by fine-grained sediments with organic horizons down to a depth of about 92 m; and down to a depth of 98 m in P35. A distinct intermediate horizon, termed the Unterer Zwischenhorizont (Lower Intermediate Horizon) (UZH), occurs at a depth of 101 m to 122 m in both boreholes, and also contains several organic horizons. The sequence below 122 m is dominated by interbedded fine to medium-grained sand with intercalations of clayey-silty horizons. Borehole P35 has slightly thicker fine-grained sections between 151 m to 163 m, 167 m to 175 m, 183 m to 187 m and from 201,5 m to 204 m. According to the current status of the analysis, the fine-grained sections between 122 m to 183 m in borehole P35, are much thinner or completely absent in borehole P34. Coarser horizons with fine to medium gravel first occur between 162 m to 167 m in P34 and between 175 m to 183 m in P35.

There is a good correlation between the sediments in both boreholes from 0 m to 122 m depth – as also highlighted by a comparison of the Gamma Ray logs run in both boreholes (Fig. 2). This changes between 122 m to 183 m where the boreholes have different lithofacies profiles and Gamma Ray logs. The distinct

change of the facies at 177 m depth in borehole P34, which is also interpreted as the Plio-Pleistocene boundary (KNIPPING 2008, ROLF et al. 2008), does not occur in borehole P35 until a depth of 183 m. The more than 10 m-thick fine-grained sequence lying beneath the Plio-Pleistocene boundary in P34 is absent at this depth in borehole P35, where it does not occur until a depth of 218 m to 232 m. The Gamma Ray logs at the same depths in both boreholes differ to a greater or lesser extent.

First analyses of the carbonate content and heavy minerals from isolated samples in borehole P35 (pers. com. C. Hoselmann, Hessisches Landesamt für Umwelt und Geologie) resulted in carbonate concentrations of 10 to 20 % down to a depth of 190 m and carbonate contents of up to 3 % between 190 m to 218 m (Fig. 3). The samples are non-calcareous beneath 218 m. The spectrum of transparent heavy minerals also reveals a difference in composition below a depth of 218 m. Garnet, epidote, alterite and hornblende dominate from 0 m to 218 m. Stable heavy minerals such as zircon and tourmaline are only present in subordinate amounts. The spectra are typical for sediments deposited in the Pleistocene Rhine. The sequences are interrupted – as also confirmed in the P34 borehole (HAGEDORN 2004, HAGEDORN & BOENIGK 2008) – by sediments deposited by streams draining the Buntsandstein of the Pfälzerwald. These are characterised by heavy minerals dominated by tourmaline and higher concentrations of zircon. Below 218 m, tourmaline and zircon are joined by significant proportions of the TiO_2 group, as well as hornblende and garnet. Similar spectra occur in borehole P34 below the Plio-Pleistocene boundary. This characteristic later Pliocene heavy mineral spectrum is also shown in other boreholes drilled in the Northern Upper Rhine Graben, such as the Schifferstadt borehole (HAGEDORN 2004) and in Viernheim (HOSELMANN 2008).

Nearly 1200 samples were taken from the P34 cores for pollen analysis. 120 samples were initially selected for a preliminary analysis and prepared at the laboratory operated by LGB Rheinland-Pfalz. This preliminary analysis

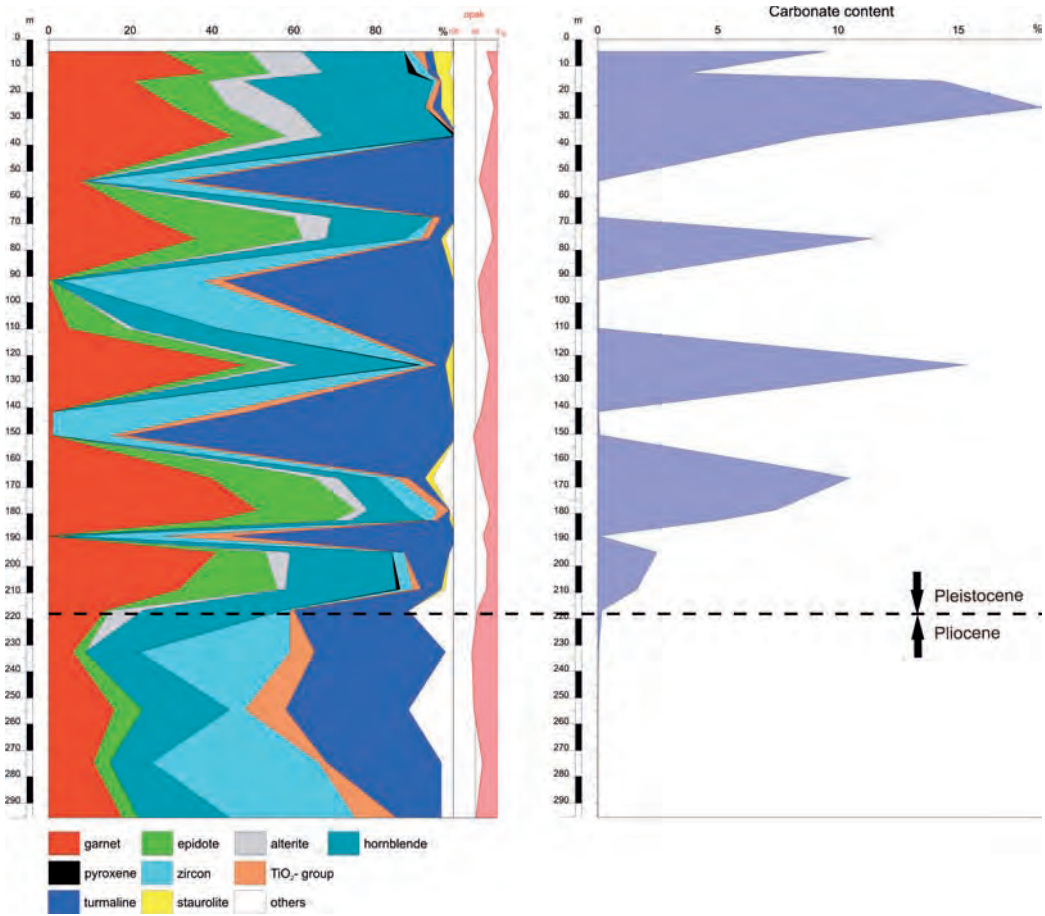


Fig. 3: Heavy mineral diagram and carbonate content of the Ludwigshafen P35 borehole (HOSELMANN, pers. com.).

Abb. 3: Schwerminerale und Karbonatgehalt der Bohrung Ludwigshafen P35 (HOSELMANN, pers. Mitt.).

involved a quick estimation of the pollen content, or counting only a minor number of pollen grains. 1600 pollen samples were taken from the P35 borehole, and 60 samples were looked at. 38 samples from the Maudach A 36 borehole also underwent preliminary analysis to roughly determine the pollen content, or subjected to counting a small number of pollen grains (Fig. 4, Fig. 5). The pollen sequences are mostly incomplete due to the variable tectonic subsidence of the URG and the fluvial dynamics which didn't allow a regular sedimentation. The preliminary investigation of borehole P34 revealed at least parts of 5 interglacials and se-

veral glacial pollen sequences (Fig. 4, Fig. 5). At a depth of 13,6 m in the lower, fine-grained section of the OKL, the pollen assemblage (Lu-I-A in Fig. 5) had high values of *Corylus* (hazel) as well as *Picea* (spruce) and *Alnus* (alder), and low values of *Quercus* (oak), *Ulmus* (elm), *Carpinus* (hornbeam) and *Abies* (fir), which may indicate the early part of an interglacial sequence or a part of a temperate interstadial. Another interglacial sequence was encountered in the OZH from 26.5 – 29.05 m. The lower part of this section (Lu-I-C) contains pollen from *Abies*, *Carpinus* and *Taxus* (yew), and muscelsae from *Azolla filiculoides* (large mosquito

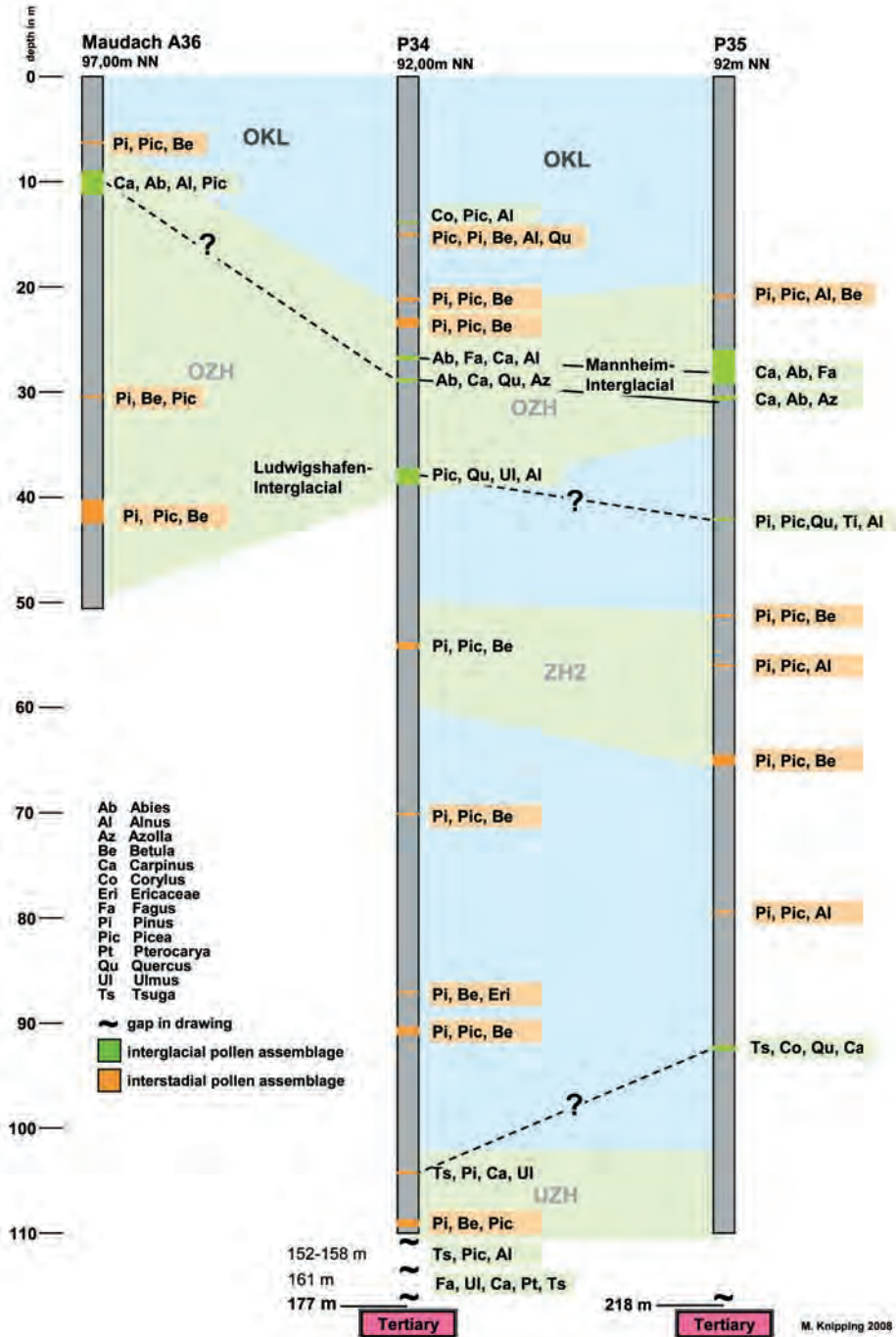


Fig. 4: Summary of the preliminary pollen analysis results from boreholes P34, P35 and Maudach A36.

Abb. 4: Übersicht der pollenanalytischen Voruntersuchungen der Bohrungen P34, P35 und Maudach A36.

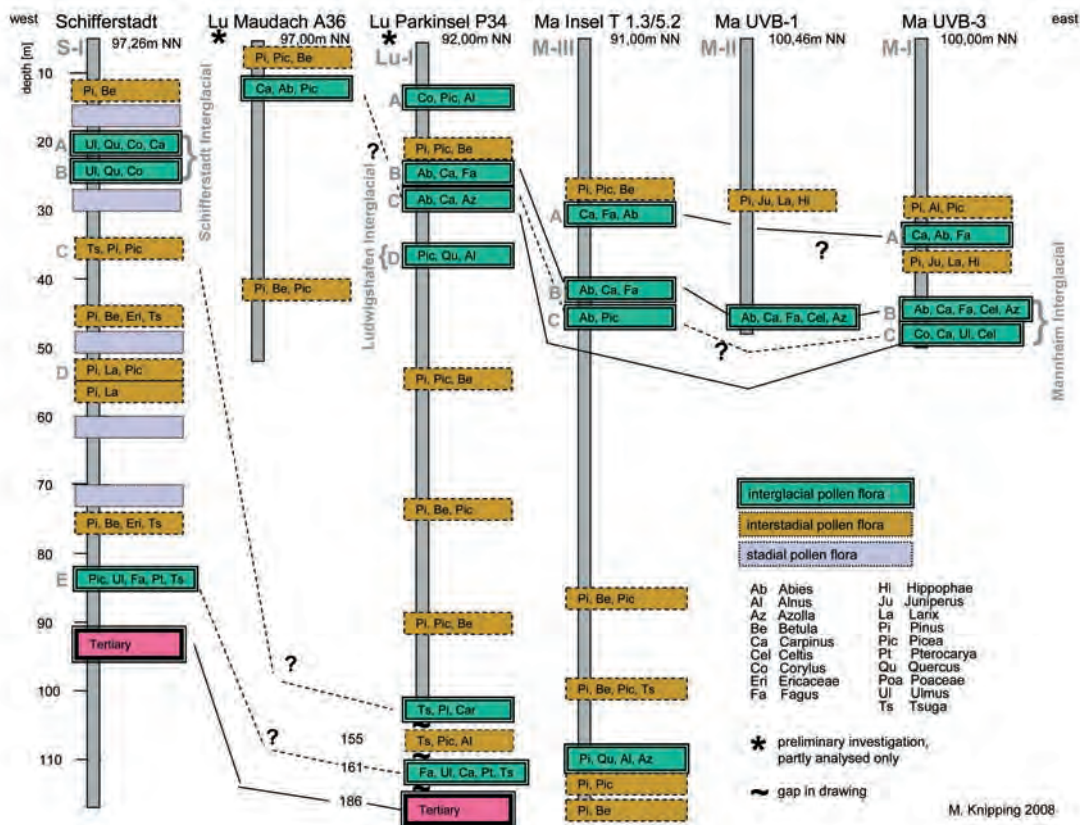


Fig. 5: Summary and preliminary correlation of the investigated pollen sections in the Schifferstadt/Ludwigshafen/Mannheim area.

Abb. 5: Übersicht und vorläufige Korrelation der untersuchten Pollenprofile im Raum Schifferstadt/Ludwigshafen/Mannheim.

fern), whilst the upper part (Lu-I-B) contains pollen from *Fagus* (beech). A further interglacial pollen assemblage (Lu-I-D, Ludwigshafen Interglacial) containing *Picea*, *Quercus*, *Alnus*, *Ulmus* and *Corylus* was present between 36.9 – 38.4 m.

Two samples from the UZH (103 m) contain several grains of *Tsuga* (hemlock) in a pollen assemblage dominated by *Pinus* (pine), together with *Picea*, *Carpinus* and *Ulmus*. Other spectra with *Tsuga*, *Picea*, *Alnus* and *Pinus* occur at a depth of 152.4 – 158.4 m. High concentrations of *Fagus* pollen alongside *Quercus*, *Ulmus*, *Carpinus*, *Pterocarya* (wingnut), *Ostrya* type (hop hornbeam) and *Tsuga*, were recovered at a depth of 161.45 m. A Tertiary

pollen spectrum was identified in two separate samples at 186 and 201 m.

In the first analysis of borehole P35, which involved the zone down to a depth of 93 m, there were at least three interglacial sequences alongside several glacial pollen spectra (Fig. 4). An analogous interglacial sequence was found at a similar depth to that in borehole P34. In the lower part of this sequence (30.4 – 30.7 m) pollen from *Abies*, *Carpinus* and *Quercus* were identified, along with massulae from *Azolla*. In the upper part (26.2 – 29.0 m), *Fagus* pollen was also found. The decline in pollen from thermophilous taxa (from 26.5 m) and the marked increase in *Pinus* pollen appears to show the end of the interglacial. A pollen

spectrum with *Pinus*, *Picea*, *Corylus*, *Quercus* and *Alnus* is also found at 42.1 m. Two interglacial pollen spectra including *Tsuga*, as well as *Corylus*, *Quercus* and *Carpinus* were identified in fine-grained horizons (91.1 – 92.45 m) below the UZH.

In the OZH at Maudach A36 a pollen sequence (9.0 – 11.2 m) shows interglacial conditions characterised by high amounts of *Carpinus* (up to 40 %) shifting to woodland richer in *Abies*, then in *Picea* and dominated at last by *Pinus* and *Betula*. Until now *Fagus* is absent, even though several samples have been analysed. Pollen of *Buxus* has values up to 5 % in the lower part of the interglacial.

3 Discussion

It should be stressed in advance that the following correlations are only tentative in character because they are based on preliminary analysis, and complete analysis of the samples still remains to be done. The pollen sequences recorded to date are often fragmentary and there are uncertainties when correlating these with other pollen sequences. Publications covering the boreholes from the Mannheim area (KNIPPING 2004, 2008) already include some of the results of the preliminary analysis of the P34 borehole, which provided an invaluable addition to and confirmation of the biostratigraphic classification of the penetrated sequences.

For the interglacial or temperate interstadial pollen spectrum at a depth of 13.6 m in the OKL in borehole P34, a Holocene or Eemian age can be rejected, but a correlation with either one of the Early Weichselian interstadials or an interglacial older than the Eemian is possible (KNIPPING 2008). No analogous pollen spectrum has so far been found in P35.

P34 contains pollen spectra (26.5 – 29.05 m) which are correlatable with a high degree of probability with the Middle Pleistocene Mannheim Interglacial (KNIPPING 2008). Borehole P35 (Fig. 4) also revealed pollen spectra at a similar depth which, because of the presence of *Carpinus*, *Abies* and *Azolla* in the lower part (30.4 - 30.7 m), and the additional occurrence

of *Fagus* in the upper part of the interglacial (26.2 - 29.0 m), correlates with a high degree of probability with the interglacial in P34 (Lu-I-B + C) and therefore with the Mannheim Interglacial (Fig. 4, Fig. 5). According to the pollen spectra identified to date, the upper part of the interglacial is represented in P35 by a section with a thickness of 2.8 m, and is thus much thicker than in P34 where the thickness is only 0.3 m. The lower part of the interglacial has a similar thickness in both boreholes with a thickness of approx. 0.3 m.

Comparison with other Middle Pleistocene interglacials indicates that the most likely correlation for the Mannheim Interglacial is with the Rhume Interglacial (MÜLLER 1986, 1992) and the Kärlich Interglacial (URBAN 1983, BITTMANN 1992). The interglacial section of Meikirch II (WELTEN 1982, 1988) was recently reinterpreted by PREUSSER et al. (2004), and the former "Eemian" is now correlated with MIS 7. There are some similarities with the Mannheim Interglacial, but *Celtis* which is present in the Mannheim Interglacial as well as the Kärlich Interglacial is not present at Meikirch.

The interglacial found in the OZH in P34 at 36.9 – 38.4 m (Lu-I-D) is primarily characterised according to the current degree of analysis by the absence of *Carpinus*, *Abies* and *Fagus*. There are some similarities to the pre-Rhume thermomere described by MÜLLER (1992) and the interglacial found by BLUDAU (2001) at approx. 47 – 52 m in the Mannheim Ergo BK 3 borehole. Interglacial pollen spectra without *Carpinus*, *Abies* and *Fagus* were only found in two horizons at 42 m in borehole P35. It is not certain at this stage whether these correlate with Lu-I-D. Should this correlation be confirmed, then there is a very marked difference in the thickness of the sequence in P34 (1.5 m) and the 0.1 m thick sequence in P35.

Another cored borehole in the Ludwigshafen area (Maudach A36) was also included in this preliminary analysis (Fig. 1, Table 1). An interglacial with high proportions of *Carpinus* is reported in the OZH at a depth of 9 – 11.2 m. Based on the current status of the preliminary analysis, this cannot be correlated with the

younger part of the Mannheim Interglacial because no *Fagus* has been found. It is possible that this interglacial correlates with the older part of the Mannheim Interglacial. If this is true, the Mannheim Interglacial represents two separate interglacials because the end of the interglacial appears to have been reached in Maudach A36 with the transition to *Pinus*-richer sections. The presence of a gastropod fauna in the same borehole at a depth of 23 – 28 m, which WEDEL (2008) interprets as indicating the Cromerian Complex, could indicate a possible classification as Cromerian. Another consequence of a correlation between Maudach with the older part of the Mannheim Interglacial would be to make the correlation between the Mannheim Interglacial with the earlier “Eemian” at Meikirch (PREUSSER et al. 2004) very unlikely (see above), because *Carpinus* pollen is very rare in the two lower interglacials of Meikirch “Holstein 1” and “Holstein 2” (WELTEN 1982, 1988), but accounts for nearly 40 % in Maudach. The Upper Pleistocene position of the interglacial at Maudach at a depth of 9 – 11.2 m cannot, however, be excluded on the basis of the current status of the pollen analysis.

Interdisciplinary analysis in the northern part of the Upper Rhine Graben, which in addition to looking at the lithostratigraphy, also looks at “Upper Pleistocene” mammal remains, gastropods, wood remains and pollen, provided no reliable stratigraphic classification of the material (KOENIGSWALD 1988). Dating of parts of the Upper Gravels (OKL) and the “Oberer Ton” (Upper Clay = OZH) to the last interglacial (Eemian) has, however, been favoured by some authors. However, the latest analysis by KNIPPING (2002, 2004, 2008) and WEDEL (2008) has shown that the OZH in the Mannheim/Ludwigshafen area cannot be assigned to the Eemian but must be older. The analysis of gastropods from the “Upper Clay” in the Mannheim-Lindenhof borehole (RÄHLE 2005) gave a Middle Pleistocene, probably Cromerian age. The same samples contained analogous pollen spectra to those found in the Mannheim Interglacial (KNIPPING, unpub-

lished). This, as well as the work of ENGESSER & MÜNZING (1991) in the Mannheim area support the palynological correlation of the OZH with the Cromerian Complex. Two boreholes of Mannheim (KNIPPING 2004, 2008) contained additional pollen from thermophilous taxa above the Mannheim Interglacial, including *Carpinus* and *Fagus* (Fig. 5). Because these are only single samples, the question still remains open of whether these can be correlated with interglacials younger than the Mannheim Interglacial. It does, however, support the correlation of the Mannheim Interglacial with the Cromerian Complex.

No Middle Pleistocene pollen spectra with *Pterocarya*, which could be correlated with the Holstein/*Pterocarya* Interglacial (MÜLLER 1974, GRÜGER 1983, DRESCHER-SCHNEIDER 2000) have so far been identified in the boreholes in Mannheim, Schifferstadt or Ludwigshafen. The Schifferstadt Interglacial described in the Schifferstadt borehole (KNIPPING 2002), is also probably equivalent to the Ferdynandowian Interglacial (JANCZYK-KOPIKOWA 1975), and has also not been identified so far in the two boreholes in Ludwigshafen.

Increased subsidence of sub-blocks during the Plio-Pleistocene in the northern Upper Rhine Graben around the Mannheim/Heidelberg area is not only indicated lithostratigraphically, but also by pollen analysis. According to the initial investigation, the P34 and P35 boreholes penetrated sediments down to a depth of 102 m in which at least 3 superposed interglacials have been partially identified (KNIPPING 2004, 2008). *Tsuga* occurs from approximately 103 m in P34 and 92 m in P35, which currently still indicates a Lower Pleistocene age. Notable in this context is the single identification of a *Tsuga* pollen grain in the lower part of the Mannheim Interglacial (UVB-3) (Fig. 5) (KNIPPING 2008). Indications that *Tsuga* could also be present in the Cromerian Complex are supported by the analysis conducted by BLUDAU (2001) in the Mannheim Ergo BK3 borehole. The author describes several isolated occurrences of *Tsuga* in an interglacial and discusses a possible Cromerian age. In addition, in the Schifferstadt

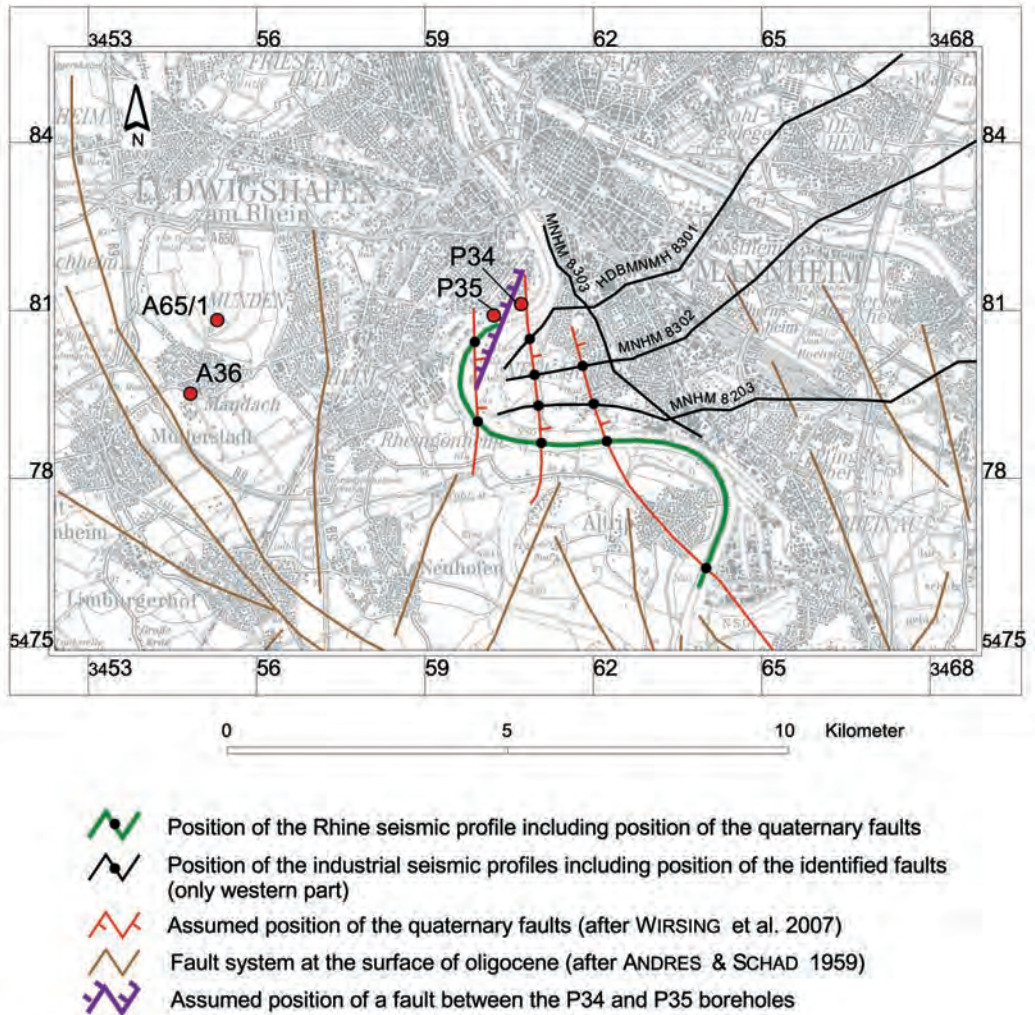


Fig. 6: Assumed position of a fault between the Ludwigshafen P34 and P35 boreholes, depicting the position of the Quaternary faults, interpreted on the basis of industrial seismic and river seismic profiles (after WIRSING et al. 2007).

Abb. 6: Vermuteter Verlauf der Störung zwischen den Bohrungen Ludwigshafen P34 und P35 mit Darstellung des Verlaufs quartärer Störungen, entwickelt aus industrieseismischen und flusseismischen Profilen (nach WIRSING et al. 2007).

borehole lying further to the west (KNIPPING 2002), which is also described as containing a Middle Pleistocene interglacial (Schifferstadt Interglacial), several occurrences of *Tsuga* have been confirmed at depths below 36 m. This section has therefore been preliminarily correlated with the Lower Pleistocene down to the Plio-Pleistocene boundary at approximately

91 m. The Middle-Lower Pleistocene boundary and Quaternary base deepen in these boreholes from west to east in the direction of the centre of the Heidelberg Basin. The Quaternary base in the Viernheim research borehole is at a depth of 224 m (HOSELMANN 2008).

Lower Pleistocene and Tertiary sediments from the northern Upper Rhine Graben have mainly

been looked at in the Schifferstadt borehole. Various interglacial and glacial vegetation sequences were described here in the older sections (KNIPPING 2002). Lower Pleistocene sediments have so far only been recorded in the P34 borehole. The occurrence of *Tsuga* pollen, together with *Picea*, *Alnus* and *Pinus* at 152.4 – 158.4 m, points more to cool climatic conditions, but no detailed biostratigraphic subdivision is currently possible. Pollen spectra with high proportions of *Fagus*, in addition to *Tsuga*, *Pterocarya*, *Carpinus*, *Quercus* and *Picea*, occur at 160.45 – 161.45 m. Because of the common occurrence of *Fagus*, it can probably be classified as lying within the Tegelen Complex, probably Tegelen A (ZAGWIJN 1963). Tertiary pollen spectra at 186 and 201 m in P34, combined with heavy mineralogical and palaeomagnetic and rock-magnetic analysis, confirm the Plio-Pleistocene boundary at 177 m. No pollen analysis has yet been carried out on the Lower Pleistocene and Pliocene sections in borehole P35.

The new results of the analyses of borehole P35 (heavy minerals, carbonate content, lithofacies profile, gamma ray log) indicate that the Plio-Pleistocene boundary in borehole P35 is not at 183 m depth as implied by the first interpretation, but probably more likely at a depth of 218 m. Because the base of the Quaternary in the P34 borehole was confirmed at a depth of 177 m (KNIPPING 2008, ROLF et al. 2008), the Plio-Pleistocene boundary in borehole P35 probably lies approximately 42 m deeper. The large difference in depth within a distance of only 500 m cannot be explained by fluvial dynamics alone. It is more likely that the two boreholes are separated by a fault, and that borehole P34 lies on the upthrown and P35 on the downthrown side of the fault. The river seismic survey shot on the Rhine, and the interpretation of industrial seismic sections, indicate the presence of young faults in the Ludwigshafen/Mannheim area (BERTRAND et al. 2006, HAIMBERGER et al. 2005, WIRSING et al. 2007). These easterly dipping faults indicate the en-echelon faulted subsidence of the Quaternary base from west to east in the direction of the centre of the

Heidelberg Basin (Fig. 6). Throws of up to 10 m at the base of seismic horizon 2 (OZH and Middle Sandy-Gravelly Series) are interpreted from the seismic sections. In general, the faults identified in the Ludwigshafen/Altrip area can be linked to three NNW-SSE running, easterly dipping antithetic downthrown faults (WIRSING et al. 2007). Because the drilling location of borehole P34 lies approximately 500 m ENE of borehole P35, it must be assumed that a sub-block lying further to the west has subsided more strongly (P35), an anomaly when compared to the regional situation. It is possible that a previously unidentified WNW-ESE running transverse fault separates this sub-block from the easterly lying sub-block on which borehole P35 was drilled. Indications of young tectonic movements in the northern Upper Rhine Graben are documented in numerous recent publications (PETERS et al. 2005, PETERS & VAN BALEN 2007a, 2007b, WEIDENFELLER & KÄRCHER 2008). Interestingly, the Quaternary faults are very closely related to faults which cut the Tertiary sediments and the base of the Tertiary (ANDRES & SCHAD 1959, DERER et al. 2005).

Because the upper 122 m of the P34 and P35 boreholes can be very well correlated in terms of thickness, as well as sedimentary facies and gamma logs, it is assumed that tectonism was particularly active prior to the start of sedimentation of the Lower Intermediate Horizon (UZH), and that it then weakened considerably. Because the OZH is assigned to the Cromerian Complex on the basis of current findings (KNIPPING 2008, RÄHLE 2005, WEDEL 2008), and because the UZH is also assigned to the transition zone between the Lower and Middle Pleistocene, the phase of tectonism giving rise to the approximately 42 m throw is considered to have taken place during the latest Pliocene and Lower Pleistocene. It seems clear that this period was characterised by more rapid subsidence of the Heidelberg Basin, and thus the Ludwigshafen-Mannheim area as well, and that this rapid subsidence was compensated for by high rates of sedimentation.

4 Conclusions

Fluctuating fluvial dynamics over very short distances leave behind strongly differentiated sequences in the geological record which can only be approximately interpreted with the information gained from several cores. The differences in the nature of the sediments between the P34 and P35 boreholes highlights that the interpretation of the climatic and fluvial history of an area on the basis of the analysis of a few boreholes becomes very difficult because the sediments are not only affected by fluvial dynamics, but also by small-scale tectonics. The complex fault pattern in the northern Upper Rhine Graben is partially responsible for the preservation and character of the sediments. Unlike major cycles which can still be recognised and correlated across several boreholes, closer analysis reveals the presence of frequent gaps which make interpretation more difficult. Sedimentation is often controlled by young tectonics where small-scale fault block movements can result in the deposition of very different sequences of sediments, as recorded in the cores. The clear characterisation and stratigraphic identification based on several independent methods, and on pollen analysis in particular, provides the information required to make reliable interpretations of the sedimentation history influenced by palaeoclimatic fluctuations and tectonic movements. The differences identified by comparing the two boreholes P34 and P35, which are only 500 m apart, emphasise that the large-scale correlation of boreholes separated from one another by several tens of kilometres, will only succeed when the sediments in the cores are analysed using analogous methods and can be stratigraphically classified.

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The Pliocene and Pleistocene fluvial evolution in the northern Upper Rhine Graben based on results of the research borehole at Viernheim (Hessen, Germany)

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Abstract: The research borehole drilled in 2006 by the Hessian Agency for the Environment and Geology (HLUG) north of Viernheim (Hessisches Ried) reached a total depth of 350 m, and penetrated high resolution fluvial and limnic-fluvial sediments (0 to 225 m) of Pleistocene age, and partially highly pedogenically overprinted limnic-fluvial sands, clays and silts of Pliocene age (225 to 350 m). The Pliocene sediments tend to be sourced locally. The sediments repeatedly show sourcing from the Odenwald which is characterised by a high percentage of green hornblende in the heavy mineral fraction. As part of the Heidelberg Basin research programme, one of the main purposes of this borehole was to analyse the Pleistocene “Normal Facies” of the northern Upper Rhine Graben, i.e. a sedimentary sequence subject to minimum disturbance, largely unaffected during the Pleistocene by material sourced from the graben margins or smaller tributaries.

The Pleistocene sedimentary sequence consists of three units: a thin horizon with reworked Pliocene material is overlain by ten cycles each beginning erosively with gravely sandy sediments and ending with silty-argillaceous to in part peat-like sediments. Internal cycles can also be identified, amongst other features. A characteristic aspect is the green-grey, strongly calcareous, micaceous and well sorted, fine to medium sands of the Rhine. These are dominated by the Rhine Group (garnet, epidote, green hornblende and alterite) in the heavy mineral fraction. These sediments are classified as the “Rhenish Facies”. The upper Pleistocene sedimentary sequences at the top of the Viernheim research borehole are dominated by several fining-upward and in part coarsening-upward sequences. The deposits in this part of the well are dominated by gravel deposited by the Neckar. The heavy mineral distribution of the sand fraction reveals, however, that there was mixing with Rhenish sediments.

Weichselian to Holocene aeolian sands form the topmost part of the well section. The stratigraphic classification of the Pleistocene sedimentary sequences is still uncertain in parts. The Pliocene-Pleistocene boundary is placed at 225 m because of the characteristic change in facies. Due to lithostratigraphic correlations with sediments within the Lower Rhine Embayment, a larger unconformity at the depth of 225 m must be accepted. Research carried out in the area around the well indicates that the youngest fine-clastic section penetrated by the well between 39.76 and 58.55 m is of Cromerian age.

[Die pliozäne und pleistozäne fluviale Entwicklung im nördlichen Oberrheingraben unter besonderer Berücksichtigung der Forschungsbohrung Viernheim (Hessen, Deutschland)]

Kurzfassung: Die 2006 durch das Hessische Landesamt für Umwelt und Geologie (HLUG) abgeteufte Forschungsbohrung nördlich von Viernheim (Hessisches Ried) hat mit einer Endteufe von 350 m hoch aufgelöst fluviale und limnisch-fluviale Sedimente (0 bis 225 m) des Pleistozäns und zum Teil stark pedogen überprägte limnisch-fluviale Sande, Tone und Schluffe des Pliozäns (225 bis 350 m) durchteuft. Die Liefergebiete der pliozänen Sedimente sind eher regional geprägt. Die Sedimente zeigen wiederholt Schüttungen aus dem Odenwald, die durch einen hohen Anteil grüner Hornblende in der Schwermineralfraktion gekennzeichnet sind. Als Teil des Forschungsprogramms „Heidelberger Becken“ zielte diese Bohrung insbesondere

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im Pleistozän auf die „Normalfazies“ des nördlichen Oberrheingrabens ab, das heißt: auf eine möglichst ungestörte Sedimentabfolge, die im Pleistozän Schüttungen von den Grabenrändern oder kleineren Zuflüssen weitestgehend ausschließt.

Die pleistozäne Sedimentabfolge besteht aus drei Einheiten: über einem geringmächtigen Horizont mit aufgearbeitetem pliozänen Material folgen zehn Zyklen, die erosiv mit kiesig sandigen Sedimenten einsetzen und mit schluffig-tonigen bis zum Teil torfigen Ablagerungen abschließen. Mitunter sind interne Zyklen zu erkennen. Charakteristisch sind grünlich-graue stark carbonatische, glimmerführende und gut sortierte Fein- bis Mittelsande des Rheins. In diesen dominiert die Rhein-Gruppe (Granat, Epidot, grüne Hornblende und Alterit) in der Schwermineralfraktion. Diese Sedimente werden als „Rheinische Fazies“ bezeichnet.

In der hangenden letzten pleistozänen Sedimentabfolge der Forschungsbohrung Viernheim bestimmen mehrere fining-upward und zum Teil coarsening-upward Sequenzen das Sedimentationsgeschehen. Die Ablagerungen dieses Profilabschnitts sind Kies dominiert, der vom Neckar geschüttet worden ist. Die Schwermineralverteilung der Sandfraktion zeigt aber an, dass es zu einer Vermischung mit rheinischen Sedimenten gekommen ist. Weichsel- bis holozänzeitliche Flugsande schließen das Profil ab.

Die stratigraphische Einstufung der pleistozänen Sedimentabfolge ist in Teilen noch unsicher. Die Plio- bis Pleistozängrenze wird auf Grund des charakteristischen Fazieswechsels auf 225 m gelegt. Eine überregionale Korrelation mit Sedimenten der Niederrheinischen Bucht spricht für eine Diskordanz mit größerer zeitlicher Lücke an der Plio-Pleistozängrenze. Untersuchungen im Umfeld der Bohrung sprechen für cromerzeitliches Alter des jüngsten feinklastischen Abschnitts der Bohrung zwischen 39,76 und 58,55 m.

Keywords: Quaternary, Pleistocene, Pliocene, Cromerian Complex, fluvial sediments, heavy minerals, Carbonate, Upper Rhine Graben, Germany

1 Introduction

The north-eastern part of the Upper Rhine Graben (URG) is known as the “Hessisches Ried” (Fig. 1). This area has become an important research area for Quaternary graben development in recent years. According to VAN GIJSSEL (2006), the URG is a non-glaciated type region for mid Central European large and medium sized upland basins.

The historical landscape development in the late and post-glacial period in the northern URG was recently studied by BOS et al. (2008), DAMBECK (2005), DAMBECK & BOS (2002), DAMBECK & THIEMEYER (2002) as well as ERKENS et al. (2009). The geometry of the Quaternary sediment body was investigated using high resolution reflection seismic by HAIMBERGER et al. (2005) and WIRSING et al. (2007). The interaction between tectonics, and fluvial and erosive processes, particularly along the western graben margin, was investigated by PETERS & VAN BALEN (2007a, b) as well as PETERS et al. (2005). The sedimentological structure of the Pleistocene sedimentary fill in the northern Upper Rhine Graben has,

however, not been looked at systematically in recent times. This was one of the reasons why the Geological Survey of Hessen (Hessisches Landesamt für Umwelt und Geologie (HLUG)) drilled several research boreholes in the Hessisches Ried in recent years. In addition to scientific aspects concerning the sedimentology, sedimentary petrography, vegetation history, palaeontology and tectonic development during the Pleistocene and Upper Pliocene, the investigation was also interested in applied geological aspects, which require a thorough understanding of the structure of the graben fill for their clarification. The focus of this aspect of the investigation was on hydrogeology, resource geology and geothermy.

The boreholes were designed to reveal the geometry of the Pleistocene sedimentary body in more detail, and to specify each of the facies in the area of investigation. The thickness of Quaternary sedimentary fill increases from the western to the eastern graben margin. There is also an increase in thickness from north to south-east in the direction of the Heidelberg Basin (Fig. 2). At the eastern graben margin of the Hessisches Ried, the fluvial or limnic-

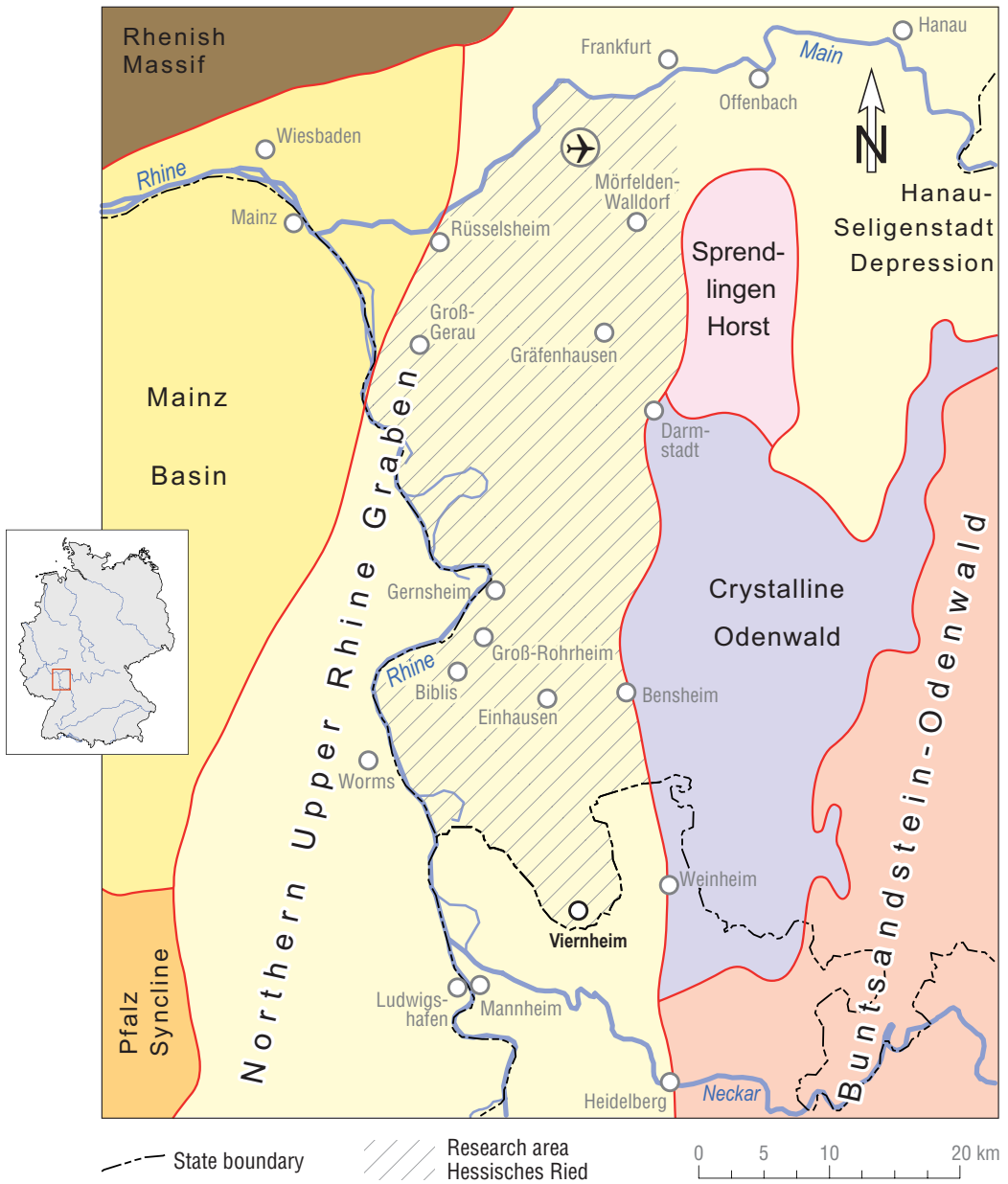


Fig. 1: Location map with the geological structural zones and the area of investigation “Hessisches Ried”.

Abb. 1: Übersichtskarte mit den geologischen Strukturräumen und dem Untersuchungsgebiet „Hessisches Ried“.

fluvial deposits repeatedly contain interbeds of redeposited sediments of hard rocks lying on higher ground further east, i.e., from the Odenwald or the Sprenzligen Horst. This hard rock, consisting of Palaeozoic crystalline rocks and sedimentary rocks of Rotliegend age (Sprenzligen Horst) and Buntsandstein, was intensively weathered during the Mesozoic and Tertiary. The resulting unconsolidated rock was easily eroded and redeposited during the Pleistocene periglacial periods. Directly along the graben margin in particular, there are also very high proportions of redeposited sediments derived from these source areas. The 111 m deep C/04 B1 borehole drilled south of Gräfenhausen north-west of Darmstadt (R 3472203 – H 5531111 based on the Gauss-Krüger coordinates of German topographic maps), clearly shows an almost 20 m thick sequence of redeposited sediments aligned east-west in this part of the northern URG, forming part of the Quaternary sedimentary sequence with a total thickness of 83.58 m. These redeposited sediments can be traced for approx. 5 km from the graben margin in the northern URG. The Pleistocene fluvial graben fill itself consists in the area of investigation of alternating fluvial sands and gravels mixed up in various proportions, as well as silts, clays and peat. This sequence is the “Normal Facies” of the northern URG.

In the north part of the Hessisches Ried, the Rhenish sediments interfinger with the fluvial sediments deposited by the Main river. Several hundred boreholes were geologically analysed and recorded by HLUK between 2003 and 2004 during the planning work for the Deutsche Bahn’s ICE high speed rail track from Frankfurt/Main airport (Rhine-Main district) to Mannheim (Rhine/Neckar zone). This revealed the presence of fluvial sediments, laid down by the Main all the way up to the area to the north of Gräfenhausen (borehole 2.3/41 drilled along the planned ICE track: R 3472332 – H 5533832). On the other hand, indisputable Rhenish sediments were also found as far north as the southern edge of Frankfurt airport. For instance, the borehole drilled close to the west

runway (BK 1200; R 3465891 – H 5542863) revealed Pleistocene, calcareous sediments of the Upper Rhine Graben beneath the Lower Pleistocene Untermain-Hauptterrassen Formation (HOSELMANN 2007a). A research borehole drilled to the north of Mörfelden (R 3468725 – H 5538979) also penetrated Main and Rhine sediments. Limnic-fluvial and fluvial sediments of Pliocene age were penetrated below the base of the Quaternary at 64.5 m below ground level. The “Walldorf Horst” postulated by ANDERLE & GOLWER (1980) with thin Quaternary sediments lying on unconsolidated Miocene sediment is therefore non-existent.

In the eastern part of the Hessisches Ried, the fluvial sediments of the northern URG are overlain by a few metres of Weichselian aeolian sands, partially drifted to form dunes. The thickness of the aeolian sands can even exceed 30 metres in places, for instance, at the western edge of the town of Darmstadt. This means that the aeolian sands may also have been redeposited and incorporated in the normal facies of the Pleistocene graben fill. Sedimentation in the western part of the Hessisches Ried ended with Holocene fluvial sediments laid down by various meander generations of the Rhine (DAMBECK 2005 amongst others).

The aim of the Viernheim research borehole was to derive a standard section for the “Normal Facies” of the graben fill in the northern URG, and to provide detailed descriptions of these sediments with regard to the sedimentology, sedimentary petrography, palaeobotany and palaeontology. It was also the intention to date these sediments by physical dating methods such as OSL, IR-RF, TAMS-²³⁰Th/U and Burial Age Dating (DEHNERT & SCHLÜCHTER 2008; PREUSSER et al. 2008; GEYH 2008) and to provide information on sedimentation rates where possible. The form of subsidence in this region means that large parts of the section can be expected to be preserved in superposition.

The work on the Viernheim research borehole is closely related to other research activities in the Heidelberg Basin (ELLWANGER et al. 2005). The first results of this work have already been published, particularly on the Ludwigshafen-

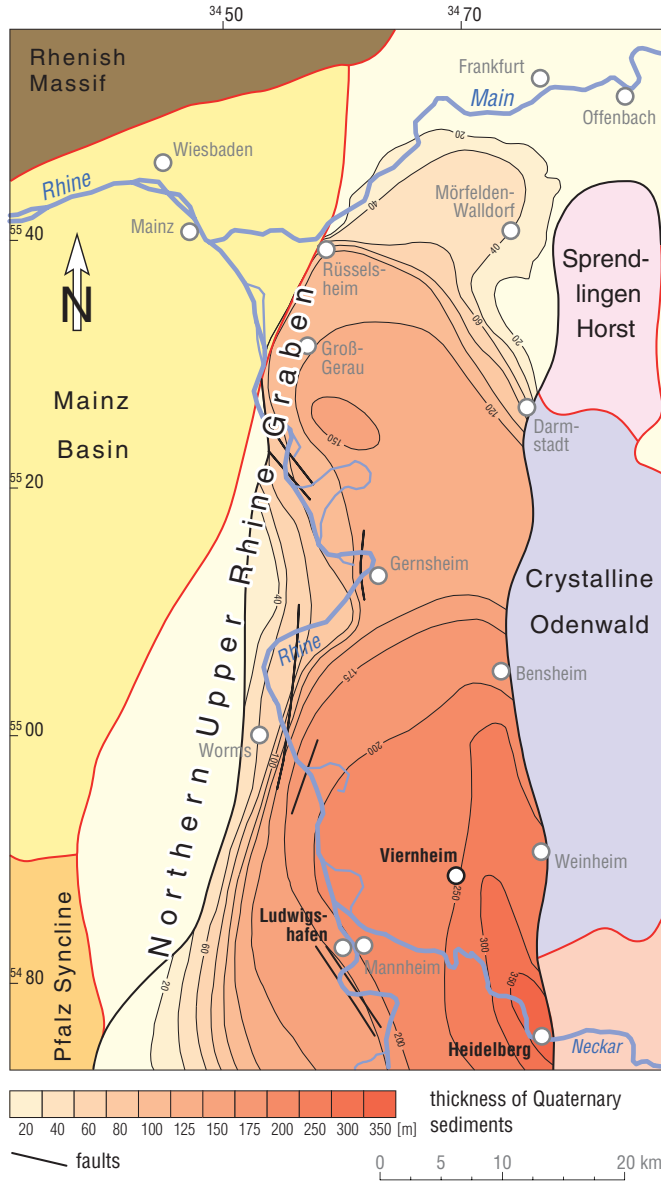


Fig. 2: Thicknesses of the Quaternary sediments in the northern URG; redrawn from BARTZ (1974), HAIMBERGER et al. (2005) as well as a great deal of new information from this work. The thickness of the Quaternary sediments increases from north to south as well as from west to east. The Heidelberg Basin begins in the Hessisches Ried south of Gernsheim. Around Heidelberg, a more strongly subsided subbasin revealing approx. 400 m of Quaternary sediments is documented.

Abb. 2: Mächtigkeitsverteilung quartärer Sedimente im nördlichen URG; umgezeichnet nach BARTZ (1974), HAIMBERGER et al. (2005) sowie mit verschiedenen Aktualisierungen aus dieser Arbeit. Generell ist eine Zunahme quartärer Sedimente von Norden nach Süden sowie Westen nach Osten zu erkennen. Das Heidelberger Becken beginnt im Hessisches Ried südlich von Gernsheim und bildet bei Heidelberg ein noch stärker abgesenktes Teilbecken mit einer maximalen Quartärmächtigkeit von rund 400 m.

Parkinsel borehole. Of particular interest here is the work on sedimentary petrography (HAGEDORN 2004; HAGEDORN & BOENIGK 2008), the fluvial development during the Middle and Upper Pleistocene (WEIDENFELLER & KÄRCHER 2008), palaeomagnetism (ROLF et al. 2008), palaeobotany (KNIPPING 2004, 2008; WEIDENFELLER & KNIPPING 2008) and tectonics (ELLWANGER et al. 2008).

2 Geological description of the Viernheim research borehole

2.1 Viernheim research borehole

The Viernheim research borehole lies almost 2 km to the north of Viernheim in the Hessisches State Forest, and specifically on the Buchnerschneise (R 3469080 – H 5492215; height of the borehole drilling location: 96.95 m asl; cf. point 5 in Fig. 10). The borehole was drilled by the drilling contractor Daldrup & Söhne from January to June 2006. Down to the total depth (TD) of 350 m, cores were cut in liners with an internal diameter of 10 cm. The sediments penetrated by the borehole were ram cored to a depth of 40.5 m. The wireline rotary coring method was then used to cut the next cores down to a depth of 64 m. The cored section from 64 m to 228 m was

again cut by ram coring. The wireline rotary coring method was again used from 228 m down to 350 m TD. Despite some technical problems during coring, the quality of the cores is very good with core recovery of approx. 97 %. The cores were sliced in halves: one half for scientific analysis and one half to be archived in the core store.

The borehole can be subdivided into two chronostratigraphic sections. From 350 m TD up to a depth of 225 m, the section largely consists of fine-clastic sediments assigned to the Pliocene on the basis of their lithological character, composition, pedogenic overprinting, and supra-regional correlation. The section from 225 – 221.21 m is a transition zone between Pliocene and the Pleistocene. The overlying sequence largely consists of Pleistocene fluvial sediments.

The discussion in the following dispenses with detailed core descriptions. This material can be requested from the author by e-mail, along with photo documentation and analysis data.

2.2 Subdivision of the sedimentological record

A strongly simplified description of the borehole section is shown in Figure 3. The Pliocene sequence can be subdivided as follows:

Unit	Depth in m below ground level	Description summary	Special aspects
10	225-228	Fine-clastic cycle with numerous calcareous clusters; slightly oxidised in the lower part, repeatedly strongly reduced at top	Without strong pedogenic overprint
9b	228-228,6	Strongly humic from here on, lignite in part	
9a	228.6-238.5	Fine-clastic sediments, similar to unit 7, dominantly reduced	Without strong pedogenic overprint
8	238.5-250.45	With fining-upward sequence starting with gravely sand (together with unit 9), repeated fine gravely horizons, silty in the upper part; reduced in part	With local sediment source probably from the Odenwald; section damaged during drilling

Unit	Depth in m below ground level	Description summary	Special aspects
7	250.45-261	Fine-clastic sediments, strongly spotted: reduced and oxidised, calcareous in clusters (similar to unit 1a, 3 and 5)	Pedogenic overprint = "Plinthosol"
6	261-270.44	Sand-dominated unit with a silt horizon at 268-268.85 m; largely reduced, also oxidised zones	Local sedimentary source, probably from the Odenwald
5	270.44-315.85	Compact unit similar to 1a, 3 and 7; silts and clays, with repeated discordant interbeds of sand, also calcareous in clusters, predominantly strongly oxidised and some reduced zones	Strong pedogenic overprint = "Plinthosol"; Unit discordantly overlain by 6
4	315.85-318.55	Sand deposits, upper part reduced, silty in part	Local sediment source, probably from the Odenwald
3	318.55-323.1	Similar to unit 1a, 3, 5 and 7, compact/homogenous	Only weak pedogenic overprint
2	323.1-330.62	Sand-dominated unit, layered in part, at 329-330 m with fine-gravelly clasts; humus in parts with redeposited lignite	Local sediment source, probably from the Odenwald
1b	330.62-333.4	Fine clastic, humus-banded, with sandy horizons, with a lot of fine sand towards the base, at the top similar to 1a	
1a	333.4-350	Fine-clastic sediments, strongly spotted: reduced and oxidised, calcareous in clusters	Strong pedogenic overprinting = "Plinthosol"

The Pliocene sediments are marked by a higher bedding density than the overlying sediments. The Pliocene sediments are followed discordantly by a short section which mainly consists

of reworked Pliocene sediments. This zone is given a preliminary chronostratigraphic classification as Plio-Pleistocene – this section consists of:

Unit	Depth in m below ground level	Description summary	Special features
11	221.21-225	Fining-upward sequence of fine sand to silt, clayey, strong humic concentrations in parts in the upper section	Transition zone with numerous reworked Pliocene sediments

This sequence is overlain by Pleistocene sediments. Up to 39.76 m below ground level, this sequence is divided into 10 cycles (I-X) which have similar internal structures that vary in

thickness and with respect to other parameters. Generally the cycles are repeated sequences of fining-upward suites which begin discordantly with sandy-gravelly sediments. Every sequence

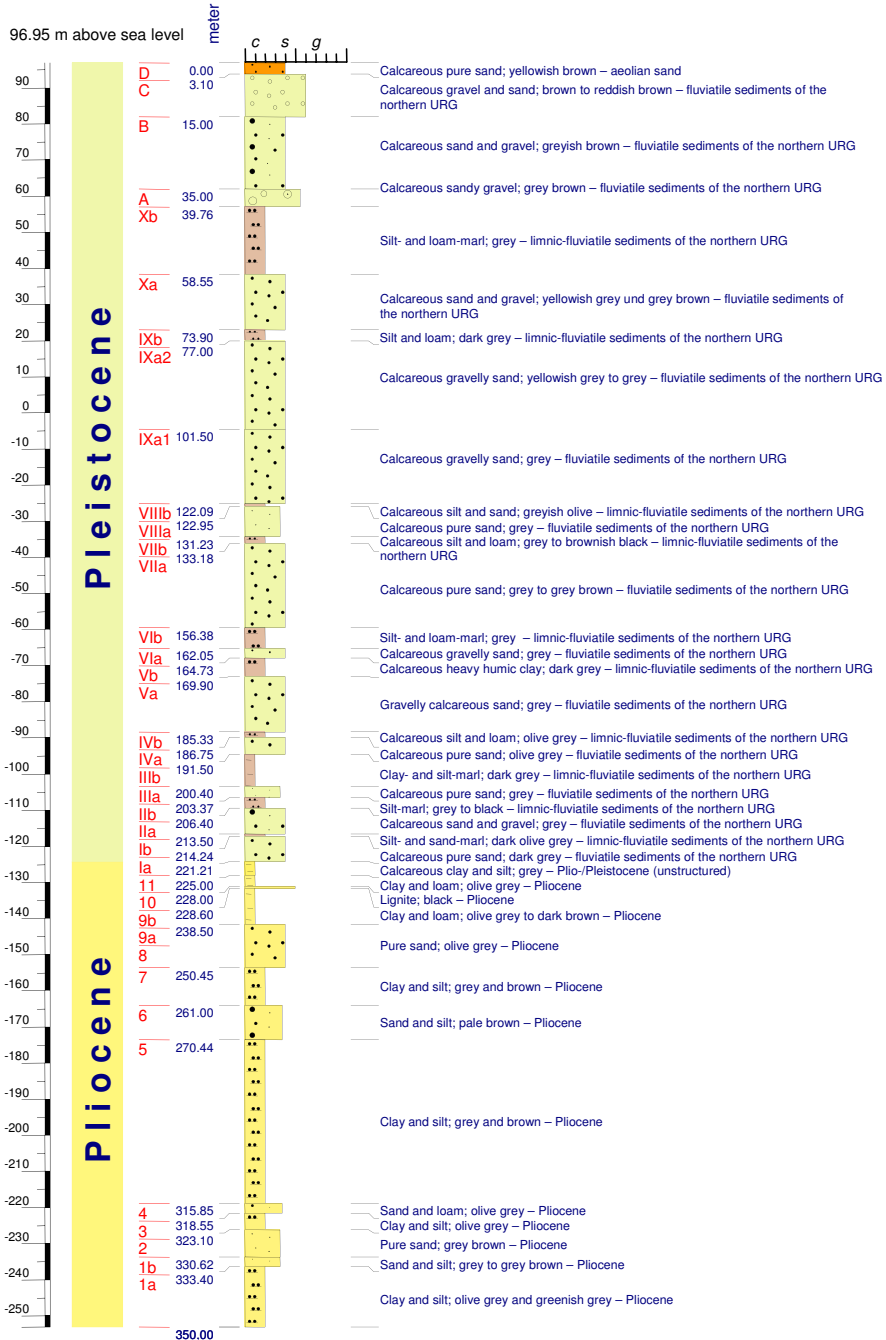


Fig. 3: Schematic geological section of the Viernheim research borehole showing a summary of the sedimentological units.

Abb. 3: Schematisiertes geologisches Profil der Forschungsbohrung Viernheim mit den zusammengefassten sedimentologischen Einheiten.

Table 1: Summary of some characteristic sediments occurring in the area drilled by the Viernheim research borehole.

Tab. 1: Zusammenfassung einiger charakteristischer Sedimente, die im Gebiet der Forschungsbohrung Viernheim auftreten.

	characteristic features
fluvial gravels and cobbles of the Rhine	Quartz, quartzite, crystalline rock, lydite, radiolarite
fluvial gravels and cobbles of the Neckar	Muschelkalk and Late Jurassic limestone, Buntsandstein sandstone, Keuper sandstone
Rhenish Facies	well to very well sorted fine up to medium grained sands, carbonate rich, greenish grey, light mica

ends with fine-clastic silts and clays. Other sequences can be identified within these cycles. The description repeatedly mentions sediments in "Rhenish Facies" (Table 1). This is a well to very well sorted grey to greenish-grey fine to medium grained sand. The carbonate content

can exceed 20 % in part.

A characteristic feature is the distinctive presence of mica. The light mica flakes can have diameters up to several millimetres.

This part of the well section has the following geological setup (cf. Figure 3):

Section	Depth in m below ground level	Description summary	Special features
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	

Section	Depth in m below ground level	Description summary	Special features
VIIIb	122.09-122.95	Thin fine sandy silts	With calcareous concretions and geodes
VIIIa	122.95-131.23	VIIIb is erosively overlain by sands with gravel grading into fine to medium-sized sands towards the top, 123-123.5 m oxidised, high concentrations of light mica in part	124.5-126.5 m in typical Rhenish Facies
VIIb	131.23-133.18	Fine clastic, reduced, more humic towards the top with sandy intercalations, oxidised in part	
VIIa	133.18-156.38	VIIb is erosively overlain by gravely sands, becoming coarser upwards as a coarsening-upward sequence, then dominant sand with gravely horizons, sand at the top	133.8-134 m in typical Rhenish Facies
VIb	156.38-162.05	Fine clastic sediments with gravely interbeds at 161.1-161.45 m, silt and clay weakly reduced with oxidation zones	Characteristic banding
VIa	162.05-164.73	VIb is discordantly overlain by next fining-upward sequence starting with gravely sands	
Vb	164.73-169.9	Fine clastic zone reduced at the bottom and more humic towards the top, from 168.2 m reduced, in part oxidised, then humic again	Gravels largely from the Neckar
Va	169.9-185.33	IVb is erosively overlain by coarse sands, gravely in part, very gravely at 182.5 m, fluctuating gravel concentrations in this section	169.9-172.5 m in typical Rhenish Facies. Pedogenic overprint in part
IVb	185.33-186.75	Silts and clays, partially reduced	Similar to IIIb
IVa	186.75-191.5	IIIb is discordantly overlain by coarser sands, grading into typical sands of the Rhenish Facies	
IIIb	191.5-200.4	Silts and clays, strongly humic in part, light grey to dark greenish-grey (reduced)	
IIIa	200.4-203.37	IIIb is discordantly overlain by well sorted, micaceous sands without gravely horizons	With typical Rhenish Facies, fining upward sequence overall

Section	Depth in m below ground level	Description summary	Special features
I b	203.37-206.4	Fine-clastic sediments, humic in part, also reduced	
I a	206.4-213.5	Ib is discordantly overlain by sandy gravel grading up into sand dominated sediments, with abundant mica in parts	Dominant limestones in the gravel fraction
I b	213.5-214.24	Mainly silts, reduced	
I a	214.24-221.21	Fining-upward sequence with gravelly sands and sands	First sediments in Rhenish Facies

This part of the section is overlain by a more coarse clastic carbonate unit more strongly influenced by Neckar sediments (A to C), top of the sequence formed by Weichselian to Holocene aeolian sands (D).

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	Gravelly 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 gravels
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 gravel

The cycles I-X reveal fluvial and limnic-fluvial sediments which were mainly deposited by the Rhine. Mixing with Neckar sediments occurs in the gravelly parts. Units A-C are, however, much more strongly dominated by gravel, and the influence of Rhenish sedimentation reduces upwards.

Gravel analysis was carried out at various depths to determine the provenance of the gravel (Table 2).

There are basically no significant changes in the gravel content. The values are similar to those for the area of the Neckar alluvial fans

described by e.g. LÖSCHER (1988). The fractions analysed are dominated by Keuper limestones and sandstones transported by the Neckar, as well as red sandstones from Buntsandstein areas. The interesting finding is that the limestones in the lowest sample in particular are partially dissolved. In addition to the Neckar pebbles, there is also mixing with a local component from the Odenwald. This is revealed by the relatively high proportion of red sandstones derived from the Buntsandstein, as well as crystalline pebbles. These are less well rounded, which indicates shorter transport distances.

Table 2: Summary of the gravel analysis at various depths in the Viernheim research borehole. The figures are percentages and add up to 100 % in total. 200 to 500 pieces of gravel were counted in the fraction higher than 5 mm.

Tab. 2: Zusammengefasste Schotteranalysen aus verschiedenen Teufenbereichen der Forschungsbohrung Viernheim. Die Werte geben Prozent an und summieren sich insgesamt auf 100 %. Gezählt wurden zwischen 200 und 500 Kiese der Fraktion größer 5 mm.

Layer	Depth [m]	quartz	crystalline	red sandstone (Buntsandstein)	limestone (Late Jurassic)	limestone (Muschelkalk)	lydite	sandstone (e.g. Keuper)	miscellaneous (chert, radiolarite, phyllite, claystone, quartzite, and marl)
C	5.3-5.75	20.5	22.5	8.5	17.0	17.2	1.6	6.7	6.0
B	31.7-31.8	16.2	11.4	3.1	18.3	29.3	2.6	4.1	15.1
IXa2	99-99.5	6.5	15.9	19.4	26.4	13.9	2.0	9.0	7.0
VIIa	148.2-148.5	26.6	10.5	12.2	16.7	20.7	1.1	9.6	2.5
VIb	161.3-161.4	12.9	11.3	3.5	39.5	13.2	6.4	7.7	5.5

The limestones are well to very well rounded because of their longer transport distances and lower hardness. The portions of up to 26.6 % quartz, and lydite occurring as an accessory component, indicate mixing with Rhine pebbles. These were transported long distances from either the Black Forest or the Alps.

3 Results

3.1 Heavy Mineral Analysis

Various systematic investigations of the heavy mineral composition of Pliocene and Pleistocene sediments in the Upper Rhine Graben have been undertaken previously (e.g. VAN ANDEL 1950; MAUS in BARTZ 1982; BOENIGK 1987; HAGEDORN 2004; HAGEDORN & BOENIGK 2008). Heavy mineral analysis is an important instrument for characterising fluvial Pliocene and Pleistocene fine sands from the Rhine system as part of work conducted in the middle Rhine area, the Lower Rhine Embayment and the Netherlands. The composition of the heavy

mineral distribution provides information on the source of the sediments as well as the weathering of the sediments.

171 samples from the Viernheim research borehole were analysed for their heavy mineral content. The main analytical method was that of BOENIGK (1983). Sodium polywolframate ($\text{Na}_6(\text{H}_2\text{W}_{12}\text{O}_{40}) \cdot \text{H}_2\text{O}$) with a density of 2.85 g/cm³ was used as the heavy liquid to separate the heavy from the light fraction in a centrifuge. The samples were boiled with concentrated hydrochloric acid prior to centrifuging to remove iron and manganese hydroxide crusts which would complicate the identification. The disadvantage of this method is the dissolution of carbonate, apatite and parts of monazite and olivine (BOENIGK 1983). This was deemed acceptable because of the benefit of being able to make comparisons with our own and other previous analyses.

Generally, the sediments in the borehole from a depth of 350 to 225 m are dominated by the stable heavy mineral group (Fig. 4). These are mainly zircon, but also tourmaline and rutile.

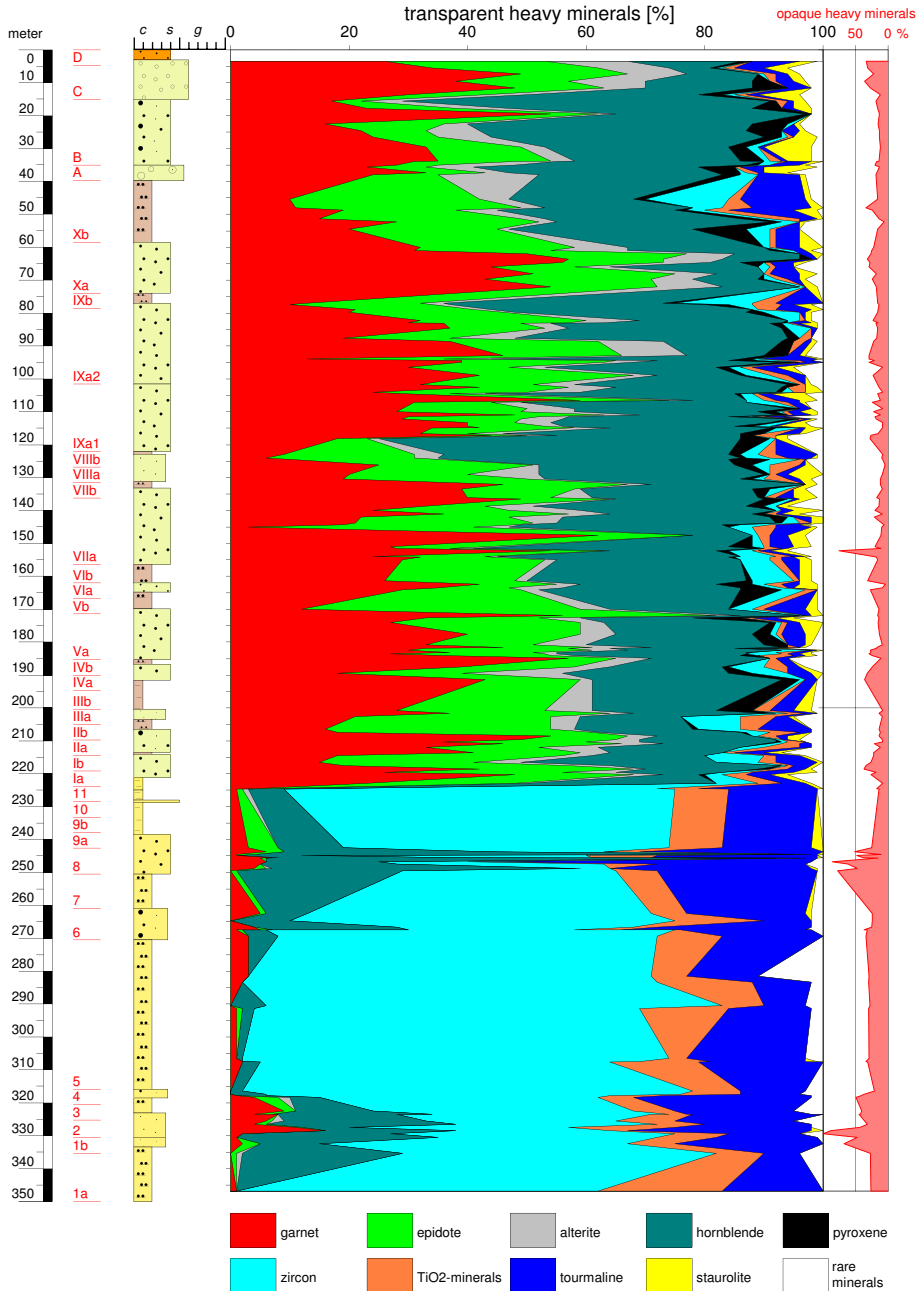


Fig. 4: Heavy mineral diagram on 171 sand samples from the Viernheim research borehole; hornblende includes green and subordinate brown hornblende; the TiO₂-group includes rutile, brookite and anatase; the rare minerals include andalusite, sillimanite, disthene, titanite, monazite and spinel.

Abb. 4: Schwermineraldiagramm an 171 Sandproben der Forschungsbohrung Viernheim; Hornblende beinhaltet grüne und untergeordnet braune Hornblende; die TiO₂-Gruppe schließt Rutil, Brookit und Anatas ein; die seltenen Minerale fassen Andalusit, Sillimanit, Disthen, Titanit, Monazit und Spinell zusammen.

Subordinately also occur heavy minerals of the Rhine Group (garnet, green hornblende and epidote) (cf. also Fig. 5). However, some samples have very high percentages of dark green hornblende (up to 86 %).

The composition of the heavy mineral spectrum changes significantly above sample 222,9-222,8 m: zircon, the TiO_2 -group and tourmaline become insignificant. These are replaced by garnet, epidote and green hornblende which determine the composition of the heavy minerals in the sand fraction, although the distribution overall remains relatively homogenous. This is emphasised even more when the heavy minerals are divided into separate groups (Fig. 5). The Rhine Group dominates with proportions between 60 and 80 %. Instable minerals have sometimes undergone strong alteration or are strongly dissolved. Garnet shows some evidence of corrosion, and green hornblende has been strongly altered in parts. Accessory minerals include the volcanic heavy minerals pyroxene and brown hornblende.

Boiling with concentrated HCl leads to the dissolution of some heavy minerals such as carbonate and apatite. A test run with 16 samples studied at the effect of the concentrated HCl treatment on the heavy mineral distribution. Figure 6 shows the distribution of selected samples with and without HCl treatment. Generally, the heavy mineral distribution remains the same with or without treatment. The differences lie within the range of statistical error. However, there is a significant rise in opaque heavy minerals (no HCl boiling), that is explained by the iron and manganese hydroxide encrustations on some grains which can therefore not be identified in transmitted light under a polarised microscope. Apatite now occurs in proportions up to 8 %, however, only in samples between 163.55 and 5.5 m. In general, counting during heavy mineral analysis becomes much more difficult, and some grains can only be identified after a lot of work. The difference for the sediments in the Upper Rhine Graben is, however, not as significant because of the presence of carbonate-rich sands which have not been subject to any very intensive

weathering processes after sedimentation (cf. Chapter 3.2). Heavy mineral analysis without boiling with HCl is very difficult in the non-calcareous Pleistocene terrace deposits with no overburden to protect them from weathering, which are characterised by strong iron and manganese hydroxide deposits, and are frequent in the Middle Rhine and Lower Rhine areas. This can give rise to erroneous results. Decisions to change the methodology should therefore be looked at very critically because changing the methodology would considerably restrict direct comparison with most of the old analysis data collected in the Rhine area.

3.2 Analysis of the Carbonate Content

The carbonate content was analysed in 136 sand samples from the Viernheim research borehole. The samples were initially ground up in a mortar before being ground down to ca. 100 μ in a ball mill. The carbonate content was determined using the method described by Scheibler (DIN ISO 10693), which involved two to three measurements. The results were used to derive an average number. Total carbonate content was determined in each case. The carbonate in sediments from the Upper Rhine Graben primarily consists of calcite (CaCO_3), and to a lesser extent also dolomite ($\text{CaMg}(\text{CO}_3)_2$).

The analysed sediments are almost all calcareous above the boundary at 225 m. Concentrations fluctuate significantly between 0.96 and 37.17 % (Fig. 7). The sediments in the lower sequences are almost completely non-calcareous. A notable finding is that the carbonate concentration in the Viernheim research borehole fluctuates very strongly in general. A similar carbonate distribution picture was also shown in sediments from the borehole drilled less than 1 km to the south down to a TD of 110 m to investigate the groundwater (STW Viernheim; R 3469366 – H 5491377; point 4 in Fig. 10). The carbonate concentration in 26 samples ranges from 0,02 to 26.85 %. The highest carbonate values are found in the fine clastic sediments (fine sands) predominantly in the

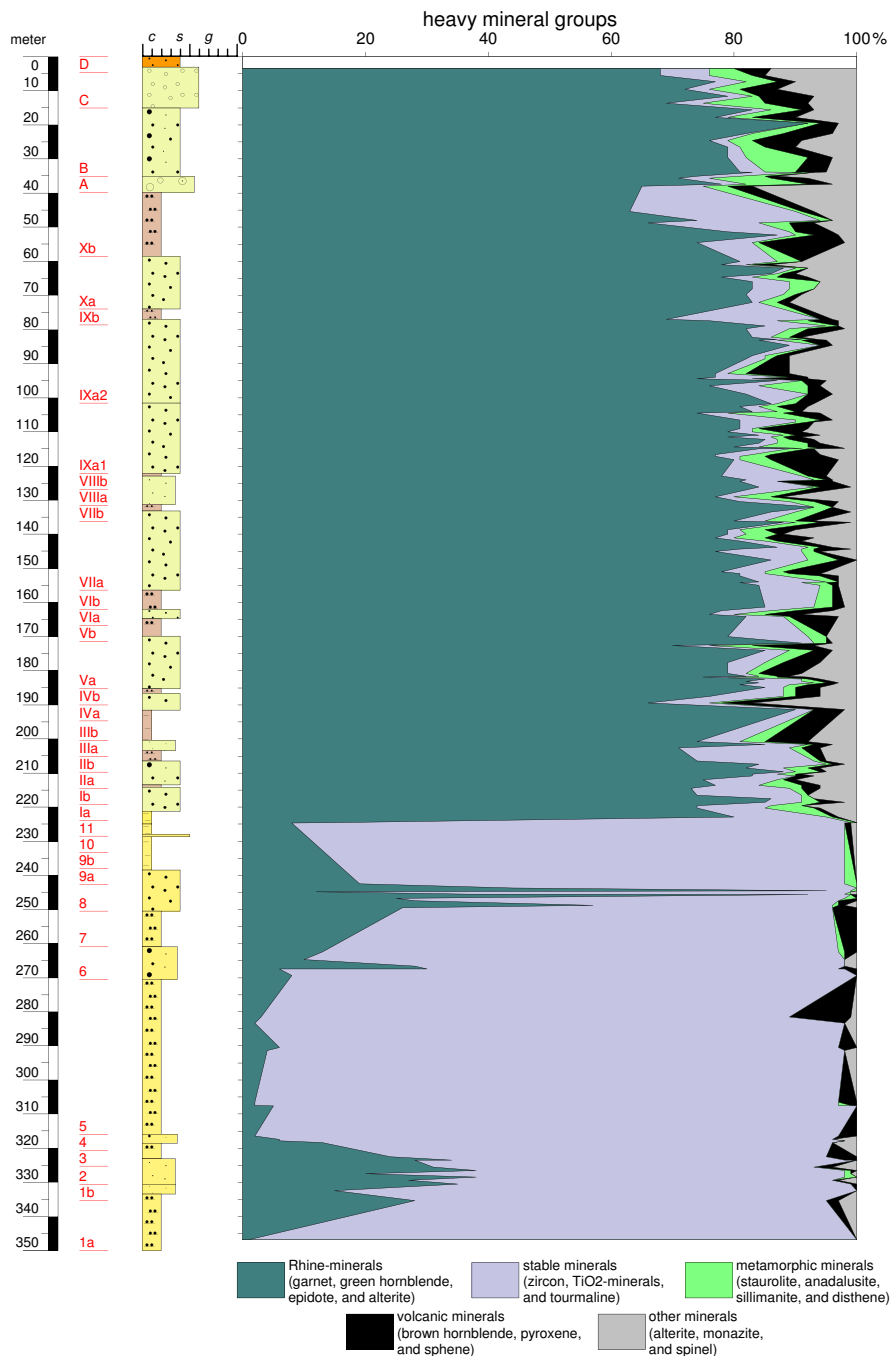


Fig. 5: Heavy mineral distribution divided into heavy mineral groups (after VAN ANDEL 1950 and VINKEN 1959), used for provenance analysis.

Abb. 5: Verteilung der Schwerminerale nach Schwermineralgruppen (nach VAN ANDEL 1950 und VINKEN 1959), die zur Liefergebietsanalyse verwendet wurden.

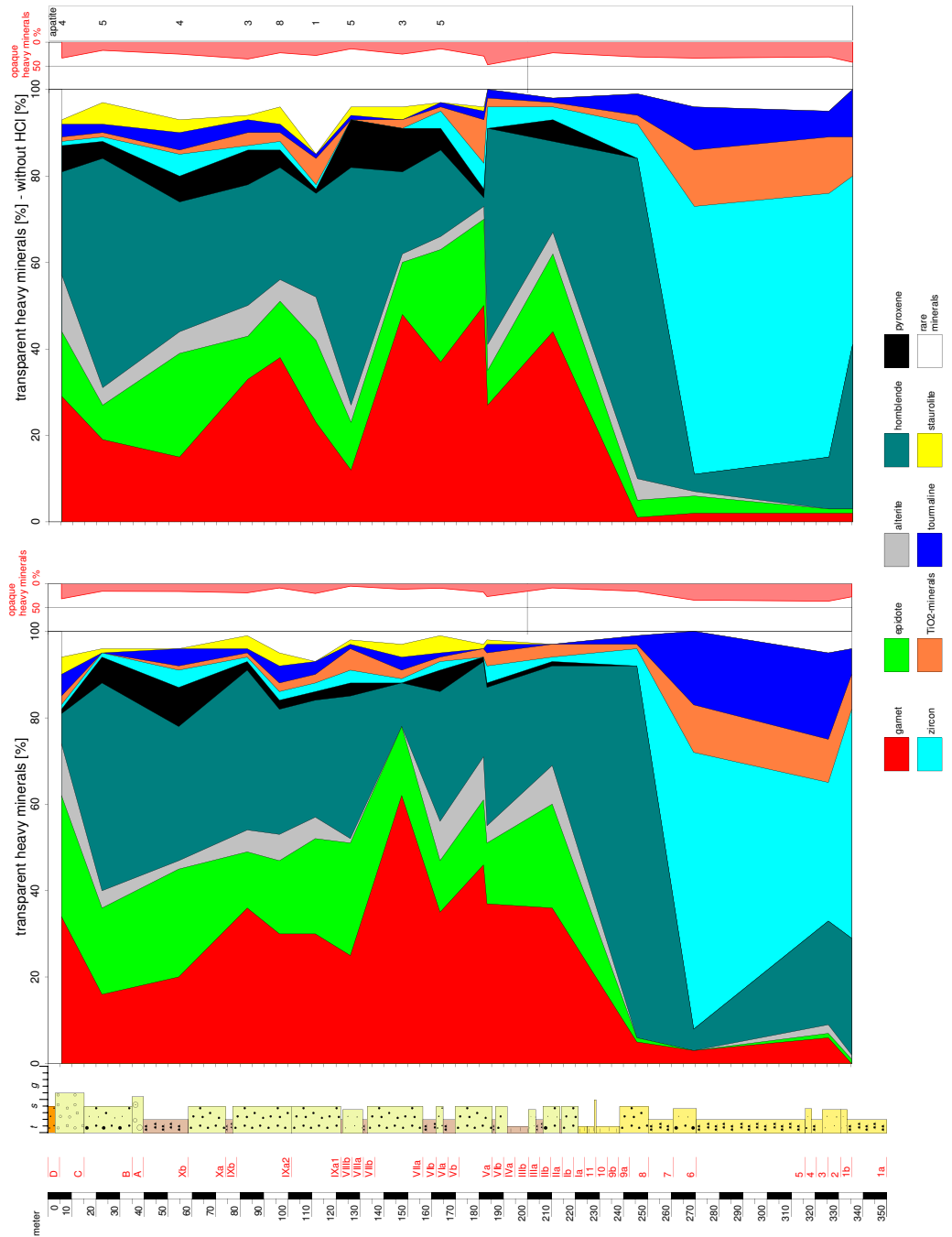


Fig. 6: Heavy mineral distribution in 16 selected samples from the Viernheim research borehole with and without HCl treatment. Apatite was counted separately in the samples prepared without boiling in HCl.

Abb. 6: Schwermineralverteilung von 16 ausgewählten Proben der Forschungsbohrung Viernheim mit und ohne Behandlung durch HCl. Bei der Probenaufbereitung ohne Kochen mit HCL wurden Apatite separat mitgezählt.

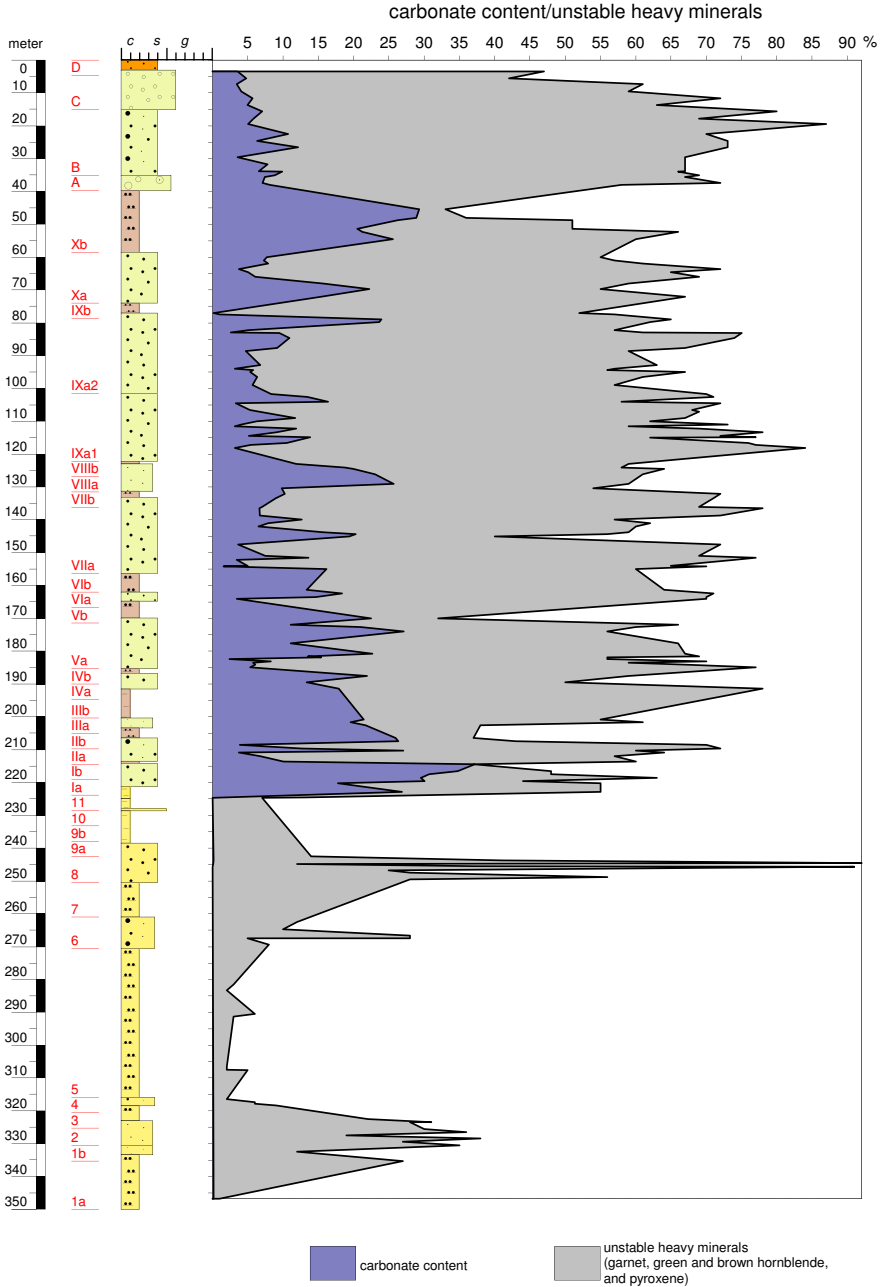


Fig. 7: Carbonate distribution from 136 sand samples from the Viernheim research borehole. The carbonate content is shown in relation to the group of unstable heavy minerals which weather easily in post-sedimentary environments.

Abb. 7: Carbonatverteilung von 136 Sandproben der Forschungsbohrung Viernheim. Der Carbonatgehalt wurde in Bezug zur Gruppe der instabilen Schwerminerale gesetzt, die postsedimentär leicht verwittern.

Rhenish Facies, as in the Viernheim research borehole. A similar picture is shown by another research borehole (C/00-BK01), lying between Biblis and Groß-Rohrheim (Fig. 1), approx. 18 km north-west of the Viernheim research borehole (R 3460250 – H 5508325). The Quaternary base lies almost 100 m below ground level. The carbonate concentrations in the 42 samples fluctuate between 1.39 and 22.71 %. They are therefore lower in general than the boreholes further to the south. However, the C/00-BK01 research borehole does not have the silty and clayey beds with fine sand intercalations which usually contain the calcareous sediments. Future work should investigate whether the distribution pattern of the carbonate concentrations can be correlated between boreholes.

4 Discussion

Subdivision of the Viernheim research borehole

The Viernheim research borehole can be divided into four sections (cf. Chapter 2). The zone from 350 to 225 m contains clay-rich, densely layered limnic-fluviatile deposits laid down on a floodplain. Some of these deposits have been subjected to strong post-sedimentary pedogenic overprinting giving rise to soils similar to plinthosols. These soils were formed by stagnant water or under the influence of groundwater, and have gley and pseudo-gley characteristics. Rust and bleach spots are typical of plinthosols. They are formed under humid-tropical climatic conditions. The fine clastic sediments contain calcareous clusters at some places and repeated intercalations of sandy deposits. Sections 8 to 9 (Fig. 3) comprise a fining-upward sequence topped by an organic sediment (lignite). The sands have partly high proportions of green hornblende indicating a local sedimentary source from the Odenwald situated to the east (Fig. 8). The extreme dominance of such an unstable mineral group (up to 86 %) must indicate the local provenance of the mineral because it would otherwise have become much more intensively mixed with

extremely stable heavy minerals. Amphiboles formed by magmatic crystallisation or by metasomatic processes are typical for the crystalline Odenwald geology. In the other cases, the heavy mineral spectrum is characterised by high percentages of stable minerals such as the dominant zircons, but also tourmaline and the TiO₂-group. The Black Forest and the Vosges are interpreted as the sources of the subordinate proportions of garnet and epidote (HAGEDORN & BOENIGK 2008). Sections 1 to 10 (Fig. 3) of the well section are classified as Pliocene.

The next section (Section 11) is a transition zone which follows discordantly from 225 to 221.21 m and is interpreted as a reworked horizon. This section contains deposits derived from the underlying sediments which have been significantly reworked. In terms of heavy minerals, it contains a rather localised stable spectrum as well as a typical Pleistocene spectrum dominated by the Rhine Group. This section is preliminarily classified as Pliocene to Pleistocene.

The zone from 221.21 to 39.76 m in the Viernheim research borehole is made up of ten basically repeated suites (I-X) which begin autocyclically and discordantly with gravely sands and end with clayey sediments or peat. The thickness of each cycle fluctuates between 6.17 m (IV) and 48.19 m (IX). Additional partial sequences were identified in each cycle. The sediments (I-X) are almost exclusively medium to strongly calcareous. A typical feature is the repeated occurrence of very well sorted sands of the "Rhenish Facies" (cf. Chapter 2.2). These were formed by fluvial flood sediments from the Rhine. The whole section in the well contains sediments dominated by the Rhenish Facies, with a gravel fraction which includes pebbles from the Neckar, and subordinately local components with crystalline pebbles from the Odenwald and red sandstones from the Buntsandstein. However, high percentages of quartz and the presence of lydite also show that the gravel fraction contains material sourced from areas to the south of the Neckar (Tab. 1 and 2).

The fluvial part of the section is topped from 39.76 to 3.1 m by highly gravely sands and

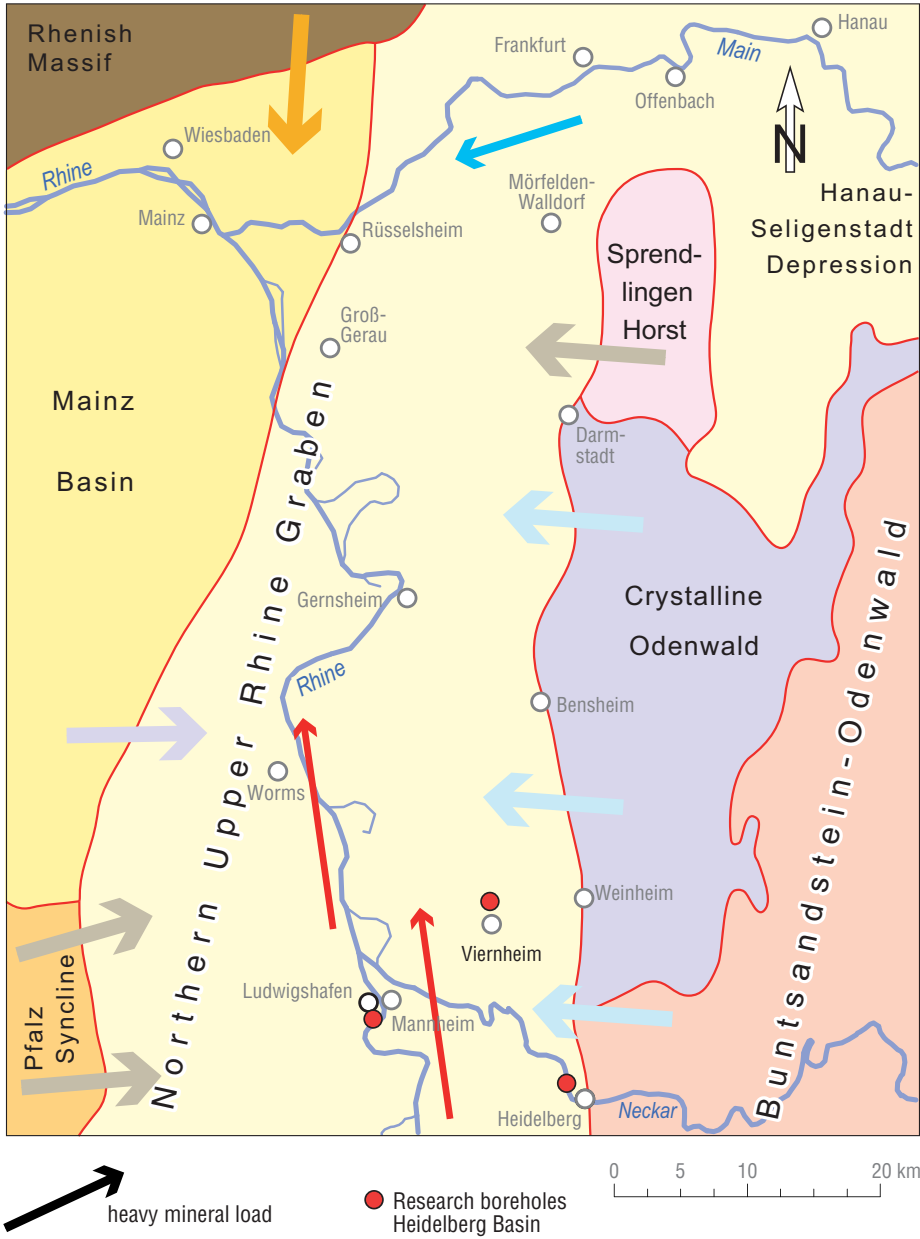


Fig. 8: Heavy mineral deposits during the Pliocene in the northern Upper Rhine Graben. Dominated by locally reworked sediments with stable heavy minerals and subordinate unstable heavy minerals derived from the Black Forest and the Vosges. Repeated local deposition from the graben margins which dominantly supply green hornblende in the eastern part of the northern URG.

Abb. 8: Schwermineralschüttungen während des Pliozäns im nördlichen Oberrheingraben. Dominant sind lokal aufgearbeitete Sedimente mit stabilen Schwermineralen und untergeordnet instabilen Schwermineralen, die aus dem Schwarzwald und Vogesen stammen. Wiederholt kommt es zu lokalen Schüttungen von den Grabenrändern, die im östlichen Teil des nördlichen URG dominant grüne Hornblende liefern.

gravels mainly laid down by the Neckar. The dominance of the Rhine Group in the heavy mineral fractions in the sands, however, indicates mixing with Rhenish sediments around Viernheim. This is also indicated by the quartz constituents and lydite in the gravel fraction (Tab. 2). This section is also marked by dominant fining-upward and subordinately coarsening-upward sequences. The top of the section from 3.1 m to the ground surface is formed by Weichselian to Holocene aeolian sands.

The unequivocal identification of the Rhine signal in the heavy mineral fraction of the sands can be deduced from the proportions of heavy minerals in the sediments. Unlike the Pleistocene Neckar sediments which only contain around 0.04 % heavy minerals, the Rhine sediments contain approx. 0.5 % heavy minerals. This means that the fluvial Rhenish sediments have around 10 times more heavy minerals than the Neckar sediments (Fig. 9). HAGEDORN (2004) reports similar findings. The change in provenance in sample 222.8 m show a strongly alpidic dominated heavy mineral spectrum, indicating the connection of the Rhine to the Alpine drainage system. The general homogenous structure of the heavy mineral distribution (Fig. 4 and 5) in the Pleistocene shows that lateral sources of sediment in the Viernheim area are almost completely absent. This locality is therefore different to that of the Ludwigshafen-Parkinsel borehole, for instance, which is marked by the periodic deposition of local sediments from the western graben margin (HAGEDORN & BOENIGK 2008).

This confirms the assumption by HAGEDORN & BOENIGK (2008), that the Rhine tended to flow in the eastern part of the northern URG during the Pleistocene. Subordinate proportions of volcanic heavy minerals (pyroxene and brown hornblende) are probably not associated with the initiation of Middle Pleistocene East Eifel volcanism prior to approx. 700 ka (BOGAARD & SCHMINCKE 1990). In principle, the Neckar could also be the source for the pyroxenes as well as the brown hornblendes, although a strong decrease in the pyroxenes is observed towards its confluence with the Rhine as a

result of dissolution and dilution by tributaries (VAN ANDEL 1950).

The importance of heavy mineral analysis for provenance analysis in the northern Upper Rhine Graben is highlighted by a comparison of the unstable heavy minerals (garnet, green and brown hornblende, and pyroxene) with the carbonate content (Fig. 7). The post-sedimentary weathering of sediments with a pedogenic overprint initially gives rise to a decrease in and relocation of the carbonate content. If the section has also undergone pedogenic overprinting, which was not obvious in the section, this would also have led to a decrease in the concentration of unstable heavy minerals. However, this is not the case in the Viernheim research borehole because zones with lower carbonate contents for instance have raised levels of unstable heavy minerals. No correlation is shown between low carbonate contents and low concentrations of unstable heavy minerals. This in turn means that the Pleistocene sediments have almost preserved their original heavy mineral association and are therefore ideal for provenance analysis. Some of the samples show signs of dissolution and traces of corrosion.

Stratigraphy

The lithostratigraphic subdivision of the Viernheim research borehole is proving difficult at the current state of the investigation because of the general absence of reliable data. The initial correlations can only be undertaken on the basis of analogies with other boreholes in the area, in the Lower Rhine Embayment (LRE), and in The Netherlands (detailed investigations in the Dutch/German border by e.g. DONDEERS et al. 2007; KEMNA & WESTERHOFF 2007; WESTERHOFF et al. 2008). The most distinct lithological changes in the borehole are at 225 m, and after the thin reworked horizon at 221.21 m. The change at 225 m is considered to be the boundary between the Pliocene and the Pleistocene. The Plio-Pleistocene boundary at 2.58 Ma is used in the sense of BOWEN & GIBBARD (2007), GIBBARD & COHEN (2008), LITT

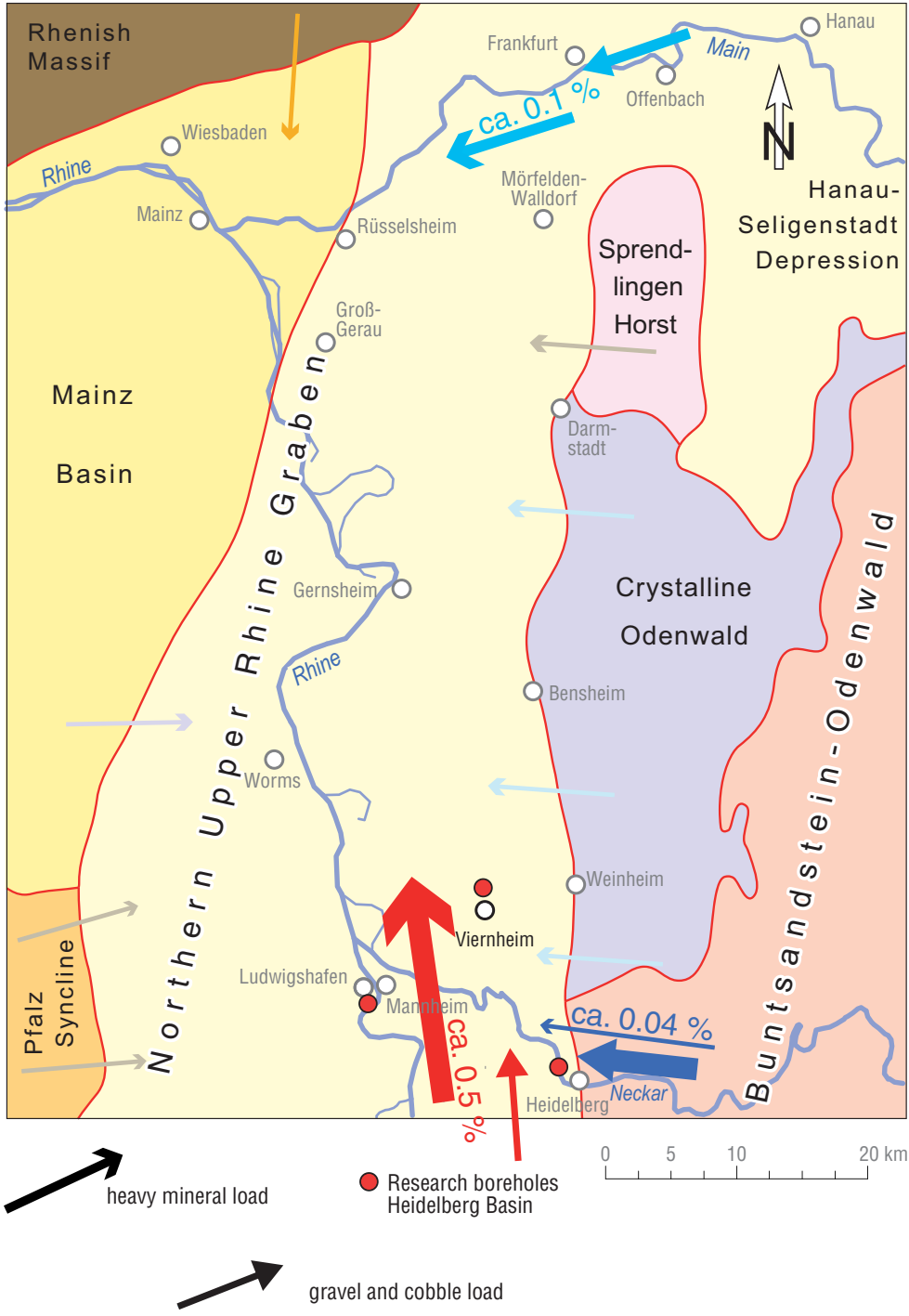


Fig. 9: Heavy mineral and gravel deposition during the Pleistocene in the northern Upper Rhine Graben.

Abb. 9: Schwermineral- und Kiesschüttungen während des Pleistozäns im nördlichen Oberrheingraben.

(2007), and PREUSSER (2008) and coincide with the palaeomagnetic Gauss/Matuyama boundary. The deposition of Rhine Group alpine heavy minerals in the URG already is of Late Pliocene age (HAGEDORN & BOENIGK 2008; PREUSSER 2008). Investigations in the LRE confirm this: a first shift from a stable heavy mineral spectra to unstable spectra of alpine type was detected in the Kieseloolite Formation (KEMNA 2005, 2008 a, b). The Öbel beds (Latest Pliocene) also contain unstable heavy mineral spectra (BOENIGK & FRECHEN 2006; DONDERS et al. 2007; KEMNA & WESTERHOFF 2007). In some parts of the LRE the Öbel beds cover the Reuver Clay s.s. and thus define the top of the Pliocene sedimentation. In the Viernheim research borehole, no typical alpine heavy mineral spectra can be found below a depth of 225 m. Consequently, these sediments are correlated with the Pliocene. A larger unconformity at this depth must be accepted. There is no doubt that a marked change in sedimentation took place at that time. At the present stage of the investigation a further stratigraphic subdivision of these Pliocene sediments is not possible. This characteristic change at 225 m in the Viernheim research borehole is comparable to the boundary at 176 m in borehole P 34 Ludwigs-hafen-Parkinsel (WEIDENFELLER & KÄRCHER 2008; WEIDENFELLER & KNIPPING 2008). There are also indications in the P 34 borehole of a palaeomagnetic change from the Gauss to Matuyama-Chron (ROLF et al. 2008). ROLF et al. also proposed that a characteristic change of the magneto-minerals from goethite (Pliocene) to greigite (Pleistocene) can be interpreted as a climatic signal. Pollen analysis from organic sediments just beneath and above the boundary at 225 m are planned to provide important information on the age of this section in the Viernheim research borehole.

Another key zone in the borehole is the Xb section (58.55 to 39.76 m). According to the hydrostratigraphic classification, this uppermost significant fine-clastic section of the borehole corresponds to a high resolution Obere Zwischenhorizont (OZH) in the sense of the hydrogeological mapping in the Rhine/Neckar zone (HGK 1999) or the sequence-stratigraphic

equivalent to the Ladenburg Horizon (Symbolschlüssel Geologie Baden-Württemberg 2007, VILLINGER 2005). This section has been classified as part of the Cromerian Complex since ENGESSER & MÜNZING (1991) on the basis of its mollusc fauna. Palynological investigations by KNIPPING (2004, 2008) on various sections in the Mannheim-Ludwigshafen area led to the conclusion that the OZH contains several interglacial flora which are assigned to the Cromerian Complex. A correlation with the Eemian interglacial is ruled out. This means that Section Xb in the Viernheim borehole can also be assumed to be of Cromerian age. Important biostratigraphic indications of the Lower Pleistocene sections in particular are provided by the investigations conducted by WEDEL (2008). No other chronostratigraphic interpretations of the Viernheim research borehole are possible at the current stage of the research.

Regional Trends

Regional trends can only be investigated at on the basis of characteristic horizons or boundaries which extend over larger areas. The horizons selected for this purpose are the Quaternary base as well as the top and base of Section Xb. This of course presupposes that the same unit or boundary could be selected for the correlation in the various wells and correlation could only use those boreholes with reliable stratigraphic logs, and gamma logs where possible (Tab. 3 and Fig. 10). Despite a large number of boreholes in the Hessisch part of the area of investigation, only eight boreholes in the Viernheim-Bensheim area are currently suitable for such a correlation.

The top of Section Xb dips to the south towards the centre of subsidence of the Heidelberg Basin. The thickness of the unit increases accordingly. The minor amount of data available on the Quaternary base indicates a similar picture with a major increase in thickness from north to SSE. Care must be taken for the difference in the quality of the samples. The absence of Section Xb in the Einhausen research borehole highlights a fundamental problem when look-

ing at the Pleistocene sedimentary fill in the northern Upper Rhine Graben: some horizons are very difficult to correlate, particularly towards the north. Despite high subsidence rates in this part of the northern URG as well, horizons are absent and can no longer be lithologically correlated even over short distances. Although correlation seems to function reasonably well in the centre of the Heidelberg Basin, there are also some fine-clastic horizons here which are only of restricted use for lithostratigraphic correlations. For instance, a fine-clastic horizon occurs in the Deponie Hirschländer borehole GWM1/03 at a depth of 21.58 to 25.65 m (thickness 4.07 m). This horizon is not seen in any of the surrounding boreholes. This is interpreted as indicating the presence of a very local channel deposit cut into the older sediments. Although easier to correlate regionally, the flood deposits of Section Xb become thinner as the distance from its subsidence centre increases, and even disappear in some cases. The conclusion drawn from this is that the use of sequence-stratigraphic models – as used for instance by LANG (2007) for the Hanau-Seligenstadt Basin (Fig. 1), a parallel structure to the URG – are more promising. Determining the A/S rates (A= accommodation space available for the sediment; S: sediment supply), and the interpretation of A/S domains can be used successfully in sedimentary basins with low subsidence rates. This approach needs to be tested first in sedimentary basins with higher subsidence rates, such as the northern URG.

Comments on supra-regional correlation and outlook

The Rhine as the link between the Alpine and North European glaciated areas also linked up different areas of provenance, accumulation areas and tectonic structural areas during the Quaternary (PREUSSER 2008; WESTERHOFF 2008). The typical sediments of the Rhenish Facies which are wide-spread in the northern Upper Rhine Graben also have equivalents downstream. At the northern edge of the URG in Wiesbaden (Fig. 1), deposits called

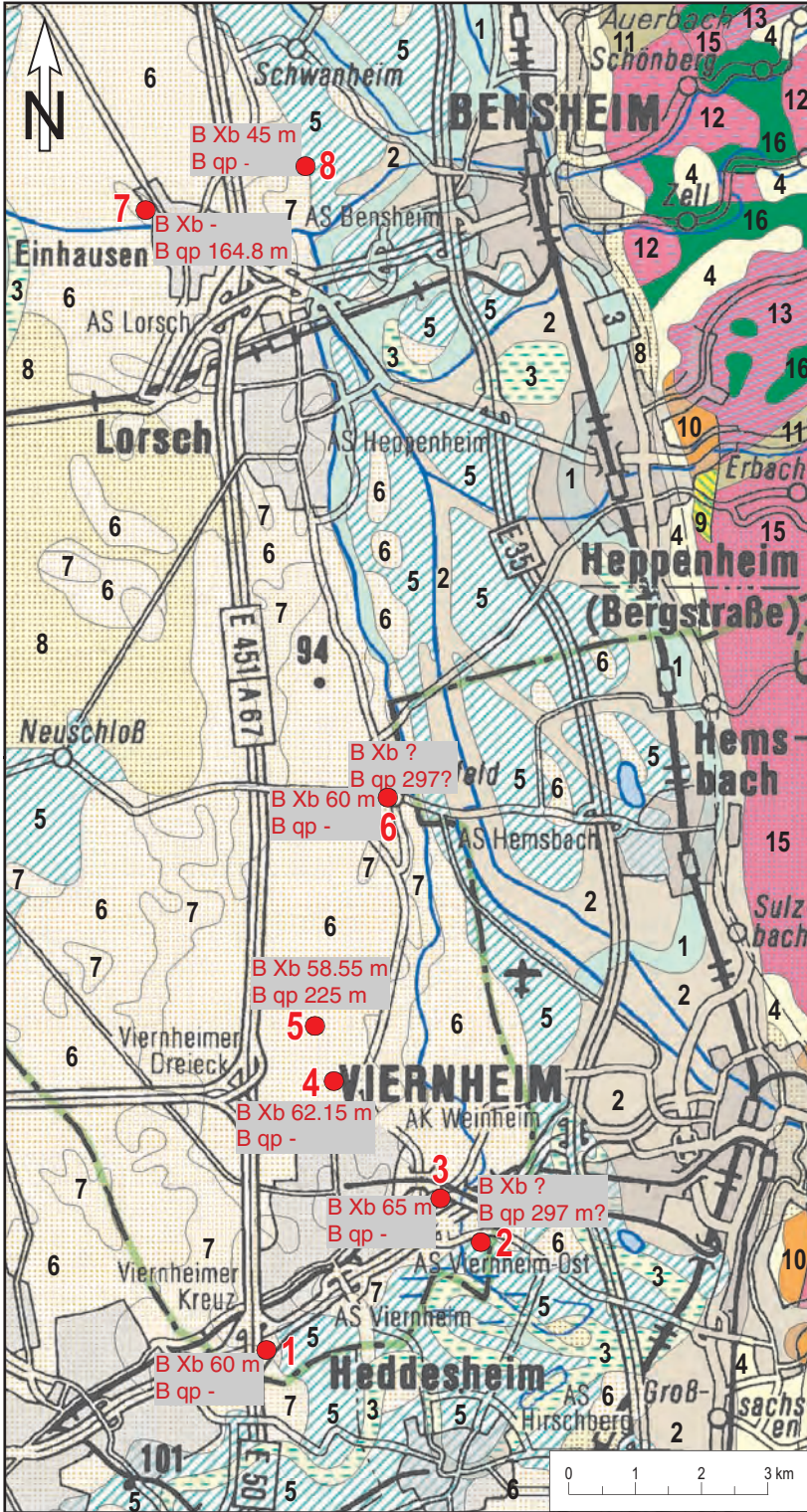
the Mosbach Sands occur which have supra-regional significance because of their rich and typical fossil content (BOENIGK 1977/78; KAHLKE 2007; KELLER 1999). In particular, the Graues Mosbach Horizon (nowadays called the Haupt-Mosbach-Subformation, HOSELMANN 2007b) assigned to the Cromerian Complex, has significant macroscopic and sedimentary petrographic similarity with the Rhenish Facies in the Heidelberg Basin in the URG. The lower Middle Rhine zone contains the Hönningen Sands in the Mittelrhein-Hauptterrassen Formation (WEIDENFELLER 2007), which is almost identical to the Rhenish Facies in the URG (BIBUS 1980; BOENIGK & FRECHEN 2006; BOENIGK & HOSELMANN 1991; HOSELMANN 1996). The Rhine Group dominates the heavy mineralogy in these sediments, and carbonate contents of almost 30 % have been measured. These sediments are probably part of the Cromerian Complex. Similar sediments also occur in the upper Middle Rhine (BIBUS & SEMMEL 1977). No other correlatable sediments are known in the other terraces of the Middle Rhine or in the Lower Rhine Embayment. The question therefore remains whether the sediments either did not accumulate in other times or whether they did not survive because they are easy to erode. In the area of the lower Middle Rhine, fluvial terrace sediments occur in a series of terraces so that the uplift of the Rhenish Massif exposed the sediments to much stronger post-sedimentary erosion than the deposits in the Heidelberg Basin.

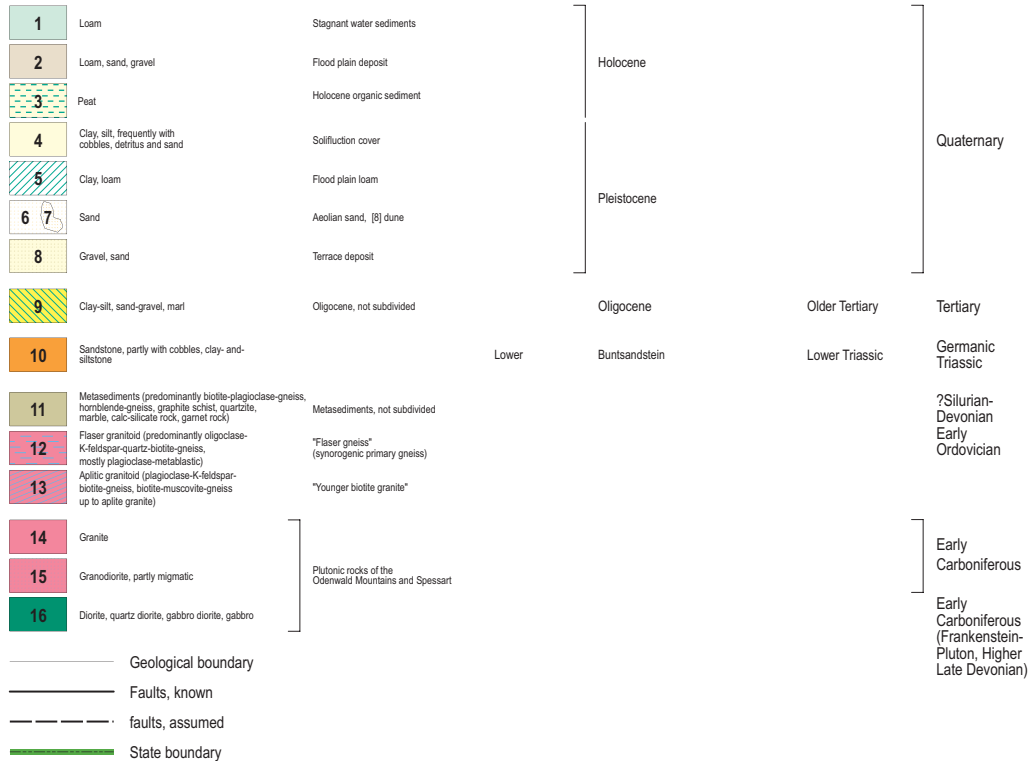
A more detailed explanation is required in the future of the type of mechanism which controlled the depositional cycles. A key role is played here by the uplift of the German Mittelgebirge and the subsidence of the URG, and in particular in this case the Heidelberg Basin. The cause for the start of a new cycle could also have been controlled by climatic conditions because periglacial environmental conditions were required to make these sediments available, particularly the coarse clastic sediments, which then accumulated in proximal locations. The interaction of sea level rises and falls on accumulation behaviour in the URG needs to

Table 3: Selected boreholes from the Viernheim-Bensheim area with the depths of the characteristic marker horizons. Petrographic descriptions of the flush drilled wells are uncertain.

Tab. 3: Ausgewählte Bohrungen aus dem Raum Viernheim–Bensheim mit der Tiefenlage charakteristischer Leithorizonte. Die petrographischen Beschreibungen der Spülbohrungen sind unsicher.

No. (Fig. 10)	Identifier Drilling method total depth (TD)	Gamma logging	Top layer Xb [m]	Base layer Xb [m]	Thickness layer Xb [m]	Base of the Quaternary sediments [m]
1	Deponie Hirschländer GWM1/03 dry drilling; TD 80 m	–	46	60	24	–
2	EWS Viernheim 2006/ 780 flush drilling (petrography uncertain); TD 300 m	–	?	?	?	297?
3	EWS Viernheim 2006/ 594 (2008) flush drilling; TD 140 m	X	45.5	65	19.5	–
4	STW Viernheim tube core drilling and flush drilling; TD 110 m	X	42.25	62.15	19.9	–
5	FB Viernheim B1/06 tube core drilling; TD 350 m	X	39.76	58.55	18.79	225
6	EWS Hüttenfeld 2007/ 581 flush drilling; TD 99 m	X	43	60	17	–
7	FB Einhausen C/01-BK4 tube core drilling; TD 205 m	X	–	–	0	164.8
8	WW Feuersteinberg GWM 75t flush drilling; TD 65 m	X	35	45	10	–





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Fig. 10: Distribution of various boreholes drilled recently in the southern Hessisches Ried used to evaluate "regional trends". The well locations are plotted on the geological base map of Hessen 1:300,000. The graben margin at the boundary to the crystalline Odenwald can be seen in the eastern part of the map; in the south-east with sedimentary rock remains of the lower Buntsandstein.

B Xb = Base section Xb in m below ground level; B qp = Base Pleistocene in m below ground level

Abb. 10: Verteilung verschiedener in den letzten Jahren abgeteufter Bohrungen im südlichen Hessischen Ried, die für die Auswertung „regionaler Trends“ verwendet wurden. Die Bohransatzpunkte wurden in die Geologische Übersichtskarte 1:300.000 von Hessen eingetragen. Im östlichen Teil des Kartenausschnitts ist an der Grenze zum kristallinen Odenwald der Grabenrand zu erkennen; im Südosten mit Sedimentgesteinsresten des unteren Buntsandsteins.

B Xb = Basis Sequenz Xb in m unter GOK; B qp = Basis Pleistozän in m unter GOK

be clarified for this purpose. Rapid rises in sea level can also cause water to back up in the hinterland and possibly trap accumulation in front of the rising Rhenish Massif and lead to increased coarse clastic sedimentation in the southern Upper Rhine Graben and in the northern URG north of the Karlsruhe High to dominant fine-clastic sedimentation. Answering these questions requires detailed analysis of cores in the Heidelberg Basin because understanding the depositional processes and palaeogeographic development of this region and the fluvial Rhine system is only possible in the high resolution deposits which are only found here in the thickest Quaternary sedimentary succession in the URG.

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Long sequence of Quaternary Rocks in the Heidelberg Basin Depocentre

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Abstract: A description and classification of the successions of the new scientific core drillings at Heidelberg is presented. Since 2002 drilling and research activities were ongoing in the Heidelberg Basin (HDB), as a mid-continental sedimentary archive within the Upper Rhine Graben (URG), Germany. The HDB is supposed to host one of the longest continuous successions of Quaternary sediments in Europe, due to continuous subsidence of the basin and sediment input from various sources. The HDB is about half-way between the Alpine source area of the Rhine and the North Sea. Here the Quaternary input is least affected by discontinuities due to climate events as alpine glacier meltdown events or periods of low sea level. Reverse, the low influence of climate leads to a larger tectonic control. The sedimentary succession of more than 500 m is considered as primarily controlled by tectonics, but with incorporated climate signals. For classification purposes, sediment provenance, lithofacies-associations, and the ratio of accommodation space and sediment input are used. Some biostratigraphic markers are also available. We suggest a sedimentary scenario where the overall fluvial environment is twice interrupted by lacustrine intervals. The accommodation space varies too: in one period it expands even beyond the eastern boundary fault of the HDB.

[Mächtige Abfolge quartärer Sedimente im Depozentrum des Heidelberger Beckens]

Kurzfassung: Die neue Forschungs-Kernbohrung aus Heidelberg wird beschrieben und gegliedert. Die Forschungs- und Bohraktivitäten im Heidelberger Becken (HDB) begannen im Jahr 2002; sie erschließen ein kontinentales Sedimentarchiv im Oberrheingraben (URG). Im HDB wird eine der längsten Sedimentabfolgen quartärer Sedimente in Europa erwartet, dank kontinuierlicher Subsidenz des Beckens in Verbindung mit kontinuierlichem Input von Sedimenten unterschiedlicher Herkunft. Das HDB befindet sich auf halber Strecke zwischen dem alpinen Einzugsgebiet des Rheins und seiner Mündung in die Nordsee. Eine kontinuierliche Sedimentation ist hier eher möglich als am Alpenrand mit seinen Schmelzwasser-Erosionsereignissen oder an der Küste mit ihren Meeresspiegelschwankungen. Dieser eher geringe Einfluss des Klimas hat zur Folge, dass die Tektonik eine umso größere Rolle bei der Steuerung der Sedimentation spielt. Die über 500 m mächtige quartäre Abfolge ist daher in erster Linie durch Tektonik kontrolliert, wobei Klimasignale ebenfalls

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erkannt werden können. Die hier vorgestellte Gliederung der Abfolge beruht auf Provenienz, Lithofazies und wechselnden Verhältnissen von Akkomodationsraum und Sedimentinput (*a/s*-ratio). Dazu kommen biostratigraphische Zeitmarken. Im skizzierten Sedimentations-Szenario dominieren fluviale Verhältnisse; dazwischen zwei lakustrine Abschnitte. Letztere sind verknüpft mit zunehmendem Akkomodationsraum, der in mindestens einer Zeitscheibe über die Grabenrandstörung hinweg sich bis in die Täler des Odenwalds erstreckt.

Keywords: Upper Rhine Graben, Heidelberg Basin, research borehole, lithofacies, sediment provenance, sequence stratigraphy, lithostratigraphy, biostratigraphy, differential uplift, subsidence.

1 Introduction

This paper summarises first results and impressions of the newly cored research borehole at Heidelberg. It is located at the depocentre of the „Heidelberg Basin“ (HDB), close to the eastern margin of the northern Upper Rhine Graben (URG). The probably largest and most complete Quaternary sediment succession along the Rhine has been exhibited, amounting to > 500 m. It proved to be a fairly continuous continental archive of the Quaternary in superposition and quite good resolution.

The sediments are strongly influenced by the input of the river Neckar, a major tributary of the Rhine which has its outlet into the URG within Heidelberg. Two other boreholes representing other parts of the HDB are the borehole of Viernheim in the geographic centre of the basin (HOSELMANN 2008), and the borehole of Ludwigshafen at the western margin (WEIDENFELLER & KÄRCHER 2008, ROLF, HAMBACH & WEIDENFELLER 2008; Fig. 1). Their sediments are both dominated by input of the Rhine.

All three boreholes are part of a multidisciplinary research project (The Heidelberg Basin Drilling Project; ELLWANGER et al. 2005, 2007, GABRIEL et al. 2008). Its aims will be to identify the interaction of climate and tectonics as controlling mechanisms of sedimentation, describe properties and 3D architecture of the infill, and contribute to the correlation of alpine and north European Quaternary stratigraphy. This paper only contributes to the basics of this research, as is it primarily focused on the Heidelberg borehole.

2 Regional geological context

The northern URG contains a more or less continuous succession of Oligocene, Neogene and Quaternary sediments, which accumulated due to ongoing subsidence since Oligocene time (DÉZES, SCHMID & ZIEGLER 2004, DURINGER 1988, GIDEON, LOPES CARDOZO & BEHRMANN 2006, ROTSTEIN et al. 2006, SCHUMACHER 2002, ZIEGLER 1992). In the Quaternary, the subsidence increased towards the east, which led to a halfgraben architecture with a maximum sediment thickness at the eastern margin of the URG, i.e. the Heidelberg deep (Heidelberger Loch, location of the Heidelberg borehole, BARTZ 1974). The western part of the Graben is slightly uplifted. As a result, the HDB lowlands are bordered in the east by the Odenwald highlands and the Kraichgau depression. West of the HDB, there is a widespread foothill landscape within the URG (PETERS 2007), and the Pfälzerwald highlands at the western URG shoulder (Fig. 1).

The subsidence of the HDB is balanced by sediment input throughout the Quaternary. The primary sediment sources are the fluvial systems of Rhine and Neckar. The deposits mainly reflect this fluvial environment, but also include intervals dominated by local coarse debris input from the nearby highlands, and also some lacustrine intervals. The HDB succession is, therefore, not only an archive of the Quaternary stratigraphic evolution, but also of the varying sedimentary dynamics.



Fig. 1: Location of the UniNord boreholes in Heidelberg and other boreholes. The Eastern Boundary Fault (EBF) of the Upper Rhine Graben (URG) separates the lowland plains of the Upper Rhine from the adjacent highlands. The Heidelberg Basin (HDB) covers approximately the area between the present River Rhine and the EBF, extending some 10 – 15 km north and south of Heidelberg. The highlands east of the EBF are two-parted, the Odenwald anticline (highlands) in the north, and the Kraichgau syncline (lowlands) in the south. The Neckar valley is incised into the southern Odenwald highlands, as is the buried early Neckar valley at Mauer.

Abb. 1: Lokation der UniNord Bohrungen in Heidelberg und anderer Bohrungen. Die „östliche Hauptverwerfung“ (EBF) des Oberrheingrabens (URG) trennt die Oberrheinebene von den angrenzenden Mittelgebirgen. Das Heidelberger Becken (HDB) erstreckt sich zwischen dem heutigen Rheinlauf und der EBF, sowie, ausgehend von Heidelberg, jeweils 10 – 15 km nach Norden und Süden. Die Mittelgebirge östlich der EBF sind zweigeteilt, im Norden die Odenwald-Antikline (als Mittelgebirge) und im Süden die Kraichgau-Syncline (als Tiefland). Das Neckartal ist in den südlichen Odenwald eingeschnitten, wie auch das verlassene Neckartal bei Mauer mit seiner Talverschüttung.

3 Definitions and methods

3.1 Definition of „Quaternary“ in the Upper Rhine Graben (URG)

Some popular stratigraphic schemes of the URG are difficult for stratigraphers to apply, as chronostratigraphic terms are used to describe lithostratigraphic units (Table 1 in GABRIEL et al. 2008). In this routine, „Quaternary“ and „Pliocene“ are defined by their sediment provenance (e.g. BARTZ 1982, HGK Rhein-Neckar 1999). I.e. „Pleistocene“ Rhine sediments enclose material of alpine origin, „Pliocene“ sediments don't. This refers to a major event in the history of the alpine Rhine, when its course changed from „circumalpine“ towards the North Sea (VILLINGER 1998).

In the URG, this provenance transition is usually a very distinct and widely recognized boundary. The alpine origin of the sediments above is easily detectable by carbonate content and identification of pebbles of alpine origin. It can further be proved by heavy mineral analysis (BOENIGK 1987, HAGEDORN 2004). According to the few yet available chronostratigraphic markers, the boundary is considered diachronic. The markers come from the non-alpine sediments below. They are located close to the present river Rhine and 30 km apart: In Sessenheim (25 km north of Strasbourg) the transition is identified still within the uppermost Neogene (BOENIGK 1987), in Marlen (5 km south of Strasbourg) at least post-Tiglian (BROST & ELLWANGER 1991, ELLWANGER 2003) or even post-Bavelian (BLUDAU 1998). In the far downriver Lower Rhine area, the provenance transition has been frequently detected as heavy mineral signal within the Pliocene Reuver Clay (BOENIGK 1970, 1987, BOENIGK & FRECHEN 2006, KEMNA 2008, KEMNA & WESTERHOFF 2007, WESTERHOFF 2004, 2008).

In this paper we use the lithostratigraphic scheme outlined below (chapter 3.3), which has been created some years ago to avoid confusion. We also use chronostratigraphic terms, but only when referring to chronostratigraphy and only based on chronostratigraphic data.

3.2 Sediment evaluation

In Applied Geology, the Quaternary sediment succession of the URG is up to now described as an alternation of units of coarse and fine-grained sediments (Kieslager, Zwischenhorizonte, BARTZ 1982). These units are widely used in e.g. in hydrogeology, serving as aquifer resp. aquitard units. Most data in archives fit in this routine.

In this paper we describe the succession as lithofacies resp. lithofacies associations and identify genetic units (FIEBIG 1999). We attempt to give a first interpretation of sedimentary cycles of various orders, stacking patterns and sedimentary control in different environments. Dealing with borehole cores of often quite coarse grained sediments, this is based primarily on first analysis of grain sizes, stratification, petrography, heavy minerals, carbonate content, pollen, molluscs, ichnofossils etc. Studies on stratification and textures are limited due to weak consolidation of the material.

We distinguish gravel, sand and fines (silt and clay) according to DIN 4022 and lithofacies terms according to MIALL (1985, 1996). The succession is related to trends of the relative base level and the sediment input (a/s -ratio). They again depend on what is considered to ultimately control the sedimentary system: tectonic uplift and subsidence, compaction, but also effects of climate as intensified mechanical erosion.

The base level concept as applied here goes back to basic studies of CROSS et al. (1993) and of CROSS & LESSENGER (1998). They combine WALTHER's law with sediment volume partitioning. This leads to a simplified way to regard sedimentary cycles as a function of accommodation space vs. sediment supply (a/s -ratio). In particular the grain size variations and changes in the recorded environment are interpreted, e.g. sequences with dominant pedogenesis represent very low input and high accommodation space, i.e. high a/s -ratio; in contrast to sequences with dominant sand preservation in the fluvial system where the fines are reworked, i.e. low a/s -ratio. The changes of increase and

decrease of the a/s-ratio are then used to identify the sequence stratigraphic frame which can be used for correlation.

3.3 Lithostratigraphy

The sedimentary succession is put in the frame of several lithostratigraphic units which were lately defined as formations and sub-formations (Fm, Sfm, SYMBOLSCHLÜSSEL GEOLOGIE BADEN-WÜRTTEMBERG 2007). They avoid the former mix-up with chronostratigraphy but, at the same time, try to continue to use major observations formerly registered. Primarily this concerns two observations: in the lower part of the succession the provenance change related to the alpine Rhine; and in the upper part (only northern URG) a specific boundary, revealing a wide-spread, externally controlled and abrupt change of lithofacies:

- Mannheim-Formation (upper unit), series of two or three sand cycles, low preservation potential of fines; topmost unit with increased ratio of local sand and gravel;
- Boundary: abrupt fine-coarse transition
- Kurpfalz-Formation (middle unit), series with basal coarse-fine-cycles of Rhenish provenance, followed by a sand-dominated section, and finally a widely recognized subunit of fines on top;
- Boundary: change of provenance;
- Iffezheim-Formation (lower unit), series of sand-to-mud cycles of local origin, usually including strongly weathered sediments.

The above patterns reflect the lithofacies of the central HDB. They are modified by local input, in this case of the Neckar close to Heidelberg. Please note that in the frame set up by these definitions,

- Provenance plays no part with regard to the upper boundary (Kurpfalz-Formation vs. Mannheim-Formation);
- Lithofacies plays no part with regard to the lower boundary (Iffezheim-Formation vs. Kurpfalz-Formation); and
- Chronostratigraphy plays no part with regard to both boundaries.

4 The Heidelberg Cores – Description and basic classifications (Litho- and Biostratigraphy)

4.1 Some general remarks

The Heidelberg research boreholes are the deepest in the project, representing the depocentre on the eastern margin of the HDB. They are located just north of the Campus of Heidelberg University, still close to the mouth of the River Neckar in downtown Heidelberg, and situated on the alluvial fan of the Neckar. For technical reasons, a first borehole was cored to 190 m, a second borehole from 184 to 500 m (UniNord 1+2). The two sites are only 300 m apart and are easily correlated (Fig. 2). Their combined depth of 500 m exhibits Quaternary sediments down to ~ 500 m which is more than originally predicted (before drilling the chronostratigraphic Plio- Pleistocene transition was expected around 400 m, i.e. we intended to recover 400 m cores of Quaternary sediments, plus 100 m of Pliocene sediments).

We present a condensed lithostratigraphic description and classification of the sediments, including some remarks on

- sediment provenance, derived from pebble and heavy minerals analysis;
- the sedimentary environment;
- the few already available biostratigraphic markers, derived from pollen analysis, that are used to set up a chronostratigraphic frame;
- and depositional trends.

Following the conventional technique of core documentation, the major units are in order from top to bottom; but subunits in upward direction. A detailed description including further data will be published later in a special volume of LGRB Informationen (Regierungspräsidium Freiburg, www.rp-freiburg.de).

The sediment succession is provisionally subdivided in three large units according to the above lithostratigraphic terms. At UniNord they were identified as follows:

- Mannheim-Formation from 56 to 0 m; as a series dominated by coarse gravels, with layers of diamicton and fines, including a well preserved fossil soil;

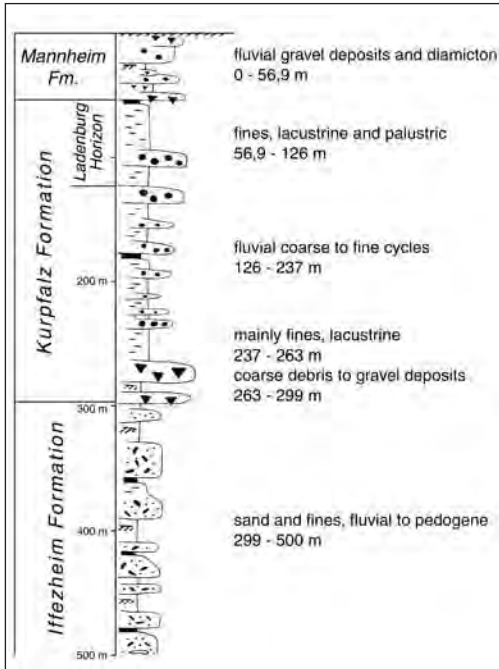


Fig 2: Overview of the sediment succession of the Heidelberg deep down to 500 m, comprised from the UniNord boreholes of 2006 (interval from 0 to 190 m) and of 2008 (interval from 184 m to 500 m). Lithostratigraphic frame, major lithologies and sedimentary environments. Legend cf. Fig. 3.

Abb. 2: Übersicht der Sediment-Abfolge im Heidelberger Loch bis 500 m Tiefe, zusammengesetzt aus den Bohrungen UniNord aus 2006 (Abschnitt von 0 bis 190 m) und aus 2008 (Abschnitt von 184 m bis 500 m). Lithostratigraphischer Rahmen, vorherrschende Lithologie und Ablagerungsmilieu. Legende vgl. Abb. 3.

- Boundary: abrupt transition of fine-coarse sedimentation
- Kurpfalz-Formation from 299 to 56 m; as a series with coarse debris at the basis (sub-unit K1); a two-parted main section with coarse debris and gravel, sand and fines in the middle part; and a fine sediment dominated section on top;
- Boundary: change of provenance and abrupt transition of fine-coarse sedimentation
- Iffezheim-Formation from >500 to 299 m; as a cyclic alternation of white-sand to clay and dark sand, altogether of non-alpine origin.

4.2 Mannheim Formation at UniNord

The Mannheim-Formation at UniNord covers the interval from 0 to 56 m. The sediments are composed of about 75 % coarse debris and gravel, 10 % sand, 5 % fines, and 10 % diamicton. Fluvial gravels dominate. However, the succession begins and ends with a layer of coarse debris, the upper one resembling the alluvial fan of the Neckar which presently undergoes erosion and terrace formation. Within the gravels there are insertions dominated by diamicton; one of them grading into fines with a fossil soil.

The succession is stratified as follows (cf. Fig. 3):

- 0 – 12 m coarse debris with boulders, gravel and layers of sand;
- 12 – 26 m fluvial gravel with some layers of sand;
- 26 – 34 m diamicton with sand cover;
- 34 – 38 m diamicton grading into mud with fossil soil ;
- 38 – 46 m fluvial gravel;
- 46 – 48 m diamicton;
- 48 – 55 m fluvial gravel;
- 55 – 56 m coarse debris and gravel;

Provenance: The petrographic spectra of the gravels reflect the up-river catchment of the present Neckar valley passing through the Odenwald highlands. There are large components of the nearby Buntsandstein which are coarse and poorly rounded. The Muschelkalk components are usually finer and better rounded; however, one boulder occurs at the basis of the unit at 56m. Some small components come from the far distant Neckar catchment, i.e. the Jurassic of the Swabian Alb. The crystalline basement with the nearby major exposure below Heidelberg castle is only poorly represented. – Most sand and fines are also of Neckar provenance, with the exception of several sand-rich strata resembling traces of Rhenish provenance by their instable heavy mineral composition. They are very well sorted and therefore supposed to come from reworked eolian input, alike to the nearby dunes, and may represent elder phases of cold climate (26 – 29 m).

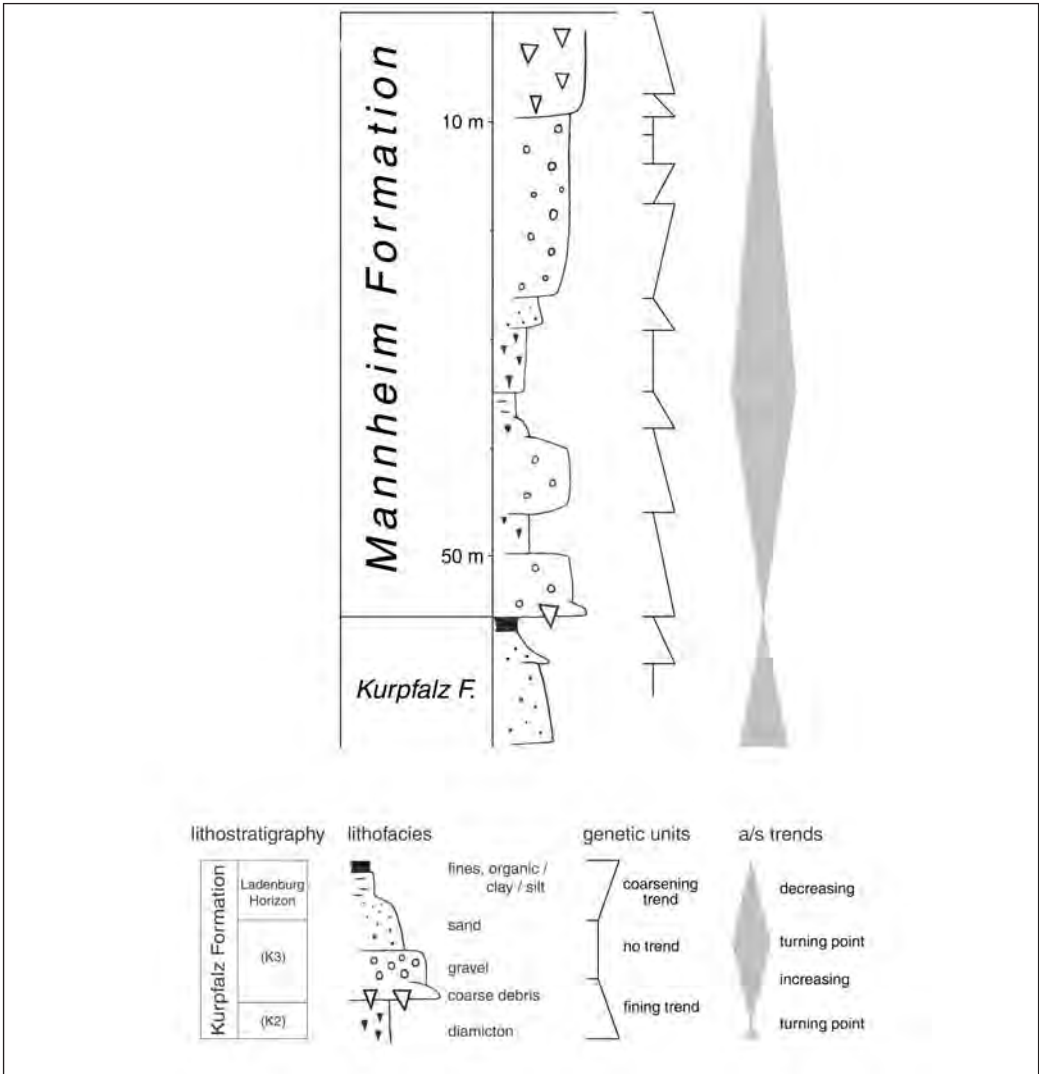


Fig. 3: The sediment succession of the Mannheim-Formation in the Heidelberg UniNord research borehole (location Fig. 1).

Centre: Stratigraphic succession as Lithofacies log (schematic), genetic units; Left: Lithostratigraphic interpretation; Right: Sequence stratigraphic trends (increasing and decreasing a/s-ratio).

The succession is dominated by coarse debris flows and proximal fluvial gravels, but also includes insertions of matrix-rich diamicton and fine sediments. – Further details on the stratigraphic succession, provenance, chronostratigraphic markers and sedimentary environment see text.

Abb. 3: Die Sedimentfolge der Mannheim-Formation aus der Forschungsbohrung Heidelberg UniNord (Lokation Abb. 1):

Mitte: Stratigraphische Abfolge als schematisches Lithfazieslog, genetische Einheiten; Links: Lithostratigraphische Interpretation; Rechts: Sequenzstratigraphische Trends (zu- und abnehmendes a/s-Verhältnis).

Die Abfolge wird beherrscht von großen Debris-Flows und proximalen Flussschottern; sie enthält auch Einschaltungen von Matrix-reichen Diamikten und Feinsedimenten. – Weitere Angaben zur Schichtenfolge, Provenienz, chronostratigraphischen Marken und Ablagerungsmilieu im Text.

Sedimentation: The depocentre of the HDB corresponds with the alluvial fan of the Neckar. The genetic units in intervals with coarse debris and fluvial gravels match well with this proximal setting (Fig. 3). One major interval with diamiction, fines and pedogenesis, occurs all over the depocentre (HGK 1999). As a result, the Mannheim-Fm shows an overall coarse – fine – coarse trend, going along with a first increasing then decreasing a/s-ratio. Any further interpretation needs a chronostratigraphic frame.

Chronostratigraphy: No chronostratigraphic markers are yet available. As the underlying unit (Ladenburg-Subformation) is supposed to date from the Cromerian, post-Cromerian ages are expected. The fossil soil at 34 m is quite well preserved, dividing the succession into an upper and a lower part. Their interpretation remains open (last and penultimate glaciation, effects of loading and compaction).

Summary and interpretation: The major part of the Mannheim-Formation at the Heidelberg UniNord borehole is built up by coarse sediments representing the proximal lithofacies of the Neckar alluvial fan. This is an input-controlled system which strongly depends on sediment supply from the nearby Odenwald highlands. As preservation in superposition is discontinuous, due to lateral shifting within the fan, the subsidence of the basin plays only a minor part.

4.3 Kurpfalz Formation at UniNord

The Kurpfalz-Formation at UniNord covers the interval from 299 to 56 m, i.e. its thickness amounts to 243 m. It is composed of 35 % debris and gravel, 20 % sand, and 45 % fines. There is a continuous background supply and preservation of fine sediments (lacustrine, fluvial overbanks), interrupted by three major intervals with coarse debris and gravel input (fluvial bedload, mass flow). Accordingly it is subdivided into lithostratigraphic subunits. The uppermost subunit (interval 56 to 126 m, Ladenburg-Subformation) is dominated by

fine sediments. The subunit from 126 to 237 m (subunit K3) subsumes two pulses of coarse input (intervals 126 – 168 m and 189 to 206 m), but throughout with layers of fines and sand. The next subunit from 237 to 263 m is again wholly dominated by fine sediments (K2). Some genetic units grade into dark fines (organic), others into clay with evidence of pedogenesis and ichnofossils. The lowermost subunit (263 – 299 m, K1) is dominated by massive coarse debris (263 – 288 m) which represents the termination of a coarsening-up trend that begins with red sands below the Formation boundary at 316 m. The definition of the lower boundary of the Kurpfalz-Formation is, as indicated above, not a matter of the local cycle architecture but of provenance. The lowermost signal of instable „alpine“ heavy minerals has been identified at 292 m; we suggest the Formation-basis at 299 m.

The sediment succession of the Kurpfalz-Formation is stratified as follows (Fig. 4):

Ladenburg-Subformation from 56–126 m = 70 m (20 % gravel, 10 % sand, 70 % fines)

- 56 – 67 m graded sand to fines and fossil peat, fluvial, some sand from eolian input and fluvially reworked;
- 67 – 74 m massive fines, lacustrine;
- 74 – 80 m graded sand to fines;
- 80 – 83 m silt and clay, ichnofossils, fossil soil, fluvial environment;
- 83 – 89 m massive fines, lacustrine;
- 89 – 92 m gravely sands, ?diamiction;
- 92 – 101 m gravel, some sand from eolian input and fluvially reworked, ?diamiction (no or poor cores);
- 101 – 103 m fines and sand, gravely, ?diamiction (poor cores);
- 103 – 109 m coarse debris, gravel, sand, ?diamiction (poor cores);
- 109 – 126 m massive fines, at the basis with coarse components, lacustrine;

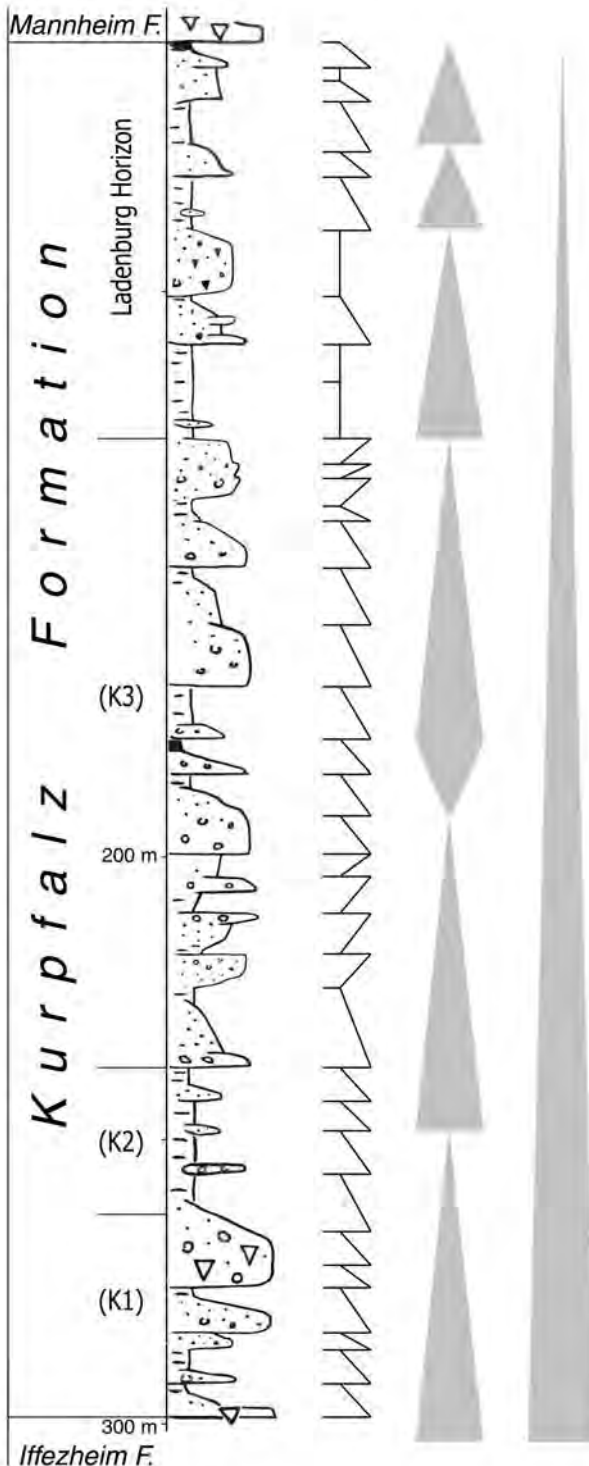


Fig 4: The sediment succession of the Kurpfalz-Formation in the Heidelberg UniNord research borehole (location Fig. 1).

Centre: Stratigraphic succession as Lithofacies log (schematic), genetic units;

Left: Lithostratigraphic interpretation (Formation and sub-units);

Right: Medium and large scale sequence stratigraphic trends.

The succession begins with sediments reflecting a lacustrine environment, with and without coarse mass flow insertions (K1, K2). It grades up into fluvial overbanks and gravels (lower K3) and a prograding system of fluvial gravels (upper K3). After abrupt change, the pattern is twice repeated on smaller scale (Ladenburg-Horizon). – Further details on the stratigraphic succession, provenance, chronostratigraphic markers and sedimentary environment see text. Legend cf. Fig. 3.

Abb. 4: Die Sedimentfolge der Kurpfalz-Formation aus der Forschungsbohrung Heidelberg UniNord (Lokation Abb. 1):

Mitte: Stratigraphische Abfolge als schematisches Lithfazieslog, genetische Einheiten

Links: Lithostratigraphische Interpretation (Formation und Untereinheiten);

Rechts: Sequenzstratigraphische Trends mittlerer und großer Ordnung.

Die Abfolge beginnt mit lakustrinen Sedimenten, zunächst mit, dann ohne Grobsediment-Einschaltungen (K1, K2). Darüber fluviale Overbank-Sedimente und Schotter (K3 unterer Teil) und schließlich ein progradierendes System fluvialer Schotter (K3 oberer Teil). Nach einem abrupten Wechsel wiederholt sich dasselbe Muster in kleinerer Ordnung zwei Mal (Ladenburg-Horizon). – Weitere Angaben zur Schichtenfolge, Provenienz, chronostratigraphischen Marken und Ablagerungsmilieu im Text. Legende vgl. Abb. 3.

Section (K3) from 126 – 237 m = 111 m
(35 % gravel, 30 % sand, 35 % fines)

- 126 – 138 m sand, gravel, debris (no or poor cores recovered);
- 138 – 152 m sand and gravel with some fines (no or poor cores recovered);
- 152 – 168 m sand and gravel (no or poor cores recovered), basis of subunit K3b;
- 168 – 179 m massive fines upon insertion of sandy gravel;
- 179 – 185 m cycle of gravel-sand-fines-peat;
- 185 – 206 m basal debris, gravel, red sand and nes, K3a;
- 206 – 211 m gravely sand and fines;
- 211 – 215 m sand;
- 215 – 217 m fines, fossil soil;
- 217 – 222 m gravel, debris;
- 222 – 227 m fines, fossil soil;
- 227 – 234 m grey and red sand;
- 234 – 237 m coarse debris grading into dark nes (organic);

Section (K2) from 237 – 263 m = 26 m
(10 % gravel, 15 % sand, 75 % fines)

- 237 – 263 m fines, sandy to muddy, with few insertions of sand (254 – 257), gravel (247 – 248) and debris (258 – 259), fossil soils;

Section (K1) from 263 – 299 m = 36 m
(60 % gravel and debris, 10 % sand, 30 % fines)

- 263 – 287 m debris of coarse gravels and some boulders, few layers of sand and fines;
- 287 – 297 m fines, silt and clay with ichnofossils, sand layers;
- 297 – 299 m debris of coarse gravels and some boulders.

Provenance: The sediment input in the Kurpfalz-Formation of the UniNord succession is classified in sediments of Rhine origin (i.e. comprising a signal of the alpine source area),

of Neckar origin, and of very local origin. They may be subdivided in:

- (a) fines and sand of the Rhine (proven by instable heavy mineral ratio > 50 %);
- (b) local or Neckar fines and sand (instable heavy mineral ratio < 50 %);
- (c) gravel of the Neckar (pebbles of Buntsandstein, Muschelkalk and Jurassic); and
- (d) coarse debris and gravel of the nearby Odenwald (mainly Buntsandstein, some crystalline and Muschelkalk).

The sediments of the Ladenburg-Subformation belong primarily to the „fines and sands of Neckar or local origin“. There are exceptions at the top of two lacustrine intervals, where minor sand pulses of Rhine origin are identified by their instable heavy mineral content. Presently we interpret this to be fluviually reworked eolian input. – The Ladenburg-deposits of UniNord are contemporaneous with similar deposits all over the HDB which are usually of Rhine origin. They also correlate with the upstream buried valley infill at the discovery site of the *Homo heidelbergensis* located near to the village of Mauer, some 15 km southeast of UniNord (LÖSCHER 1996). From this it follows that the sediment of this subunit covered the whole HDB, and extended beyond the Eastern Boundary Fault (EBF) of the URG. I.e. the nearby Neckar valley within the Odenwald highlands is also, as a drowned valley, part of the basin.

The subunits K1 and the lower part of K3 show strong evidence of periodical fluvial input from the alpine Rhine, with continuous input from the Neckar upheld. In K1, the arrival of the Rhine and the preservation of fines even when alternating with coarse local sediments indicate a strongly increasing a/s-ratio. In K2, only sand on fines of non-Rhine origin are yet identified. The K3 succession begins with an alternation of local and Rhine input. Then the local input is gradually replaced by supply from the Neckar, and still further up the input from the Rhine terminates. From 190 m upwards only sediments of Neckar provenance are left.

The three intervals of coarse debris and gravel (subunits K1 and K3) refer to expanding erosion into the Odenwald, probably due to uplift.

There is always an overall dominance of coarse Buntsandstein, but the ratio of the supplementary components varies (Muschelkalk, crystalline). The first coarse pulse (299 – 297 m, basis of K1) contains much local crystalline (Heidelberg Granite). The main pulse (287 – 263 m) is mainly Buntsandstein, but with an upward increasing ratio of Muschelkalk; i.e. the sediment source is first „downtown Heidelberg“, then expanding towards the Muschelkalk scarp. – Two coarse intervals of fluvial gravels follow (206 – 189 m and 168 – 126 m, subunit K3). Within the latter a first small then increasing ratio of Jurassic is present, and there are some layers where the Muschelkalk components dominate. This is also characteristic regarding some correlative gravelly insertions in the succession of the Viernheim sister drilling (interval 180 to 80 m, HOSELMANN 2008).

Sedimentation: The overall fluvial evolution of the UniNord succession is interrupted in the Kurpfalz-Formation by intervals of lacustrine conditions and by intervals of strong supply of mass flows. Our interpretation as a lacustrine environment is based upon the abrupt occurrence of coarse-grained debris flows within the fine-grained background sedimentation. In some parts of the succession, even matrix supported mass flow deposits including boulders and cobbles occur (high density mass flows). The abrupt alternation of mass flow- and background-sedimentation is a clear indication for lacustrine deposition in a lake system with a minimal water depth of several meters.

The first lacustrine interval is at the basis of the Formation where mass flow deposits of coarse debris are embedded in fines, with almost no transition (subunit K1). It carries on in subunit K2 with few coarse layers in the fines. Eventually the lacustrine sedimentation terminates, first indicated by fossil soils in the fines (transition of subunits K2 to K3), then by cycles of fluvial gravels to fines (subunit K3). The lower gravel seems to represent a local event; the upper reflects a long and overall coarsening-up series of a prograding fluvial system. – The transition to follow, from fluvial gravels to

the fines of the Ladenburg-Subformation, is again very abrupt. The fines are massive; again with insertions of mass flow deposits (here the components are well rounded i.e. reworked from the gravels). This again represents a lacustrine environment. Towards the top of these fines, the environment migrates back towards fluvial, first visible in traces of plant roots and ichnofossils, still within the fines, then by re-establishing the prograding fluvial system (lacustrine-to-fluvial cycle from 126 to 89 m = 37 m). In the upper part of the Ladenburg-Subformation, two more lacustrine to fluvial transitions are identified (89 – 74 m = 15 m; and 74 – 56 m = 18 m). They range from massive fines up to graded sands, i.e. they comprise no gravelly sediments. Further up, the lacustrine to fluvial cyclicity is drowned by the input of the Mannheim-Formation.

The Kurpfalz-Formation shows an overall decreasing a/s-ratio throughout the succession, with various symmetrical and asymmetrical cycles of lower order (Fig. 4). The maximum accommodation space is given in the first lacustrine cycle (K1, K2), which hosts not only coarse local debris but also alpine sands of the Rhine. The alpine influence is then pushed back by prograding coarse-grained fluvial Neckar deposits (K3).

Chronostratigraphy: Some biostratigraphic markers based on pollen analysis are already available. They are preliminarily interpreted.

- At 57 – 70 m and at 82 – 85 m, „coniferous forest“: Interpretation: cool to cold „interstadial“ climate.
- At 81 – 82 m and at 87 m, „typical middle Pleistocene pollen spectra“ but no *Fagus* and no early Pleistocene species. Interpretation: Cromerian.
- At 162 – 163 m, pollen spectra with *Pinus*, *Picea* and *Tsuga*, no *Carya*, no *Eucommia*. Interpretation: Bavelian (per se uncertain, but in the succession quite likely).
- At 180 – 208 m, pollen spectra with *Tsuga* and *Pterocarya*. Interpretation: Waalian (certain, HAHNE, ELLWANGER & STRITZKE 2008).

- At 212 – 249 m „coniferous forest“: Interpretation: rather cold climate, may match with the Eburon cold period.

The Cromerian age of the Ladenburg-Subformation was first published by ENGESSER & MÜNZING (1991); also the „Maurer Sande“ are of Cromerian age (LÖSCHER 1996).

Summary and interpretation of the Kurpfalz-Formation: The succession of the Kurpfalz-Formation at UniNord, within its upper „sequence“-boundary and its lower „provenance“-boundary, comprises one overall large scale cycle, from a basal lacustrine environment up to a large and input controlled subunit of prograding fluvial gravels. The fines to follow are, although in parts lacustrine and related to a final pulse of subsidence, only of local importance. We suggest this has to do with the pattern of Rhine sediments through time in the HDB. This is confirmed by the sister boreholes of the Heidelberg project at Ludwigshafen and Viernheim, where, as it seems, the Rhine signal comes, in each case, at a different chronostratigraphic level.

- In Heidelberg, the overall depocentre of the HDB, the Rhine signal begins at 299 m (Eburonian) and ends at 190 m (Waalian);
- In Ludwigshafen at the western margin of the HDB, the Rhine signal begins at 177 m close to the Pliocene-Pleistocene transition (WEIDENFELLER & KÄRCHER 2008 and WEIDENFELLER & KNIPPING 2008), then comes to a standstill (increase of local sediments with only stable heavy minerals) at 137 – 127 m and 109 – 97 m (Waalian);
- In Viernheim at the geographic centre of the HDB, the Rhine signal begins at 220 m (HOSELMANN 2008, KNIPPING 2002, 2008). As discussed above we suggest correlating a subunit of prograding gravels from Heidelberg (Bavelian) with the 80 – 180 m interval at Viernheim. This leaves 40 m with little evidence of any greater hiatus. From all this we suggest the provenance change to happen in the Waalian.

The above may be interpreted as „alpine“ Rhine sediments migrating in three steps through what later became the HDB: This story

begins in Ludwigshafen close to the Pliocene-Pleistocene transition; then it continues in Heidelberg in the Eburonian; and finally comes to Viernheim in the Waalian.

The subunit of prograding gravels (K3, Bavelian) appears to be the first unit covering the HDB in its present extent as a whole, as do the above units of the Ladenburg-Subformation and the Mannheim-Formation. Below the gravels (K3) there is a patchwork of local units which are difficult to correlate. Here the units including alpine Rhine sediments may be of small extend. Their west-to-east migration (from Ludwigshafen to Heidelberg) seems to be subsidence-controlled, as a result of the Halfgraben architecture. Their return back west to Viernheim is related to Neckar input. The upper units covering the entire HDB are related to input pulses of Rhine and Neckar which include potentially increased sediment volumes during the Pleistocene glaciations.

All sediments below the „alpine“ Rhine, regardless of their chronostratigraphic position, are not part of the Kurpfalz-Formation. They belong, to the Iffezheim-Formation.

4.4. Iffezheim Formation at UniNord

The Iffezheim-Formation at UniNord covers the interval from >500 to 299 m, i.e. >202 m. It is a cyclic alternation of fluvial deposits with an average composition of 55 % sand, and 45 % fines. The succession is subdivided in several lithostratigraphic subunits, according to lithofacies variations. They are dominated by sand at 316 to 382 m (I5); by fine sediments at 382 to 411 m (I4); again by sand at 411 to 452 m (I3); fine sediments at 452 to 467 (I2); and finally by sand at 467 to >500 m (I1). The uppermost subunit at 299 to 316 m (I6) resembles the transition to the lacustrine environment and to the onset of coarse local debris of subunit (K1).

The main fluvial lithofacies are:

- Grey, coarse to medium-grained sands. They contain only few gravely components, the small pebbles being well rounded and often with weathering halos. Quite frequently there are other coarse components,

as pieces of wood of various size (in the cores with diameters up to 25 cm), but also reworked pieces of fine sediment, ranging from small rounded pieces up to large strata. Their original stratification may be preserved, including organic-rich laminae or even peat. They were probably in a frozen state during transport.

- White, fine to medium-grained sands, usually very well sorted. They either alternate with the grey sand lithofacies, or grade into green silt with sandy laminations, and finally into the silt and clay cycles.
- Clay-rich overbank fines, forming sequences of green and sandy silt grading into clay-rich silt up to almost pure clay, usually speckled with brownish (wet) and reddish (dry) colours resembling pedogenesis. Colours are intense, apart from the transition to the Kurpfalz-Formation where they become dark and gloomy. The sequences are rich in ichnofossils, in some layers also in authigenic grains (authigenic siderite, Dr. Martin, Freiburg). Some are fractured due to diagenetic compaction.

The sediment succession of the Iffezheim-Formation is stratified as follows (cf. Fig. 5):

Section (I6) from 299 to 316 m = 17 m
(55 % sand and 45 % fines)

- 299 – 305 m dark fines, sandy silt to clay;
- 305 – 316 m massive dark grey sand and graded dark red sand;

Section (I5) from 316 – 382 m = 66 m
(65 % sand and 35 % fines)

- 316 – 323 m fines (sandy silt to clay with organic-rich top);
- 323 – 329 m brightly coloured fines (sandy silt to clay);
- 329 – 340 m alternation of
 - grey sand with reworked wood and fines, and of
 - white sand with kaolin;
- 340 – 342 m brightly coloured silt and clay, ichnofossils;
- 342 – 351 m grey sand with reworked wood and fines;

- 351 – 382 m grey sand with reworked wood and fines;

Section (I4) from 382 – 411 m = 29 m
(10 % sand and 90 % fines)

- 382 – 399 m fines, green silt to coloured clay with ichnofossils and brown and red fossil soils;
- 399 – 402 m white sand and grey silt;
- 402 – 411 m brightly coloured fines, green silt to coloured clay with ichnofossils and brown and red fossil soils;

Section (I3) from 411 – 451 m = 40 m
(70 % sand and 30 % fines)

- 411 – 421 m grey sand with reworked wood and fines, grading into white sand;
- 421 – 425 m white sand grading into silt and clay with ichnofossils;
- 425 – 440 m alternation of
 - grey sand with reworked wood and fines, and of
 - white sand with kaolin matrix;
- 440 – 443 m brightly coloured silt and clay with ichnofossils;
- 443 – 451 m alternation of
 - grey sand with reworked wood and fines, and of
 - white sand with kaolin matrix (445 – 447);
 - fines, laminated with white sand (443 – 445);

Section (I2) from 451 – 467 m = 16 m
(almost 100 % fines)

- 451 – 467 m brightly coloured fines, green silt to coloured clay with ichnofossils and brown and red fossil soils;

Section (I1) from 467 – 500 m = 33 m
(80 % sand and 20 % fines)

- 467 – >500 m grey sand with reworked wood and fines;

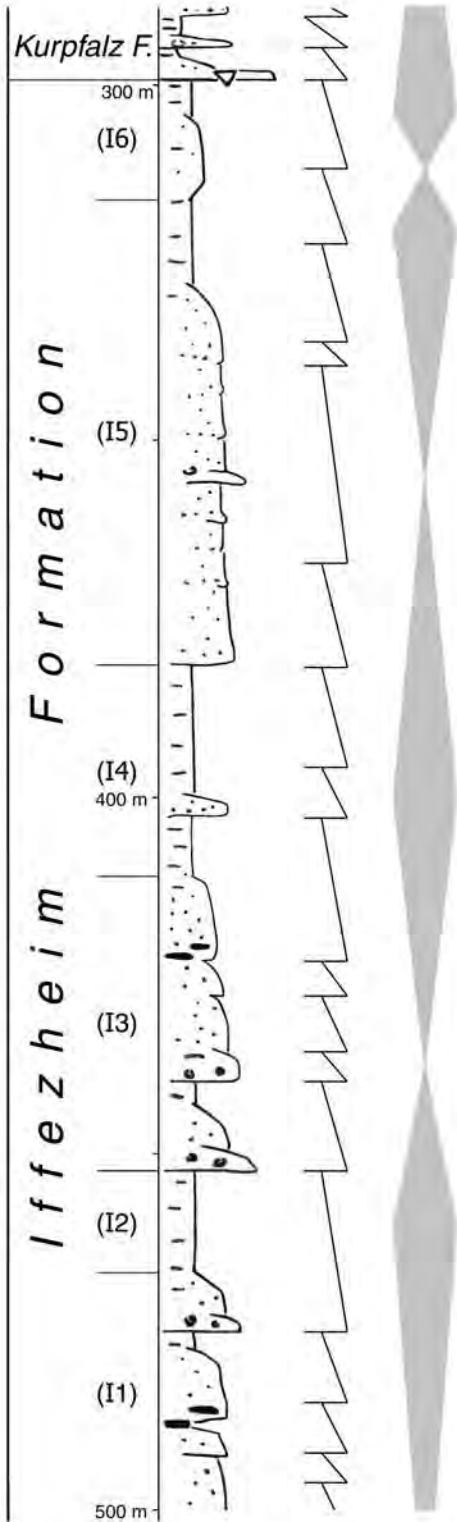


Fig. 5: The sediment succession of the Iffezheim-Formation in the Heidelberg UniNord research bore-hole (location Fig. 1).

- Centre: Stratigraphic succession as Lithofacies log (schematic), genetic units;
- Left: Lithostratigraphic interpretation (Formation and sub-units);
- Right: Sequence stratigraphic trends (increasing and decreasing a/s-ratio).

The succession (I1 - I5) is composed of alternating fluvial bedload sands and overbank silt and clay. The sands contain large pieces of reworked overbank fines, the silt and clays are frequently altered by ichnofossils and pedogenesis. The uppermost unit (I6) includes proximal local sand insertions reflecting the beginning of local mass flow as in unit K1. – Further details on the stratigraphic succession, provenance, chronostratigraphic markers and sedimentary environment see text. Legend cf. Fig. 3.

Abb. 5: Die Sedimentfolge der Iffezheim-Formation aus der Forschungsbohrung Heidelberg UniNord (Lokation Abb. 1):

- Mitte: Stratigraphische Abfolge als schematisches Lithofazieslog, genetische Einheiten
- Links: Lithostratigraphische Interpretation (Formation und Untereinheiten);
- Rechts: Sequenzstratigraphische Trends (zu- und abnehmendes a/s-Verhältnis).

Die Abfolge (I1 - I5) besteht aus fluvialen Sanden (bedload) und schluffig-tonigen Overbank-Schichten. In den Sanden sind große Feinsedimentstücke aufgearbeitet, während die Overbanks durch Spurenfossilien und Bodenbildung verändert sind. In der obersten Einheit (I6) sind proximale Lokalsande eingeschaltet, die zu den Massflows der darüber folgenden Einheit K1 überleiten. – Weitere Angaben zur Schichtenfolge, Provenienz, chronostratigraphischen Marken und Ablagerungsmilieu im Text. Legende vgl. Abb. 3.

Provenance: The „local“ i.e. non-alpine origin of the sediments determines their allocation to the Iffezheim-Formation. This is primarily based on heavy mineral analysis. Many of the few coarser components are of Buntsandstein origin, as are several layers of red sands. – The provenance of the other sediments remains uncertain. The well sorted white sands are probably far transported, but this may be an inherited feature if they are reworked from nearby Neogene sands. The grey sands are related to various sources, including crystalline and Buntsandstein (Odenwald? Black Forest?). They seem confined to a fluvial system close to the eastern margin of the URG. The source of the silt and clay is again undetermined, probably nearby Neogene.

Sedimentation: By alternation of the grey sand lithofacies and the silt-to-clay lithofacies, several genetic units are stacked to symmetrical cycles of increasing and decreasing a/s-ratios. Within the fines, the turning points often coincide with intensive pedogenesis i.e. almost no input; within the sands they coincide with sections where large pieces of reworked fines indicate a/s minima. These bedload-dominated units represent the greater part of the succession. The preserved cycles are in the 10 to 50 m order. – The minima of accommodation space are likely to be related with increasing sediment supply, maybe due increased sediment volumes as a result of colder climate (some of the reworked fines are still stratified, they may have been frozen when redeposited). As opposed to this the sediment input is generally much reduced in silt-clay sections, with ichnofossils and traces of more or less reworked fossil soils. Preservation is primarily related to local subsidence of the depocentre, but differentiated uplift within and at the northern margin of the URG may also be involved.

Chronostratigraphy: Some biostratigraphic markers based on pollen analysis are already available. They are preliminarily interpreted.

- At 312 m, „open coniferous forest with herbs“. Interpretation: cool to cold climate

(per se indifferent, but in the succession most likely Eburonian).

- At 316 m, „*Pinus*-dominated coniferous forest“. Interpretation: cool to cold climate, could match with the Eburon cold period.
- At 358 – 359 m, reworked strata. They host a pollen spectrum with > 30 % „tertiary taxa“ immediately overlying a typical „early Pleistocene pollen spectra“ with *Fagus* but no *Tsuga*. Interpretation: reworked Reuverian (Reuverian A) overlying reworked late Tiglian. Deposition age: late Tiglian to early Eburon.
- At 417 – 500 m (lowest sample at 499.94 m): several typical „early Pleistocene pollen spectra“ with *Fagus* and tertiary taxa increasing downward, no *Tsuga*. Interpretation: Tiglian (certain) in stratigraphic superposition.
- At 481 m: „*Pinus*-dominated coniferous forest“. Interpretation: cooler climatic period within the Tiglian (possibly Tiglian B, sensu ZAGWIJN 1963).
- There are no hints for in-situ sediments from the Pretiglian or Reuverian period. The reworked sample at 358 m illustrates the pollen spectra to be expected from in-situ Reuverian.

Summary and interpretation of the cored part of the Iffezheim-Formation: According to the definition outlined above, the succession is not influenced by instable heavy minerals of the alpine Rhine. The above pollen markers show that the succession terminates well above the base of the Quaternary, i.e. in a time slice when the course of the alpine Rhine was already bound within the URG (in the western HDB proved by WEIDENFELLER & KNIPPING 2008). At the same time, maximum sedimentation rates occur on the eastern side at Heidelberg UniNord. All this requires a two-partition of the HDB, with a western basin hosting the alpine Rhine, and an eastern basin with maximum sedimentation rates. The transition between the basins is suggested to be represented by the Viernheim drilling where the Rhine sediments arrived latest.

4.5 Below 500 m

As the cored UniNord boreholes end at 500 m still within the Tiglian, the lower part of the Tiglian, the Pretiglian, and the transition into the uppermost Neogene unit i.e. the Reuverian remain uncored. In order to estimate compaction and tectonic subsidence rates, serving as accommodation space of the cored units, some basic informations on the deeper subsurface are introduced here. They rely on three available data sets: (a) the sediment log of the „Radium Sol“ borehole in downtown Heidelberg, approx. 1 km south of the UniNord location; (b) the „vertical seismic profiling“ (VSP) log which was recorded by the Leibniz Institute for Applied Geophysics in the UniNord 1 borehole; and (c) the sediment log of the geothermal flush borehole from Weinheim, approx. 14 km north of UniNord.

- (a) The Radium Sol drilling was finished in the early 1920ies (SALOMON 1927, BARTZ 1951, FEZER 1998). Its overall cyclicity follows a coarse-fine-coarse pattern, with
- 0 ~ 350 m coarse and fine grained sediments;
 - 350 ~ 700 m only fine grained sediments;
 - 700 ~ 1000 m coarse and fine grained sediments.
- (b) The VSP log was recorded as part of the geophysical downhole logging program (data recorded down to 181 m, velocity information to 160 m). It is plotted as corridor stack down to 1000 m (BUNESS, pers. comm.). We do not interpret individual reflections but evaluate their frequency. There are many strong reflections in the upper part and again several though weaker reflections in the lower part, but almost none in a middle interval from 350 down to 700 m. This is unlikely in a succession of alternating coarse and fine sediments. We conclude fine sediment dominance down to 770 m.
- (c) Also at Weinheim where two boreholes were flushed down to ~ 1000 m, a long interval of fine sediment domination has been detected down to 700 m.

We conclude that, at UniNord, the high ratio of fine sediment will continue or even increase in the interval 500 to 700 m, estimated 50 % of fines or more. It will then again decrease in the 700 to 1000 m interval where coarse and fine sediments alternate. Assuming constant sedimentation rates as derived below, the transition from the Neogene Reuverian unit to the Pleistocene Pre-Tiglian stage is now predicted close to or below 600 m.

5 The Heidelberg Boreholes – Synopsis, Conclusions, Outlook

5.1 Sedimentation rates, Accommodation Space and Subsidence

The patterns of Lithostratigraphy and Chronostratigraphy are compared in Table 1. Basically the lithostratigraphic boundaries are controlled by tectonics whereas Chronostratigraphy refers to climate. They are identical where the lithostratigraphy is based on the local lithofacies patterns, i.e. the boundaries of the Ladenburg-Sfm and the Mannheim-Fm.

Table 1 also includes „absolute“ ages (STD 2002, OGG, OGG & GRADSTEIN 2008). They serve as a geochronologic frame to estimate average sedimentation rates for the units (Tab. 2, Fig. 6). They are, as yet, uncorrected, but may be used to calculate the non-tectonic i.e. compaction part of the subsidence.

5.2 Input and uplift

The local aspect: The relatively rapid increase of sediment thickness during Eburonian time is associated with coarse, clastic deposits. Clastic fragments are derived from Triassic sedimentary rocks and the Heidelberg granite and associated crystalline rocks of the Variscan basement. This sediment composition is interpreted to reflect the synchronous uplift of the adjacent rift shoulder and the initiation or reactivation of faults in the Heidelberg town area, which trend both subparallel with and at a high angle to the major, eastern rift bounding fault. Activities at these faults led to the exposition of the crystal-

Table 1: Lithostratigraphy and Chronostratigraphy of the sediment succession of the borehole Heidelberg UniNord. The lithostratigraphic boundaries are controlled by tectonics (cf. Tab. 2); the chronostratigraphic stages refer to the alpine and north European glaciations and to the pollen-based biostratigraphy of NW-Europe. Sediment thicknesses are uncorrected.

Tab. 1: Lithostratigraphie und Chronostratigraphie der Sedimentfolge aus der Bohrung Heidelberg UniNord. Die lithostratigraphischen Grenzen sind tektonisch gesteuert (vgl. Tab. 2); die chronostratigraphischen Stufen beziehen sich auf die alpinen und nordeuropäischen Vergletscherungen und auf die pollenanalytisch begründete Biostratigraphie NW-Europas. Sedimentmächtigkeiten sind nicht korrigiert.

Lithostratigraphy			Chronostratigraphy			
Formation	Sub-formation	Interval thickness	original Interval thickness	Pollen		
Mannheim-Fm 0 – 56 m	Neckar fan	12 m	56 m		Holocene; Würm/Weichsel; Riss/Saale; Hoßkirch/Elster	
	fluvial gravel	44 m				
	Diamicton and fines, fossil soil					
	fluvial gravel, diamicton					
Kurpfalz-Fm 56 – 299 m	Ladenburg-Sfm (mass flow, fluvial, lacustrine)	70 m	70 m		Cromerian	
	(K3) two fluvial pulses 126 – 237 m	111 m	44 m	<i>Pinus, Picea</i> <i>Tsuga</i>	Bavelian 126 – 170 m	
			40 m	<i>Tsuga,</i> <i>Pterocarya</i>	Waalian 170 – 210 m	
	(K2) lacustrine 237 – 263 m	26 m	130 m	Coniferous forest 210 – 250 m	Eburonian 210 – 340 m	
	(K1) mass flow, fluvial, lacustrine, 263 – 299 m	36 m				No pollen
(I6) lacustrine fines and Sand 299 – 316 m	17 m	Open forest at 312 m				
Iffezheim-Fm 299 – 500 m	(I5) fluvial sand 316 – 382 m	66 m	160 m	early Pleistocene pollen spectra with <i>Fagus</i> and Tertiary taxa increasing downward	Tiglian 340 – >500 m	
	(I4) fluvial overbank 382 – 411 m	29 m				Coniferous forest at 316 m
	(I3) fluvial sand 411 – 451 m	40 m				
	(I2) fluvial overbank 451 – 467 m	16 m				
	(I1) fluvial sand 467 – 500 m	33 m				
Not drilled: Iffezheim-Fm 500 > 700 m	sand and fines	?	estimated 20 m			
	sand and fines	?	estimated 80 m		Praetiglian	
	mainly fines	?	estimated > 100 m		Reuverian	

Table 2: Average sedimentation rates of the chronostratigraphic units, according to uncorrected thickness and age data of Table 1. Cf. Fig. 6.

Control of the subsidence: We suggest tectonic control only if the sedimentary environment relies on tectonic subsidence. This is, presently, only the case in the lacustrine intervals covering small areas in the Heidelberg deep (Eburonian, Cromerian). In all other cases we suggest that considerable parts of the accommodation space come up from compaction of underlying fine sediments. This will be considered in future studies, including correction of the sedimentation rates. - The discussion of the potential tectonic control of the input remains unaffected.

Tab. 2: Durchschnittliche Sedimentationsraten für die chronostratigraphischen Einheiten, unter Verwendung der unkorrigierten Sedimentmächtigkeiten und Altersdaten aus Tab. 1. Vgl. Abb. 6.

Steuerung der Subsidenz: Wir diskutieren eine tektonische Steuerung nur dann, wenn das Ablagerungsmilieu nicht anders hergeleitet werden kann. Das ist bisher nur in den lakustrinen Abschnitten der Fall, die nur kleinräumlich im Heidelberger Loch nachgewiesen sind (Eburon, Cromer). Ansonsten gehen wir derzeit davon aus, dass ein erheblicher Teil des Akkomodationsraums auch durch Kompaktion der Feinsedimente aus dem Liegenden herzuleiten ist. Bei der weiteren Bearbeitung sind die Sedimentationsraten entsprechend nach oben zu korrigieren. - Die potentielle tektonische Steuerung des Inputs bleibt unberührt.

Chronostratigraphy	Sediment Thickness	Amount of fine sediments	Duration	Sedimentation rates (uncorrected)		Suggested major control of Subsidence
				Meter/ka	mm/a	
Würm, Riss, Hosskirch	56 m	< 5 m	450 ka	56 / 450	0.1	Non-tectonic
Cromerian	70 m	40 m	350 ka	70 / 350	0.2	Tectonic
Bavelian	44 m	5 m	600 ka	44 / 600	< 0.1	Non-tectonic
Waalian	40 m	15 m	200 ka	40 / 200	0.2	non-tectonic
Eburonian	130 m	70 m	200 ka	130 / 200	0.7	Tectonic
Tiglian	160 m (180 m)	60 m	600 ka	180 / 600	0.3	non-tectonic

line rocks, fragments of which are now found in the basin sequence. The subsequent period is characterized by thin sediments and may be related to a peneplanation observed at the rift shoulder. The ultimate stage, represented by the Mannheim Formation, with a more rapid increase in sediment thickness is lasting until the present and associated with renewed uplift of the rift shoulders. This uplift is accompanied by a dissection of the peneplain surface, caused by the reactivation of pre-existing faults.

The regional aspect: The first onset of alpine sedimentation in the HDB, as identified in the Ludwigshafen succession (WEIDENFELLER & KNIPPING 2008) reflects the redirection of the al-

pine Rhine into the URG, prior to the Pliocene-Pleistocene transition at ~ 2.6 Ma (e.g. HOSELMANN 2008). The redirection happens in the Mulhouse area where the first signal of alpine heavy minerals in the URG fill goes along with an abrupt coarsening of the lithofacies of alpine and local sediment. This composition has been interpreted as to reflect the starting uplift of parts of the southern Black Forest (ELLWANGER 2003), probably related to the latest folding in front of the Jura mountains (GIAMBONI et al. 2004).

Two subsequent uplift markers are identified in the UniNord succession. They are related to the coarse input at the basal Kurpfalz-Formation (Eburonian, ~ 1.7 Ma) and to the coarse input

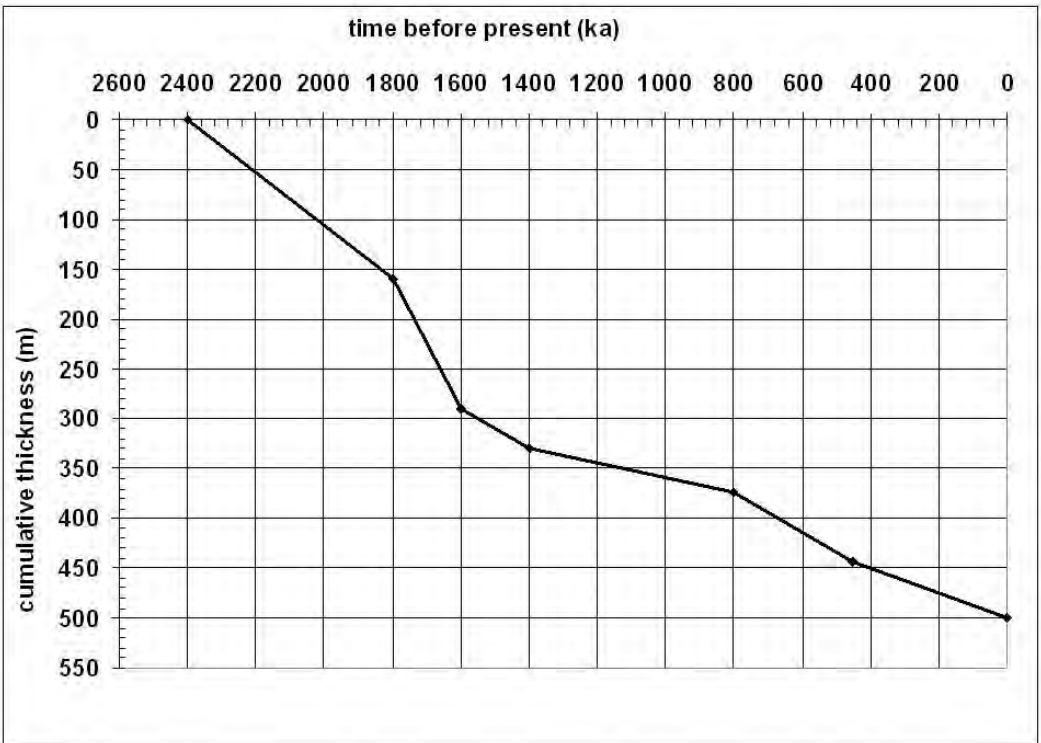


Fig. 6: Net accumulation of the sedimentary sequence through time of the Heidelberg UniNord borehole, using thickness and age data of Table 2.

Abb. 6: Sedimentmächtigkeiten der Bohrung Heidelberg UniNord gegen stratigraphische Zeitskala, unter Verwendung unkorrigierter Mächtigkeiten und Alter aus Tab. 2.

at the basal Mannheim-Formation (post-Cromerian, ~ 0.5 Ma). Both reflect uplift periods of the Odenwald. The post-Cromerian uplift is contemporaneous with the main uplift of the Rhenish Massif (cf. various studies of the lower main terrace e.g. HOSELMANN 1994).

All this leads to a scenario of Quaternary uplift = input events in cycles of ~1 Ma, proceeding from south to north:

- ~0.5 Ma Rhenish Massif plus Odenwald (?plus Black Forest);
- ~1.7 Ma Odenwald (?plus Black Forest);
- ~2.6 Ma southern Black Forest.

Each uplift event potentially provides sediment supply which will more easily be activated if

the mechanical erosion is facilitated by climate changes (e.g. VANDENBERGHE 1993, 1995, BUSSCHERS 2007). In case of the middle input event the Tiglian to Eburonian transition is proved to occur well below the coarse input is recorded. This supports the above scenario. The other events lack of chronostratigraphic data.

5.3 Outlook

The descriptions, basic data and correlative patterns presented here are all focused upon the specific situation of the Heidelberg deep i.e. the cores of the borehole of the UniNord location in Heidelberg. In the interpretative steps to follow, the controlling parameters of sedimentation will have to be more thoroughly identified,

e.g. are increasing a/s-ratios due to tectonic subsidence or to compaction within the basin, or due to uplift around the basin. This can only be accomplished if the entire HDB is regarded. Additional boreholes (e.g. HAIMBERGER, HOPPE & SCHÄFER 2005, WIRSING et al. 2007, HOSELMANN 2008, HUNZE & WONIK 2008, SIMON 2008, WEIDENFELLER & KNIPPING 2008) as well as seismic and logging data (BUNESS, GABRIEL & ELLWANGER 2008) will have to be included

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The Heidelberg Basin drilling project: Geophysical pre-site surveys

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Abstract: Currently, the Heidelberg Basin is under investigation by new cored research boreholes to enhance the understanding concerning the control on Pliocene and Quaternary sedimentation by (neo)tectonics and climate. The Heidelberg Basin is expected to serve as a key location for an improved correlation of parameters that characterise the climate evolution in North Europe and the Alpine region. The recovery of sediment successions of high temporal resolution that are complete with respect to the deposition of Pleistocene glacials and interglacials in superposition is of special importance. Prior to the new research boreholes in Viernheim and Heidelberg geophysical pre-site surveys were performed to identify borehole locations that best achieve these requirements. In the area of the Heidelberg Basin the strongest negative gravity anomaly of the entire Upper Rhine Graben is observed (apart from the Alps), hinting at anomalously thick sediment deposits. However, especially reflection seismic profiles contributed significantly to the decision about the borehole locations. In the city of Heidelberg for the first time, the depocentre of the Heidelberg Basin, as indicated by additional subsidence compared to its surroundings, was mapped. In this area, sediments dip towards the eastern margin of the Upper Rhine Graben. This is interpreted to represent a rollover structure related to the maximum subsidence of the Upper Rhine Graben in this region. At the Viernheim borehole location the seismic survey revealed several faults. Although these faults are mainly restricted to depths greater than 225 m, the borehole location was finally adjusted with respect to this information.

[Das Bohrprojekt Heidelberger Becken: Geophysikalische Voruntersuchungen]

Kurzfassung: Das Heidelberger Becken wird aktuell durch neue Kernbohrungen untersucht, um das Wissen hinsichtlich der Steuerung der pliozänen und quartären Sedimentation durch Klima und (Neo)Tektonik zu erweitern. Es wird erwartet, dass das Heidelberger Becken eine Schlüsselstelle für eine verbesserte Korrelation von Parametern darstellt, welche die Klimaentwicklung in Nordeuropa und im alpinen Raum charakterisieren. Besondere Bedeutung hat daher die Gewinnung von Sedimentsukzessionen hoher zeitlicher Auflösung, die im Hinblick auf die Ablagerung kalt- und warmzeitlicher pleistozäner Sedimente in Superposition möglichst vollständig sind. Im Vorfeld der neuen Kernbohrungen bei Viernheim und Heidelberg wurden geophysikalische Vorerkundungen durchgeführt, um Bohrlokationen zu identifizieren, die diesen Ansprüchen am besten genügen. Im Bereich Heidelberg wird die größte negative Schwereanomalie des gesamten Oberrheingrabens beobachtet (mit Ausnahme der Alpen), was auf ungewöhnlich mächtige Sedimentablagerungen hindeutet. Aber insbesondere reflexionsseismische Messungen haben zur Auswahl der Bohrpunkte beigetragen. Im Stadtbereich von Heidelberg ist zum ersten Mal das Depozentrum des Heidelberger Beckens kartiert worden, abgebildet durch eine zusätzliche Absenkung gegenüber der Umgebung. In diesem Gebiet fallen die Sedimente zum östlichen Grabenrand hin ein. Dies wird als 'Rollover' Struktur interpretiert, die in Verbindung mit der maximalen Subsidenz des Oberrheingrabens in diesem Bereich steht. An der Bohrlokation Viernheim konnten durch die Seis-

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mik zahlreiche Störungen abgebildet werden. Obwohl diese im Wesentlichen auf Tiefenbereiche größer 225 m beschränkt sind, wurde der Bohransatzpunkt schließlich aufgrund dieser Informationen gewählt.

Keywords: Heidelberg Basin, depocentre, research borehole, reflection seismic, gravity

1 Introduction

The Heidelberg Basin is located in the eastern part of the Northern Upper Rhine Graben

(URG; Fig. 1), bordered by the dominating master fault of the URG to the east. The boundary fault is assumed to extend deep into the crust, by 15 - 24 km according to MAUTHE,

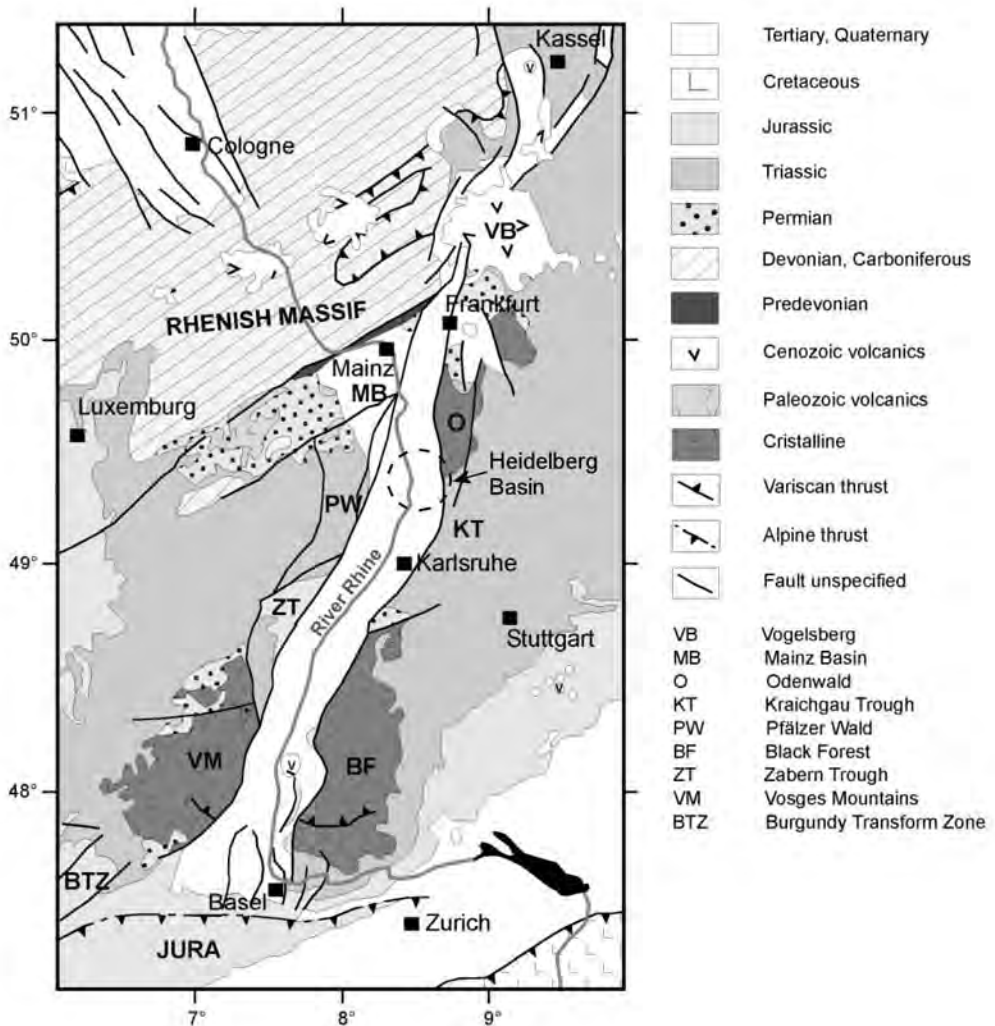


Fig. 1: Tectonic sketch map of the Upper Rhine Graben and its surroundings (after PETERS 2007). The approximate position of the Heidelberg Basin is marked by a dashed line.

Abb. 1: Tektonische Skizze des Oberrheingrabens und seiner Umgebung (nach PETERS 2007). Die Lage des Heidelberg Beckens wird durch einen gestrichelten Kreis angedeutet.

BRINK & BURRI (1993). The sedimentary fill of the URG is characterised by synthetic and antithetic faults which strike parallel or sub-parallel to this boundary fault. The area of the Heidelberg Basin was subjected to continuous and strong subsidence since late Oligocene (SCHUMACHER 2002). Up to 1500 m sediments were deposited in early Miocene alone (comprising *Cerethia*, *Corbicula*, and *Hydrobia* beds). The thickest succession of Quaternary sediments can be found here (up to 350 m according to BARTZ 1974; Fig. 2, top).

The Heidelberg Basin constitutes therefore the most complete sediment archive of the whole URG. Sediments of different geosystems interfere with each other: the local system of the Neckar River (Odenwald), the regional Upper Rhine Graben-Highlands (Black Forest, Vosges) system, and the supra-regional Alps-Upper Rhine Graben system. Especially the distal deposits of the system Alps – Upper Rhine Graben are supposed to contain information about both the alpine and north European climate history, which can not be observed elsewhere (e.g. ELLWANGER et al. 2005). The term Heidelberg Basin stands for the Miocene to Quaternary depocentre of the northern URG with an extent of some tens of km. The term ‘Heidelberger Loch’, sometimes also found in literature, was introduced by SALOMON (1927) and denotes the locally, very delimited centre of subsidence around the city of Heidelberg.

The exploration of this sediment archive is the aim of two new research boreholes sponsored by the Leibniz Institute of Applied Geophysics (LIAG – former Leibniz Institute for Applied Geosciences, GGA-Institut) and the geological surveys of Baden-Württemberg (LGRB) and Hessen (HLUG) (Fig. 2, bottom). One of the boreholes was drilled exactly in the depocentre of the basin, close to the outlet of the Neckar River, from the Odenwald to the plain of the Upper Rhine Graben (‘Heidelberg UniNord’, ELLWANGER et al. 2008). The other one was drilled in the geographic centre of the Heidelberg Basin about 17 km to the northwest, near the city of Viernheim (HOSELMANN 2008). These two locations are complemented by two

boreholes in Ludwigshafen that were drilled on the ‘Parkinsel’ island on the western margin of the basin (Fig. 2, bottom; WEIDENFELLER & KNIPPING 2008).

Prior to drilling these boreholes, a number of seismic profiles were carried out to facilitate a profound drilling design (Fig. 2; bottom). This included an estimation of depths for the geological targets, i.e. Pliocene and Pleistocene strata as well as the detection of possible fault zones. The latter point was important because the boreholes were to serve as a stratigraphic reference for the Quaternary sediment succession. Furthermore, high resolution seismic profiles in general are able to reveal tectonic and sedimentological events caused by the continued subsidence of the basin.

This article presents the results of the pre-site surveys. However, at the present stage, as there is not even a consistent and homogenous interpretation of all available borehole data, it is not possible to make conclusions about the basin dynamics (e.g. a seismostratigraphic interpretation). A more exhaustive analysis of the data could be done in the framework of a project, which was proposed to the German Research Foundation and is currently under evaluation.

2 Geological setting and nomenclature

Knowledge about the deeper subsurface of the URG is based mainly on seismic profiling and evaluation of boreholes of the oil and gas industry, which was quite intensively done in this area. According to MAUTHE, BRINK & BURRI (1993), about 5000 km seismic lines were recorded between 1970 and 1992. An extensive presentation of these activities is not possible due to the lack of corresponding publications. However, isobath maps derived from these data were published, e.g. for the Tertiary by DOEBL & OLBRECHT (1974) or for the Quaternary by BARTZ (1974).

Two deep-reaching reflection seismic profiles running across the southern and northern URG were carried out in 1988 in the framework of the DEKORP-ECORS Project (BRUN & GUTSCHER 1992). The northern profile crosses the URG

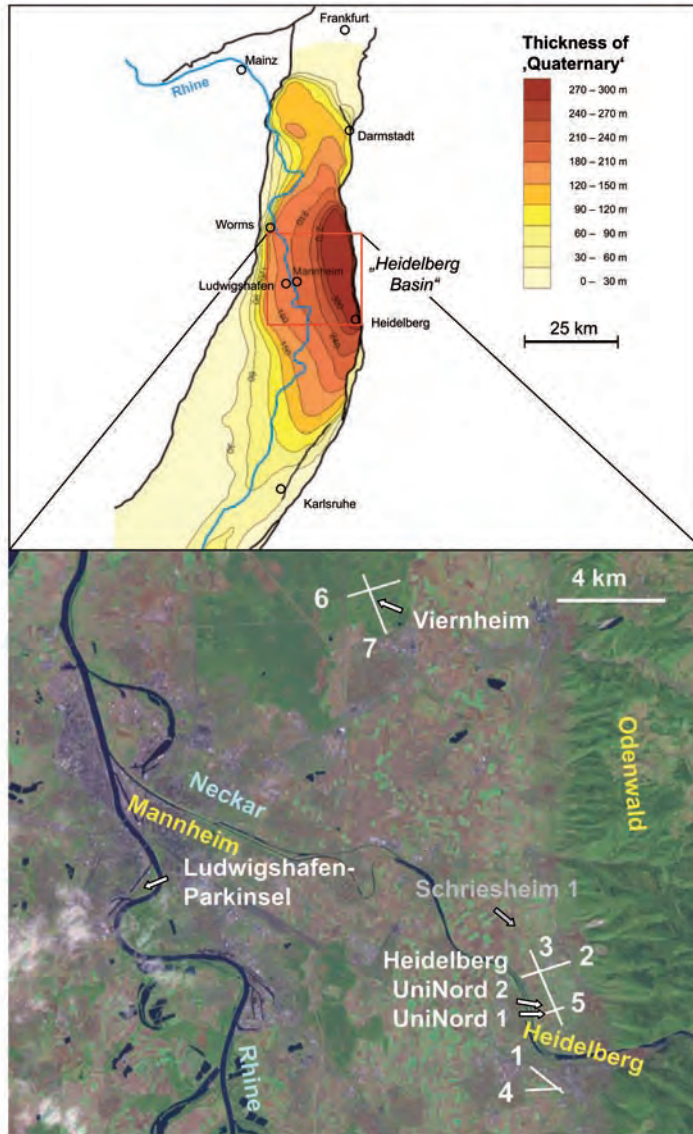


Fig. 2: *Top*: Thickness of 'Quaternary' in the northern part of the Upper Rhine Graben, adopted from BARTZ (1974, updated by HAIMBERGER, HOPPE & SCHÄFER 2005). *Bottom*: Satellite view of the Heidelberg area (created with NASA World Wind, Vers. 1.3.2). White arrows: research boreholes Heidelberg UniNord (two boreholes in 2006 and 2008), Viernheim (2006), and Ludwigshafen-Parkinsel (two boreholes in 2002 and 2006); grey arrow: hydrocarbon well 'Schriesheim 1'. White lines: reflection seismic profiles 1-7 that are discussed in this paper.

Abb. 2: *Oben*: Mächtigkeiten des 'Quartär' im nördlichen Teil des Oberrheingrabens nach BARTZ (1974, aktualisiert durch HAIMBERGER, HOPPE & SCHÄFER 2005).

Unten: Satellitenaufnahme des Heidelberger Raums (erstellt mit NASA World Wind, Vers. 1.3.2). Weiße Pfeile: Forschungsbohrungen Heidelberg UniNord (zwei Bohrungen in 2006 und 2008), Viernheim (2006) und Ludwigshafen-Parkinsel (zwei Bohrungen in 2002 und 2006); grauer Pfeil: Kohlenwasserstoffbohrung 'Schriesheim 1'. Weiße Linien: reflexionsseismische Profile 1-7, die in diesem Artikel diskutiert werden.

about 20 km north of Heidelberg. The varying reflector signature of the lower crust images the asymmetric structure of the URG. The largest Tertiary sediment thickness of 3400 m is observed close to the eastern rim of the graben, it reduces stepwise to 300 m below the western border. However, depths corresponding to the Quaternary, the Pliocene, and down to the middle Miocene units are poorly resolved.

The interaction between tectonic and sedimentation in the northern URG was investigated by DERER by means of sequence stratigraphy (DERER 2003, DERER et al. 2003, DERER, SCHUMACHER & SCHÄFER 2005), who found the area to be divided into two halfgrabens with opposing tilt directions by a transfer zone. The study was based on seismic profiles and geophysical borehole measurements made by the oil and gas industry. Seismic facies were assigned to several lithostratigraphic units from Eocene to the upper Miocene.

About 190 km of industrial seismic lines were shot between 1981 und 1993 in the Heidelberg Basin. However, these lines do not extend to the depocentre of the Heidelberg Basin ('Heidelberger Loch'). Furthermore, only little information about the structure of Plio-Pleistocene sediments can be derived from the hydrocarbon seismic lines, because these focused only on storage or sealing horizons, and hence deep structures. The existing gap in the reflection seismic data, near to the city of Heidelberg, can be partly filled by information from boreholes related to hydrogeological investigations. In the Heidelberg area two flush boreholes are available that reveal large thickness of Quaternary sediments: the 350 m deep Entensee borehole from 1973 (CONRADS & SCHNEIDER 1977), about 1 km north, and the 1022 m deep Radium-Sol Therme borehole from 1918 (SALOMON 1927), about 1 km south of the new borehole Heidelberg UniNord 1 (Fig. 3).

Interpretation of these two boreholes is controversially discussed. The main problem here are the confusing definitions and applications of the terms Quaternary and Pliocene (and some other stratigraphic terms) in publications, reports and archives related to the URG. The

most frequently applied 'traditional' definition relates 'Quaternary' to the onset of alpine sediments, i.e. to a change in sediment provenance. 'Pliocene' sediments come from local sediment sources, e.g. Black Forest, Vosges, and Odenwald; 'Quaternary' sediments include or correlate with sediments of alpine origin. This change of provenance is, of course, a matter of lithostratigraphy, although chronostratigraphic terms are used.

This different use of the term 'Quaternary' is illustrated by the controversial interpretations of the Radium-Sol Therme borehole by SALOMON (1927), BARTZ (1951) and FEZER (1997). SALOMON and BARTZ suggest depths of '382 m' and 'almost 400 m', both referring to the lithostratigraphic version of 'Quaternary'; FEZER suggests a depth of 650 m which is based upon a calculation of sedimentation rates i.e. he applies the term Quaternary in its proper chronostratigraphic sense.

The aim of this paper is not to solve problems of stratigraphic nomenclature but report seismic and gravimetric activities prior to the new drilling activities. Our references are the old data (publications, reports, and archive data) which are, at this state, not re-interpreted but used as they are. Stratigraphic terms are set in quotation marks where we feel the authors use the lithostratigraphic version (e.g. 'Quaternary'), and without quotation marks when used as chronostratigraphic term (e.g. Quaternary).

A reflection seismic profile that focused especially on the Quaternary deposits was published by HAIMBERGER, HOPPE & SCHÄFER (2005). This river seismic profile - recorded along the Rhine River between Mainz and Mannheim, and parts of the Neckar River over a length of 150 km in total - revealed high-resolution information which was able to define the base of 'Pleistocene' sediments mainly north of Mannheim. Maximum thickness of 'Pleistocene' was found in the area of Mannheim and confirmed the map published by BARTZ (1974; Fig. 2). This map images an increase of 'Pleistocene' sediment thickness towards the eastern boundary fault of the Upper Rhine Graben, where it amounts to more than 350 m.

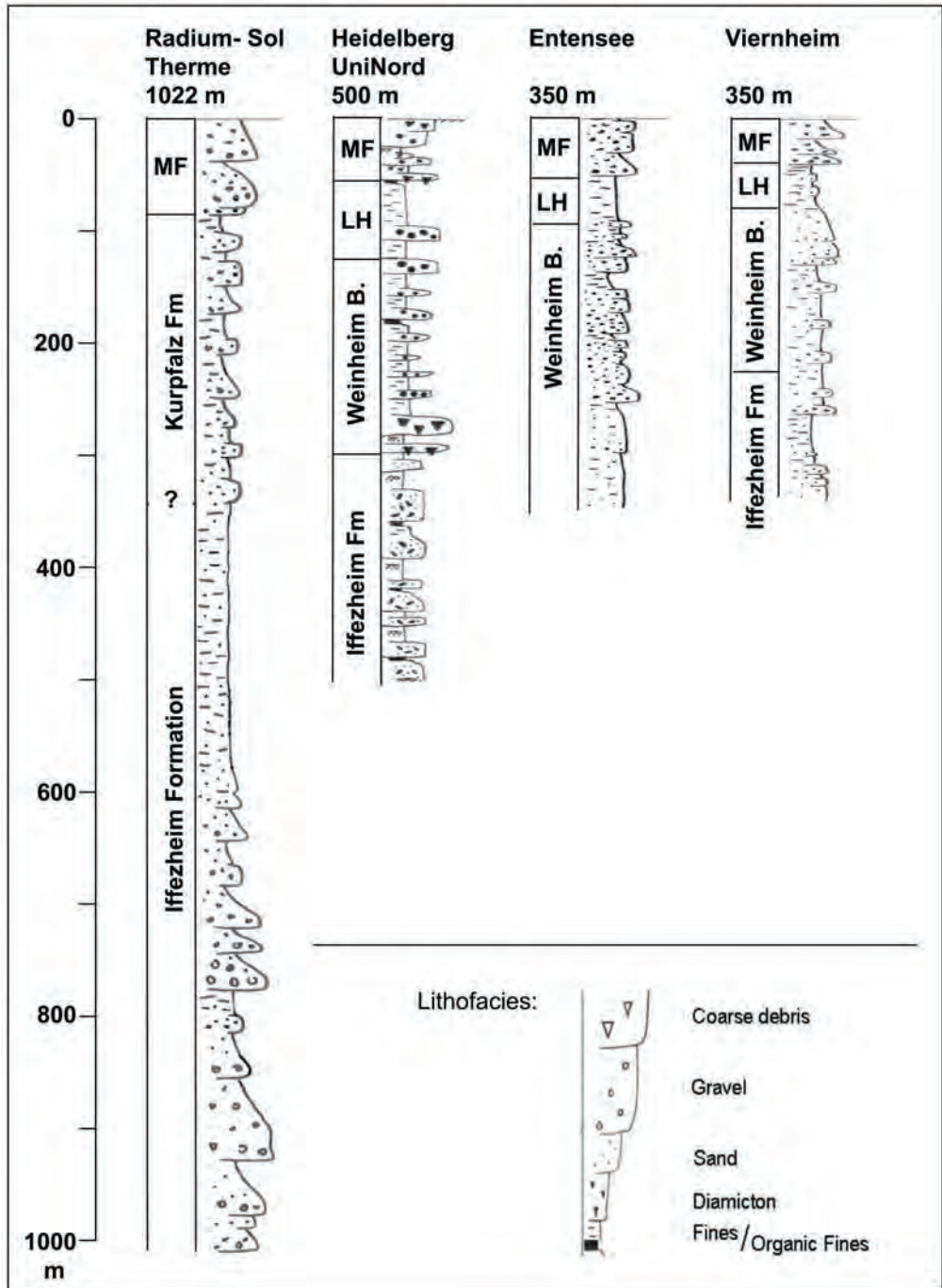


Fig. 3: Lithofacies logs from four boreholes in the Heidelberg Basin (MF: Mannheim-Formation, LH Ladenburg-Horizon).

Abb. 3: Lithofazieslogs von vier Bohrungen im Heidelberger Becken (MF Mannheim-Formation, LH Ladenburg-Horizont).

Table 1: Different classifications of Plio-Pleistocene sediments, as used in the Upper Rhine Graben. The terms ‘Altquartär’ and ‘Pliocene’ (senso Bartz) are defined by lithofacies, rather than a strict geochronological classification.

Tab. 1: Gegenüberstellung verschiedener Einteilungen der plio-/pleistozänen Sedimente für den Oberrhein-graben. Die Begriffe ‘Altquartär’ und ‘Pliozän’ (nach Bartz) definieren eher Sedimenttypen, denn eine strikte geochronologische Einordnung.

Baden-Württemberg <i>Symbolschlüssel Geologie Baden-Württemberg</i>		Rheinland-Pfalz <i>Weidenfeller & Kärcher 2008 Weidenfeller & Knipping 2008</i>	Hessen	Bartz 1982
			Aeolian sand (Pleistocene to Holocene)	
Mannheim-Formation		Upper gravel layer (OKL)	Sand-gravel layers <small>Neckar dominated, less alpine</small>	Oberes Kieslager (OKL) <small>contains coarse material (alpine and local)</small>
Kurpfalz-Formation	Ladenburg Horizon	Upper interlayer (OZH)	Interlayer <small>predominant fine-clastic sediments</small>	Obere Zwischenschicht (OZ) <small>only fine material</small>
	Weinheim Beds	Middle sand-gravel layer	Alternation of sand-(gravel) layers frequently in "Rhenish Facies" and	Mittleres Kieslager (MKL) <small>contains coarse material</small>
		Lower interlayer (UZH)		Untere Zwischenschicht (UZ) <small>only fine material</small>
		Lower sand-silt layer	fine-clastic interlayers	Unteres Kieslager (UKL) <small>contains coarse material</small>
				'Altquartär 1 and 2' (AQ1 and AQ2) <small>transition unit, contains the lowermost alpine material</small>
	<small>Rhine (alpine) material, less Neckar</small>			
<small>major change of provenance</small>				
Iffezheim-Formation		Clay-silt-sand-layer	Clay-silt- and sand-layers <small>material of local provenance</small>	'Pliocene I-III' <small>material of local provenance</small>

The interpretation of the river seismic data revealed well the alternating sequence of coarse-grained layers (aquifers, so called ‘Kieslager’) and fine-grained layers (aquitards, so called ‘Zwischenhorizonte’) typical of the Heidelberg Basin. This hydrostratigraphic classification (Table 1) is often used in the northern part of the Upper Rhine Graben due to a lack of geochronological data concerning Pliocene and Pleistocene strata. It is based on a macroscopic description of the sediments, their colours and carbonate content, and as complementary information on gamma logs, where available. The hydrostratigraphy was broadly introduced by BARTZ (1982). It is presently much used in hydrogeology (HGK 1999) and was last updated by WEIDENFELLER & KÄRCHER (2008). On a larger scale it distinguishes between three coarse-grained layers separated by two fine-grained

layers, which are regionally distributed. In the system after BARTZ (1982) this alternating succession is separated from the underlying provenance change by another unit called ‘Altquartär’ (‘Early Quaternary’). This term again is used as description of a sediment type rather than a stringent chronostratigraphic classification. In more recent publications and reports, the lowest coarse-grained bed is defined as Early Quaternary (WEIDENFELLER & KÄRCHER 2008). The thickness of each layer is quite variable in a lateral direction. Locally, additional fine- or coarse-grained layers of smaller extent can be intercalated. The occurrence of fine-grained sediments was traditionally related to interglacial periods, and the occurrence of coarse-grained layers to glacial periods (BARTZ 1982). One of the aims of this drilling project is to correlate the before mentioned hydrostratigra-

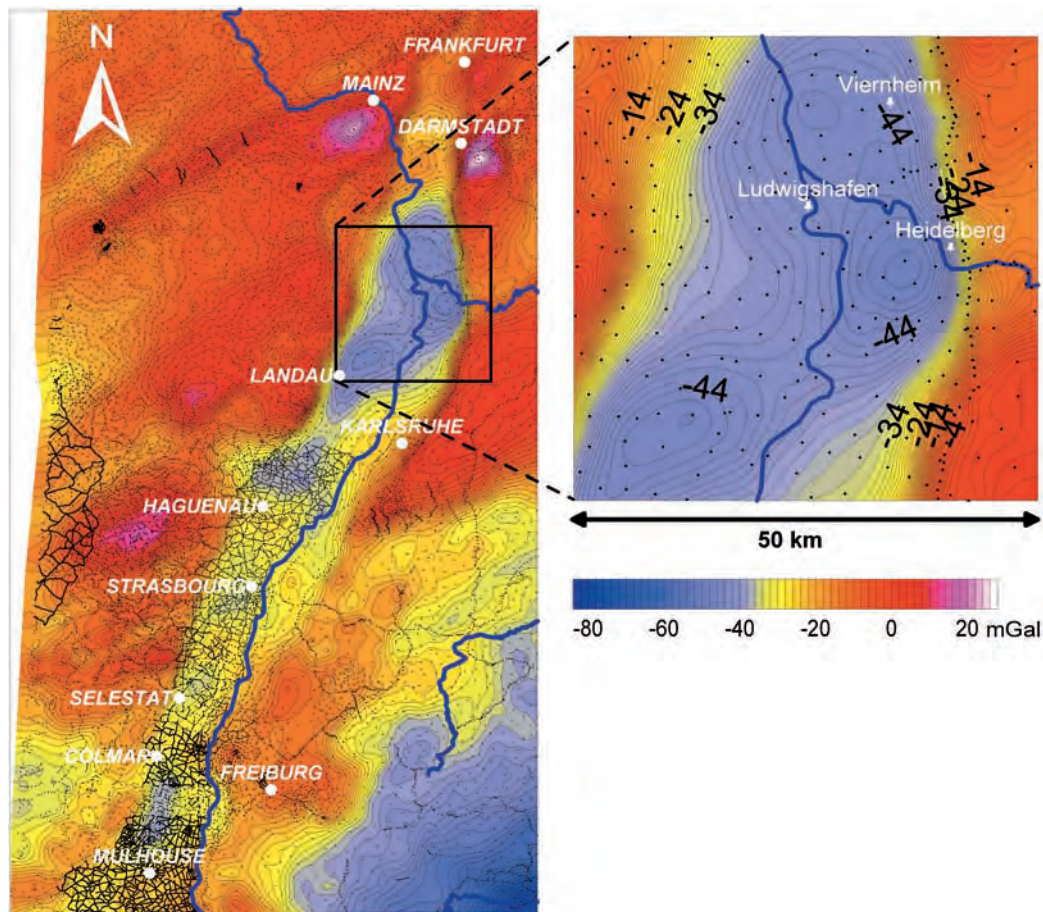


Fig. 4: Bouguer map of the entire Upper Rhine Graben (after ROTSTEIN et al. 2006) and the Heidelberg area in more detail, with the three research borehole locations Ludwigshafen-Parkinsel, Viernheim, and Heidelberg. Contour intervals: 2 mGal (regional map) and 1 mGal (local map); black dots: gravity stations.

Abb. 4: Bouguer-Karte des gesamten Oberrheingrabens (nach ROTSTEIN et al. 2006) und des Heidelberger Raums im Detail mit den drei Lokationen der Forschungsbohrungen Ludwigshafen-Parkinsel, Viernheim und Heidelberg. Isolinienabstand 2 mGal (regionale Karte) und 1 mGal (lokale Karte); schwarze Punkte: gravimetrische Messpunkte.

phy with the lithostratigraphy of the southern part of the Upper Rhine Graben (SYMBOL-SCHLÜSSEL GEOLOGIE BADEN-WÜRTTEMBERG). The upper gravel layer can be quite well correlated with the Mannheim Formation, and the upper interlayer (fine-grained layer) with the Ladenburg Horizon. The succession of the middle sand-gravel layer and lower sand-silt layer and the interlayer in between is only roughly equivalent to the Weinheim Beds (Table 1).

3 Gravimetry

Generally, gravity anomaly data is expected to reflect a first-order pattern of the shape of the Heidelberg Basin, especially varying thickness of (unconsolidated) Pliocene/Pleistocene sediments. The mean density of Plio-Pleistocene sediments should be reduced compared to Tertiary or even older and more compacted sediments. Therefore the anomalous thick sedimentary fill of the Heidelberg Basin should cause a

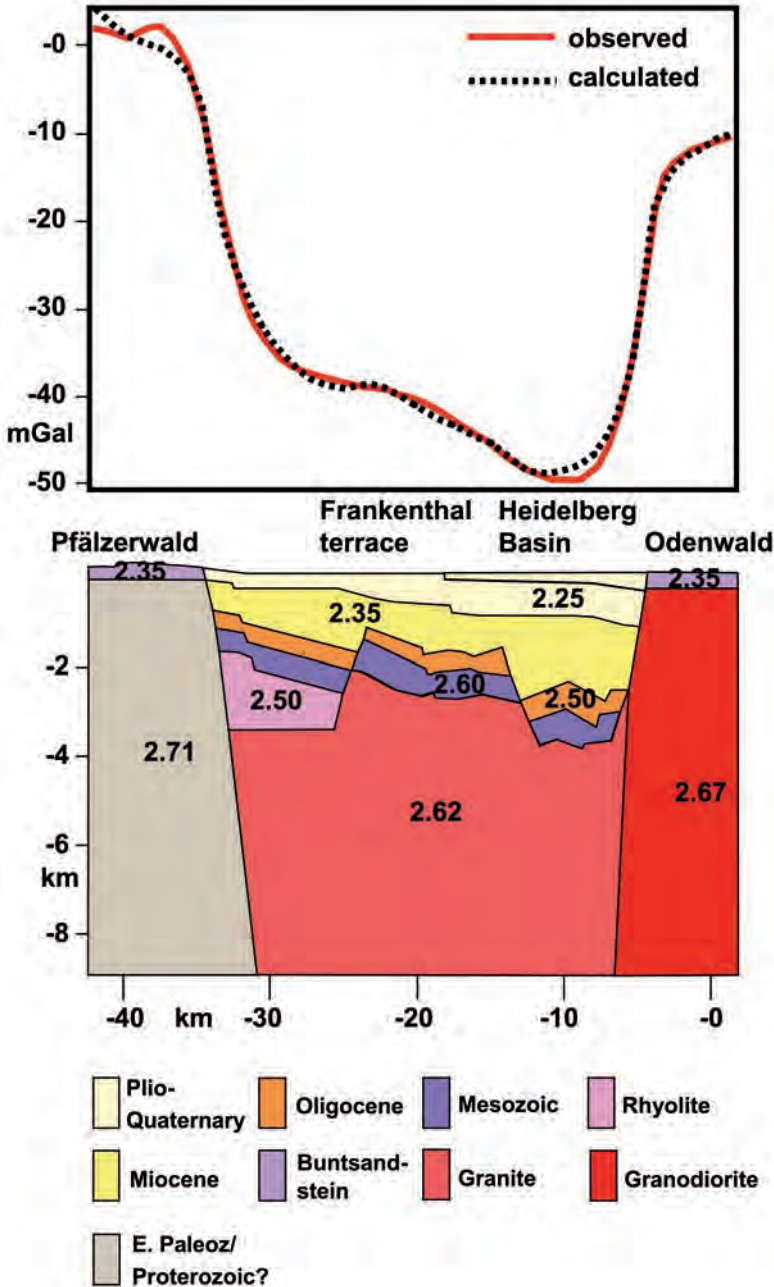


Fig. 5: 2-D gravity profile across the Rhine Graben in the Heidelberg area. Although the regional gravity field correlates well with sediment thickness, additional density contrasts in the basement are required to explain the observed anomalies in the western part.

Abb. 5: Gravimetrisches 2-D Profil über den Rheingraben im Raum Heidelberg. Wenggleich das regionale Schwerefeld gut mit den Sedimentmächtigkeiten korreliert, müssen Dichtekontraste im Basement angenommen werden, um die beobachteten Anomalien des westlichsten Profilabschnitts zu erklären.

negative gravity anomaly that can be related to the extent of the basin and its depth. But in fact the situation is more complicated.

Gravity data

Gravity data for the Upper Rhine Graben is available from several sources. ROTSTEIN et al. (2006) have compiled the most recent Bouguer gravity map of the Upper Rhine Graben comprising all the available data from France and Germany (Fig. 4; regional map). This map is based on about 33.000 Bouguer gravity values. About one third of the data was provided by the Leibniz Institute for Applied Geophysics covering the German part of the Rhine Graben. This dataset consists of data available from the Geophysikalische Reichsaufnahme, but also some local surveys. The French data is mainly based on two data sources, which are themselves compilations of numerous surveys. The first dataset – about fifty percent of the French data - was provided by the Bureau de Recherches Géologiques et Minières, the second one by Mines de Potasse d'Alsace. A complete Bouguer anomaly was recalculated for the entire new dataset, using a density of 2670 kg/m^3 and considering newly-calculated terrain reductions. The distribution of the gravity stations is strongly inhomogeneous; their spatial coverage varies significantly from about 0,25 stations/km² in some parts of the Vosges and Black Forest and 40 stations/km² in some parts of the graben itself. Note, the complete Bouguer map is based on a calculated grid of 1 km.

The area of the Heidelberg Basin (Fig. 4; local map) is characterised by the strongest gravity anomaly of the entire Upper Rhine Graben (apart from the most southern part where the regional trend is strongly affected by the Alps). It is in the order of -40 to -50 mGal, with the absolute minimum close to the outlet of the Neckar River, from the Odenwald in the plain of the Upper Rhine Graben (Fig. 4).

Preliminary Interpretation

When interpreting the anomaly apparently related to the sediment fill of the Heidelberg Basin

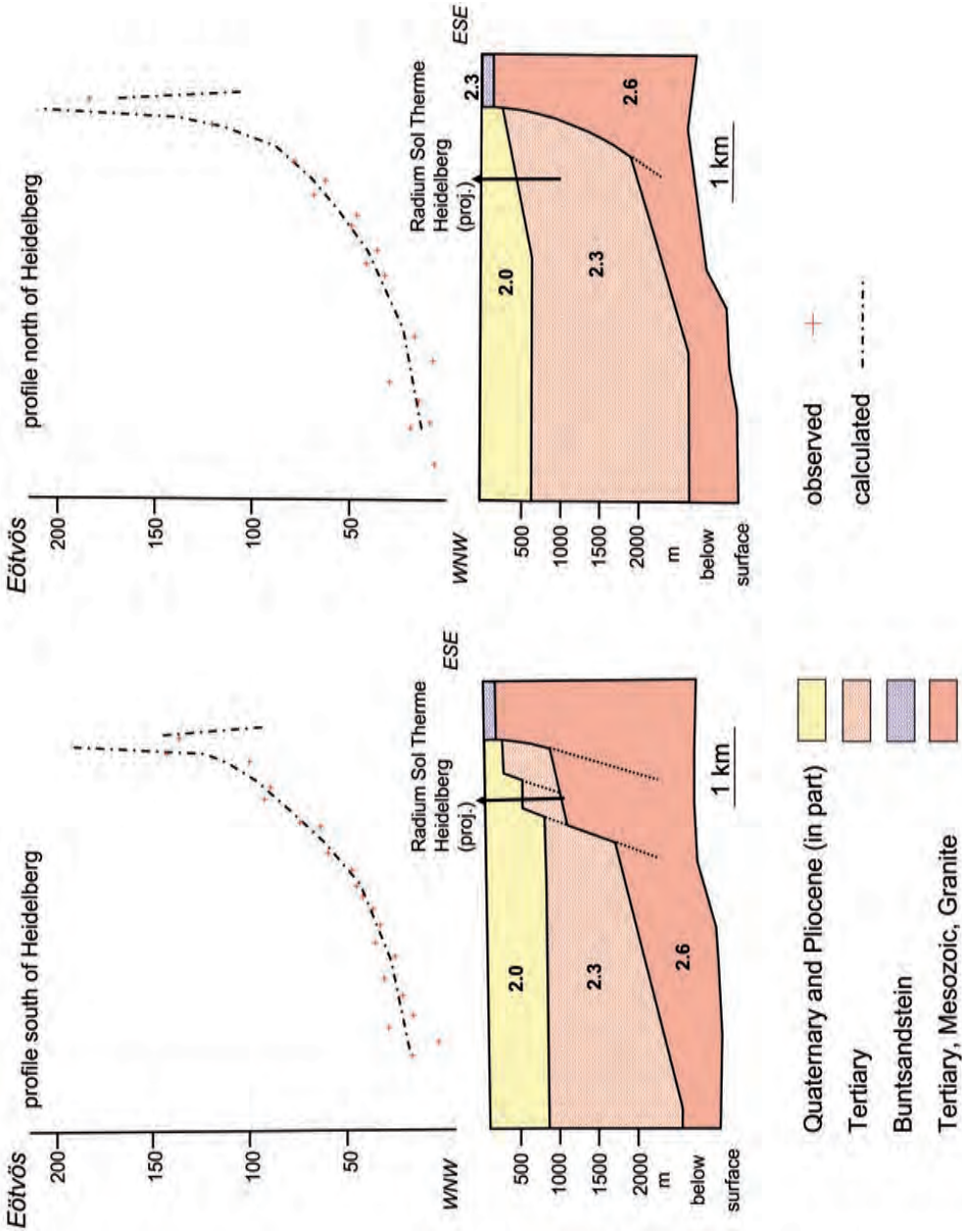
some general knowledge regarding the source of gravity anomalies in the Upper Rhine Graben has to be considered. ROTSTEIN et al. (2006) performed some two-dimensional calculations along profiles approximately perpendicular to the strike of the graben. Although these profiles were restricted to the southern part of the graben and a two-dimensional approach is not suitable to investigate the complex graben geology in great detail, their general result showed, that the gravity anomalies are not only caused by the sediment fill of the graben, but that they are also strongly affected by density inhomogeneities in the crystalline basement. One of the profiles south of Karlsruhe revealed increasing sediment thicknesses from west to east accompanied by increasing Bouguer gravity values – the structure of the sediments was well constrained by reflection seismic data and drilling information. Therefore, for this profile the gravity effect of the sediments must be overcompensated by density contrasts in the basement.

Density of the basement must be assumed to be of high lateral variation as that of rocks of the adjacent graben shoulders is. Therefore, some impact on the Bouguer anomalies observed in the area of the Heidelberg Basin must also be assumed. A preliminary 2-D profile which crosses the Rhine Graben close to Heidelberg is shown in Fig. 5. Although the structural resolution of the sediments is rather low, the necessity to introduce at least lateral density contrasts at the western and eastern boundary faults and within the most western part of the basement is obvious.

Nevertheless, the sediments of the Heidelberg Basin will undoubtedly contribute to the negative gravity anomalies observed in this region. Confirming the map of 'Quaternary' thickness in the Upper Rhine Graben published by BARTZ (1974), the new reflection seismic profiles recorded in the framework of the pre-site surveys reveal anomalously thick Neogene sediments. From the downhole logging experiments conducted in the Heidelberg Drilling Project, low densities between 2000 kg/m^3 and 2300 kg/m^3 can be estimated for the Pleistocene sediments (HUNZE & WONIK 2008), whereas Tertiary sedi-

Fig. 6: First quantitative interpretation of the horizontal gradient (in Eotvös, $1 E = 10^{-9} s^{-2} = 0.1 \text{ mGal/km}$) of the gravity field related to the sediments of the Heidelberg Basin after CLOSS (1943). No density contrasts in the basement are considered, although a strong lateral variation of density must be assumed. The dip of the youngest sediments towards the west contradicts the new reflection seismic data.

Abb. 6: Erste quantitative Interpretation des Horizontalgradienten (in Eotvös, $1 E = 10^{-9} s^{-2} = 0.1 \text{ mGal/km}$) des Schwerfeldes im Hinblick auf die Sedimente des Heidelberger Beckens nach CLOSS (1943). Obwohl von starken lateralen Dichtekontrasten im Basement auszugehen ist, werden diese nicht berücksichtigt. Das Einfallen der jüngsten Sedimente nach Westen steht im Widerspruch zu den neuen reflexionsseismischen Ergebnissen.



ments in the Upper Rhine Graben are known to have densities between 2350–2450 kg/m³, strongly depending on the amount of evaporates in the Early Oligocene strata, at least in the southern part of the graben (ROTSTEIN et al. 2006).

Interpreting the observed gravity anomalies only in terms of varying sediment thickness, two main centres of deposition can be distinguished: one between Heidelberg and the Rhine River, and the second northeast of Landau (Fig. 4). Considering the information about Base ‘Quaternary’ (Fig. 2), the anomaly close to Landau is not related to anomalously thick Quaternary deposits. The source of this anomaly must be assumed to be at greater depth. This source can be located either in the older sediments, e.g. thick deposits of Early Miocene age in agreement with SCHUMACHER (2002), or in the basement, e.g. an extension of units that outcrop in the northern Odenwald today, to the southwest. Only the large negative gravity anomaly east of the Rhine River can be discussed in terms of varying thickness of Quaternary sediments, even though this interpretation is ambiguous. This anomaly is related, at least to some extent, to the ‘Heidelberger Loch’. The Ludwigshafen boreholes are located on the western margin of this basin structure; the Heidelberg UniNord borehole close to the absolute gravity minimum. With respect to the map of ‘Quaternary’ sediments (BARTZ 1974, improved by HAIMBERGER, HOPPE & SCHÄFER 2005), the Heidelberg Basin extends further southwards, towards Karlsruhe. This extension cannot be traced unequivocally in the gravity map, where north and west of Karlsruhe a relative gravity high occurs. The contour lines delimiting this relative gravity high from the more distinct gravity low to the north strike nearly W-E, which is not in accordance with the map of Base ‘Quaternary’. But these observations correspond well with the quantitative interpretation of the gravity anomalies south of Karlsruhe discussed previously and presented by ROTSTEIN et al. (2006). High density Early Palaeozoic and Proterozoic rocks in the basement are suggested as source for this anomaly

compensating the gravity effect of sediments. First quantitative interpretations of the gravity field of the Heidelberg Basin were published by CLOSS (1943). Based on torsion balance data, models were derived that explain the observed horizontal gradient (in Eotvös, $1 \text{ E} = 10^{-9} \text{ s}^{-2} = 0.1 \text{ mGal/km}$) running from the Odenwald in the east about 5 km into the Upper Rhine Graben in the west (Fig. 6). Therefore, only the local situation at the eastern graben boundary fault was investigated. The thickness of Pleistocene and youngest Pliocene sediments is estimated to be ~ 500 m at the Heidelberg UniNord location. Furthermore in CLOSS’s model the uppermost sediment unit dips with about 45° towards west – strongly contradicting the result of the new reflection seismic surveys. Density contrasts in the crystalline basement of the URG in this region were not taken into account, although from the adjacent Odenwald a large variety of alkaline (high density) and acidic (lower density) rocks are known.

4 Seismic survey and data processing

Seismic measurements in urban areas often encounter considerable difficulties. Restrictions exist for seismic sources (only low-energy seismic sources, services lines, endangerment of supply lines, services pipes, traffic restrictions, etc.) as well as for the recording side (sealing of the surface, enhanced noise level, etc.). The design of seismic profiles is therefore more often dictated by logistics than by geological reasoning.

At the start of the project a borehole location in Heidelberg was proposed at the abandoned freight depot south of the Neckar River (Fig. 7). A 1.5 km long profile (profile 1) was therefore measured, that showed strong inclinations of deep reflectors. To check the true inclination of the reflectors, the data were supplemented by a second profile later on (profile 4). The profiles could not be connected to existing industry profiles (the next one being about 1.5 km apart to the SW), but the interpretation could be done by comparison of reflection patterns. The borehole Radium Sol Therme could barely be

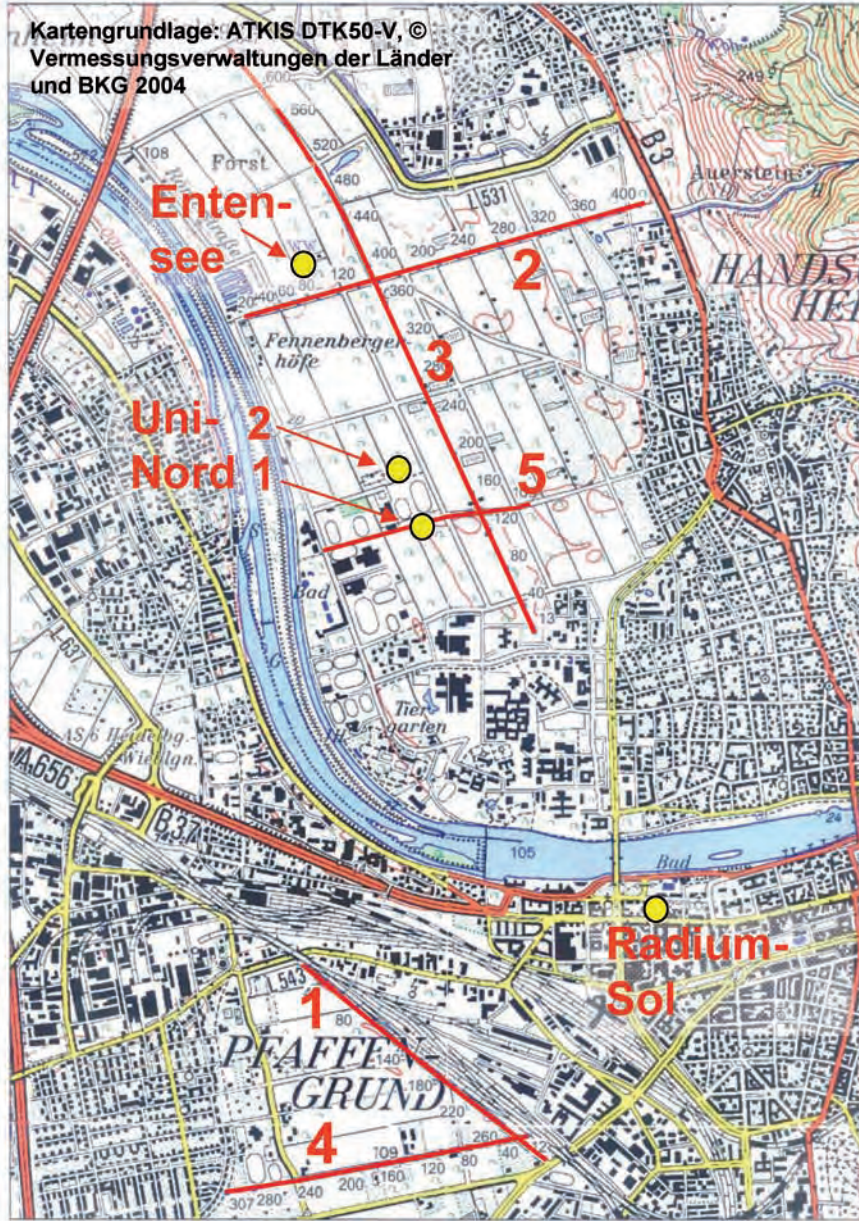


Fig. 7: Seismic reflection profiles (red lines) measured in Heidelberg. Yellow points indicate boreholes mentioned in this paper.

Abb. 7: In Heidelberg aufgezeichnete reflexionsseismische Profile (rote Linien). Gelbe Punkte markieren die in diesem Artikel genannten Bohrungen.



Fig. 8: Seismic hydraulic vibrator, constructed for near surface high resolution profiling.

Abb. 8: Seismischer Hydraulikvibrator, der für hoch auflösende oberflächennahe Messungen entwickelt wurde.

used for interpretation due to its controversial interpretation. In addition, data quality suffered due to extreme electromagnetic disturbances of 16 2/3 Hz and its multiples up to 500 Hz. North of the Neckar River the building density is lower, improving the conditions for seismic

measurements. First, a profile perpendicular to the general trend of faults was shot (profile 2). After some encouraging results, a longer north - south trending profile (profile 3) was registered. However, continuation to the exploration

Table 2: Recording parameters used in the reflection seismic surveys.

Tab. 2: Aufnahmeparameter der reflexionsseismischen Messungen.

	Profiles 1-4	profiles 5-7
Seismic source	GGA-vibrator MHV 2.7	GGA-vibrator MHV 2.7
Sweep	16 s linear, 30-180 Hz	16 s linear, 30-180 Hz
Recording length	20 s	20 s
Source/recording point distance	10 m / 10 m	5 m / 5 m
Reflexion point distance	5 m	2,5 m
Geophone type	SM4, 20 Hz	SM4, 20 Hz
Group length/geophones in group	5 / 6	- , 1
Spread	fixed spread (p. 1, 4) end on (p. 2,3)	end on
Recording instrument	Geometrics (StrataVisor or Geodes)	Geometrics (Geodes)
Recording mode	unstacked, uncorrelated	unstacked, uncorrelated
Number of channels	72 (p. 1,2), 120 (p. 3, 4)	120
Vertical stacks	4	2
Samplerate	1 ms	1 ms

borehole 'Schriesheim', about 1 km further north (Fig. 2), could not be established. After fixing the location of the research borehole 'Heidelberg UniNord 1', another short profile (profile 5) was shot to check for possible fault zones. In this area signal quality was degraded by waves originated from service pipes (called 'pipe waves' for simplicity further on). These are especially visible on profile 2 and profile 5. The two profiles at the Viernheim location (profiles 6 and 7 in Fig. 2) were laid out perpendicular to each other, with the proposed position of the research drilling at their crossing point. The profiles were surveyed in a forested area without further problems.

All profiles were shot by a small hydraulic vibrator (Fig. 8), which yields a maximum peak force of 30 kN and a frequency range of 20 - 500 Hz (for recording parameters cp. Table 2). This vibrator was developed especially for high-resolution shallow profiling (BUNESS & WIEDERHOLD 1999, VAN DER VEEN et al. 2000). It is very appropriate for use in an urban environment, due to its relatively low impact on the surface, compared to e.g. weight dropping or other impulsive sources. Data were processed using a commercial processing system (ProMAX, Landmark Corp.). Processing steps are listed in Table 3, details regarding the single steps are discussed e.g. in YILMAZ (2001). In addition, to the processing steps listed in Table 3, waves in profiles 2 and 5 were suppressed by trace-mixing algorithms. Trace mixing, although a very simple algorithm, proved to be most effective of a variety of other algorithms, including spatial 2D filters, f-k based filters and eigenvector filtering. However, the incoherency of this kind of noise prevents elimination without damaging the reflection signal.

The velocity fields for finite-difference migration and for the subsequent depth conversion were derived from the smoothed stacking velocity fields. The deviations from the velocities derived by the VSP (vertical seismic profile) measurements carried out in the UniNord 1 borehole down to a depth of 180 m turned out to be very small, hence the former were kept during later

processing. The structure of the uppermost low velocity layer down to a depth of 20 m could not be determined adequately, causing some uncertainty about the overall depth level of the profiles. As a consequence the depth level was calibrated using the VSP reflections, which have a known depth. A reference level of 100 m a.s.l. was chosen for all profiles.

Table 3: Typical data processing flow

Tab. 3: Typischer Prozessingsablauf

1. correlation with field sweep
2. quality control
3. vertical stack
4. automatic gain control 300 ms
5. deconvolution (spiking, zero phase, 30 ms operator)
6. bandpass 30-180 Hz, 24db/oct.
7. surgical mute of surface waves and shear waves
8. attenuation of air-coupled waves
9. refraction static to floating datum
10. velocity analysis and iterative application of residual static (initial step with lowpass 90 Hz)
11. stack to final datum (100m)
12. FX deconvolution (60 – 180 Hz)
13. automatic gain control 500 ms
14. FD migration
15. depth conversion

5 Results

Heidelberg – the region north of the Neckar River

The profile which is best tied to an existing deep borehole is the north-south trending profile 3 in the area north of the Neckar River: the Schriesheim borehole is located about 1 km further north (Fig. 2). Although no direct connection between the newer seismic line and the borehole exists, the geological structures can be controlled by hydrocarbon seismic lines. Both, E-W and N-S trending profiles that cross the Schriesheim borehole are available.

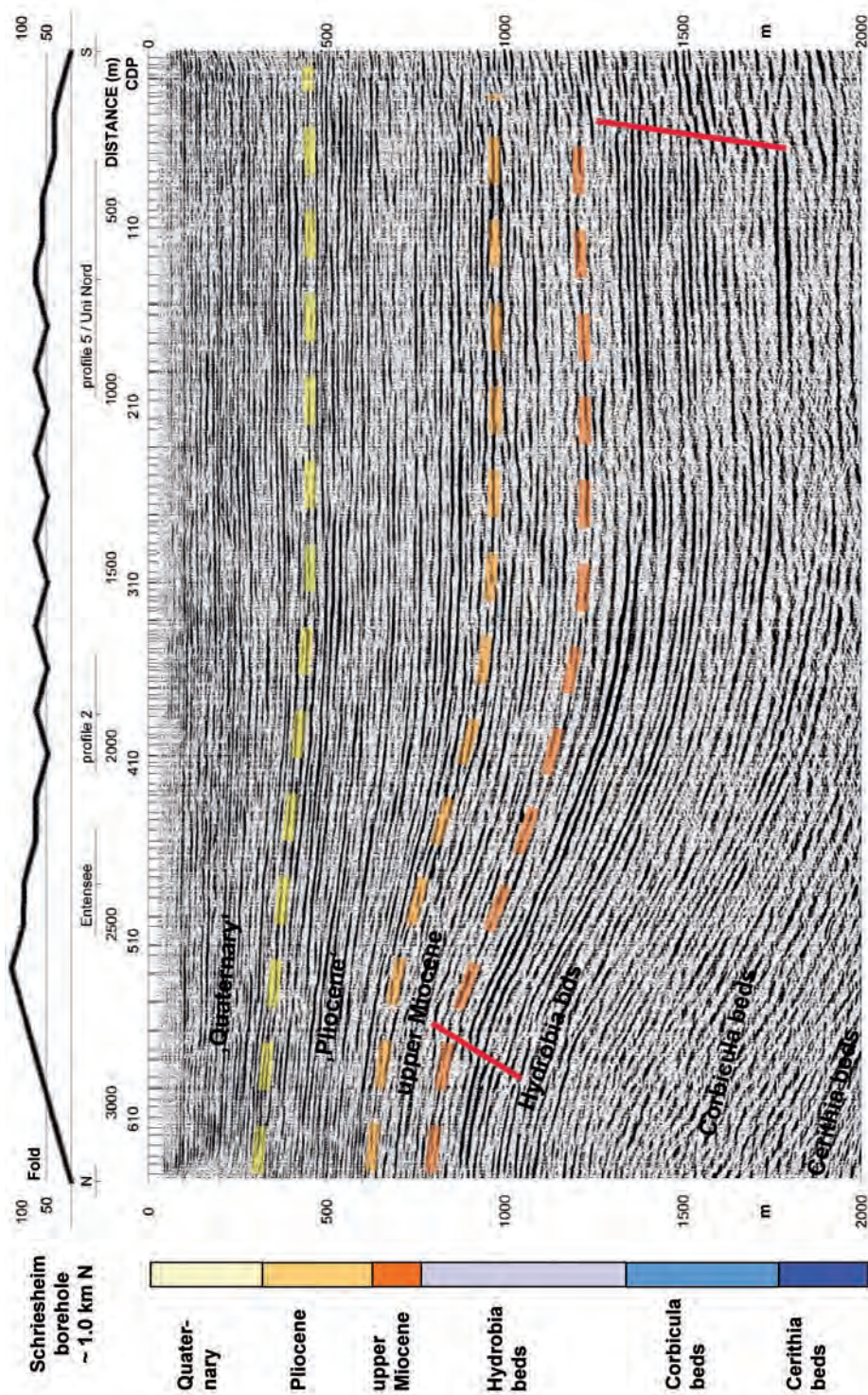


Fig. 9: Migrated seismic reflection profile 3. The 2650 m deep borehole 'Schriesheim' is located about 1 km north of profile 3.

Abb. 9: Migriertes reflexionsseismisches Profil 3. Die 2650 m tiefe Bohrung 'Schriesheim' liegt etwa 1 km nördlich des Profils 3.

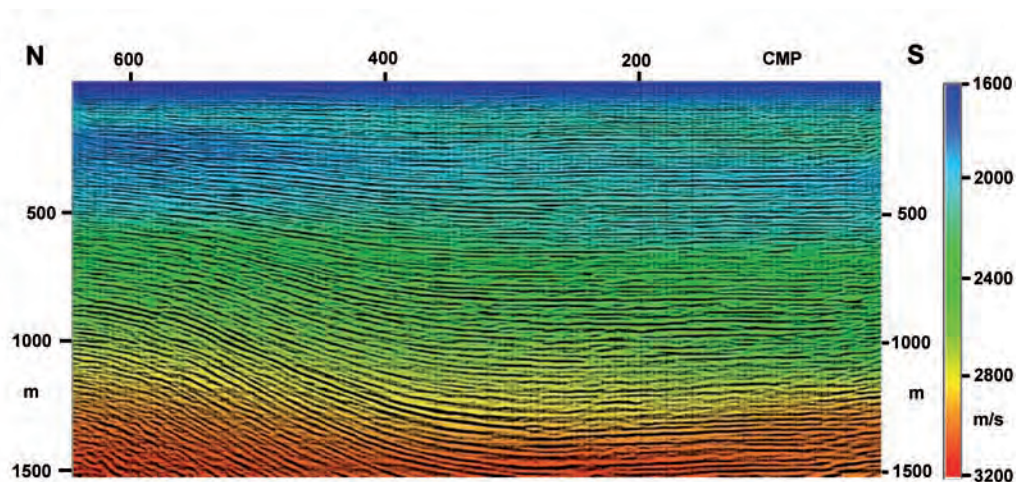


Fig. 10: Estimation of the interval velocity field along reflection profile 3 derived from stacking velocities. The colour-coded interval velocities are overlain by the migrated seismic section shown in Fig. 9.

Abb. 10: Intervallgeschwindigkeiten abgeschätzt aus Stapelgeschwindigkeiten entlang des Profils 3. Die farbkodierten Intervallgeschwindigkeiten sind von dem in Abb. 9 dargestellten migrierten seismischen Profil überlagert.

Fig. 9 shows the preliminary stratigraphical interpretation of profile 3 that considers Base 'Quaternary' and Base Late Miocene. Only based on seismic data, the separation between these units remains ambiguous because of missing continuous reflections, e.g. Base 'Quaternary'. Furthermore, no distinct changes in the reflection patterns mark these boundaries.

Concerning deposits of Quaternary age, segments with strong continuous reflections that reveal correlation lengths in the order of 500 to 1000 m alternate laterally with sections characterised by low reflection energy and sub-parallel deposition. This can be interpreted in the sense of repeating changes of the depositional environment. At the southern end of the profile, where subsidence was greatest, more continuous reflections become apparent in the deeper part of the Pleistocene than in the north. A throughout, low-reflective, sub-parallel bumpy to chaotic reflection pattern as described by HAIMBERGER, HOPPE & SCHÄFER (2005) for some parts of the river seismic along the Rhine was not observed in our sections. This gives some evidence for lateral changes of the depositional environments. The reflection patterns caused by the underlying Pliocene and Miocene strata consist of

more continuous reflections. Especially in the most southern part of profile 3, some areas at depths between 600 and 700 m are of low reflectivity. The most distinct reflector appears just below the top of the *Hydrobia* beds.

The additional subsidence in the 'Heidelberger Loch' – the depocentre of the Heidelberg Basin – amounts to 160 m, with respect to the transition Pliocene / Pleistocene and 300 m with respect to Base Pliocene. This subsidence is not linked to any faults with large displacements but to flexures of the beds that were probably formed syn-sedimentarily. Faults with distinct displacements are related to the upper strata of the *Hydrobia* beds only. At the northern end of profile 3, normal faulting is observed. The fault interpreted at the southern end of the profile reveals a pattern that is typical for diffractions although the section is migrated and therefore no diffraction should occur. But this holds only if the fault strikes perpendicular to the seismic profile. Consequently a large fault, proximately south of the profile, is assumed parallel to the course of the Neckar River.

Often, the seismic velocity field images geological structures by sharp vertical and lateral velocity changes. But for the area of the

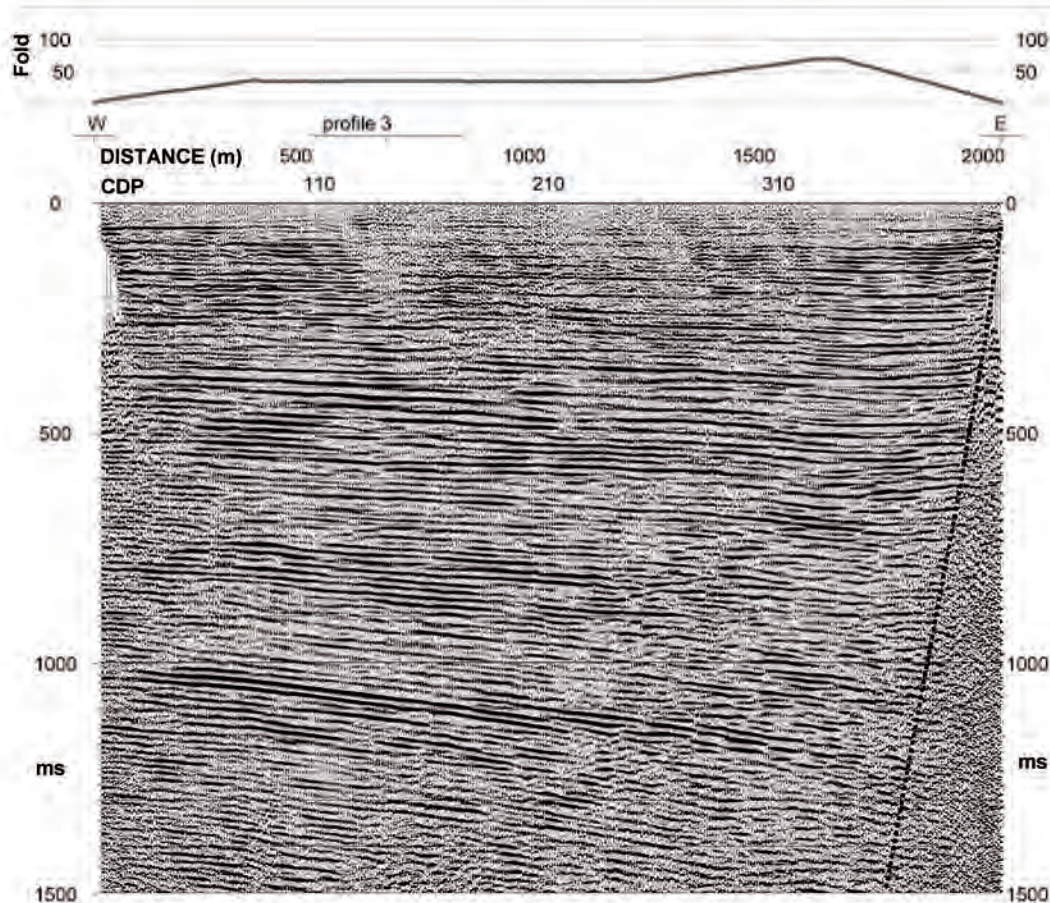


Fig. 11: Stacked seismic reflection profile 2 in time domain, unmigrated. Areas with very low reflectivity in the upper 300 ms in the central part of the profile are due to strong noise and do not reflect geology. A wedge-shaped zone at the eastern edge of the profile, indicated by a dotted line, maybe caused by the transition of the sedimentary infill of the URG to the crystalline basement of the Odenwald.

Abb. 11: Gestapeltes reflexionsseismisches Profil 2 im Zeitbereich, unmigriert. Die stellenweise sehr geringe Reflektivität in den oberen 300 ms in zentralen Teil des Profils wird nicht durch die Geologie, sondern durch starke Störungen hervorgerufen. Eine keilförmige, durch eine punktierte Linie markierte Zone am Ostrand des Profils kann durch den Übergang von der Sedimentfüllung der ORG zum kristallinen Grundgebirge des Odenwaldes verursacht werden.

Heidelberg Basin investigated with the new reflection lines no distinct information is provided by the velocity fields. An estimation of the velocity field of profile 3 (Fig. 10) reveals interval velocities that increase gradually from 1600 m/s near the surface to 3200 m/s at a depth of 1500 m. These velocities are deduced from stacking velocities, which do not constitute physical seismic velocities, since

they are affected by layer dips, side reflections, diffractions and other seismic particularities. The reliability of stacking velocities decreases strongly with depth, depending on the maximum offset of the seismic survey. The velocities cannot be controlled by other methods, e.g. VSP measurement, due to the lack of deep VSP (vertical seismic profile) or sonic measurements. However, they coincide

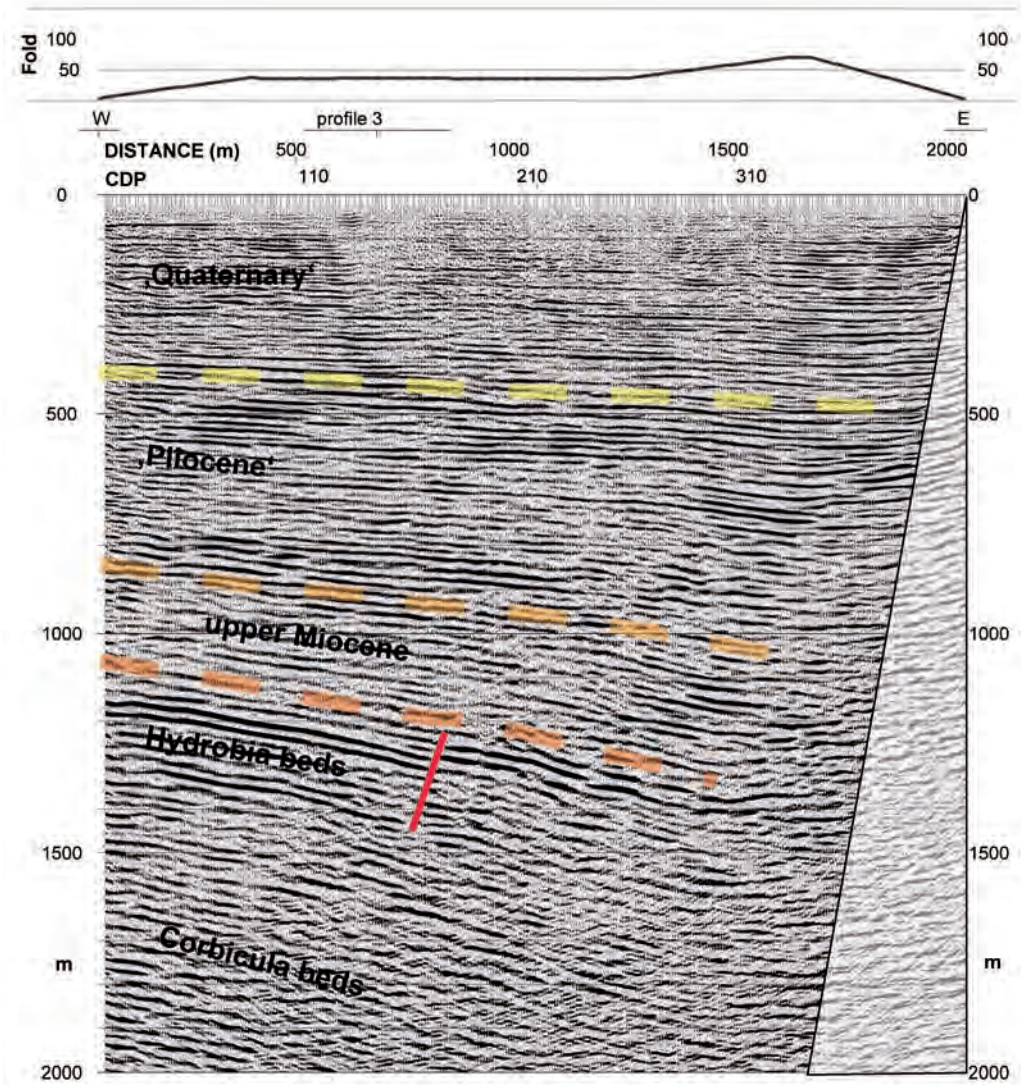


Fig. 12: Migrated seismic reflection profile 2. Seismic signals inside the transparent triangle are not interpretable because of geometrical reasons and migration artefacts. The strongly-bent reflectors close to the triangle zone maybe caused by drag folding.

Abb 12: Migriertes reflexionsseismisches Profil 2. Seismische Signale innerhalb des transparenten Dreiecks können aufgrund der geometrischen Verhältnisse sowie von Migrationsartefakten nicht interpretiert werden. Die stark gekrümmten Reflektoren unmittelbar neben diesem Dreieck könnten auf eine Schlepplafte deuten.

quite well with the regional velocity trend derived from deep boreholes in the Heidelberg Basin. A remarkable feature is a zone of low velocity, which has a depth of approximately 180 – 250 m at the northern edge of the profile and of 300 - 450 m at its southern edge. The upper limit of this zone corresponds therefore

well with the lower boundary of the coarse-grained Weinheim beds (cf. Fig. 3).

Profile 2 is located between the Neckar River and the eastern boundary fault of the Upper Rhine Graben (see Fig. 7). The profile approaches at its eastern edge the topographic border of the URG and hence the master boundary fault of the URG.

Both the unmigrated time section (Fig. 11) and the migrated depth section (Fig. 12) are presented, so the reader can better judge the influence of the migration processing step. Generally, the reflections dip to the east, with dips becoming greater with increasing depth. A wedge-shaped zone at the easternmost depth (marked by the dotted line in Fig. 11) shows no coherent signal. This wedge is probably due to the eastern master fault which juxtaposes the sedimentary infill of the URG against the crystalline basement of the Odenwald. A very rough estimation of its dip yields values of approximately 80° . Adjacent to this wedge, flat or slightly westward dipping reflectors are displayed at travel times greater 700 ms. This feature is interpreted as a drag fold. Below 700 ms, reflections again dip towards east. Some hints for diffractions can be seen, that could be caused by the fault zone (e.g. CMP 260 at 800 ms). Areas of apparently low reflectivity as observed in the youngest Quaternary, e.g. between CMP 110 and CMP 310, are caused by strong noise due to pipe waves.

After migration (Fig. 12), the bending of the sediment strata next to the assumed boundary fault becomes more obvious. However, it is difficult to separate real reflections from migration artefacts that always occur at the ends of a seismic profile. Signals inside the marked triangle should not be considered as real reflectors. The continuous subsidence of sediments beneath the central and western part of the profile is revealed by an increasing dip angle with depth. The apparent dip of Base 'Quaternary' is 1.5° towards east and 4.5° for Base Pliocene. A fault with a distinct displacement is again only visible in the upper part of the *Hydrobia* beds. Profile 5 was recorded with modified acquisition parameters (smaller CMP spacing, smaller offset, cp. Table 2), which yielded a higher resolution (Fig. 13). Again, in the eastern part the image of Quaternary deposits is significantly affected by pipe waves. At depths between 600 and 700 m, reduced reflectivity is observed at the western end of the profile. The dip angles of Base 'Quaternary' and Base Pliocene, 2° and 6° respectively, are slightly increased with respect to those of profile 2.

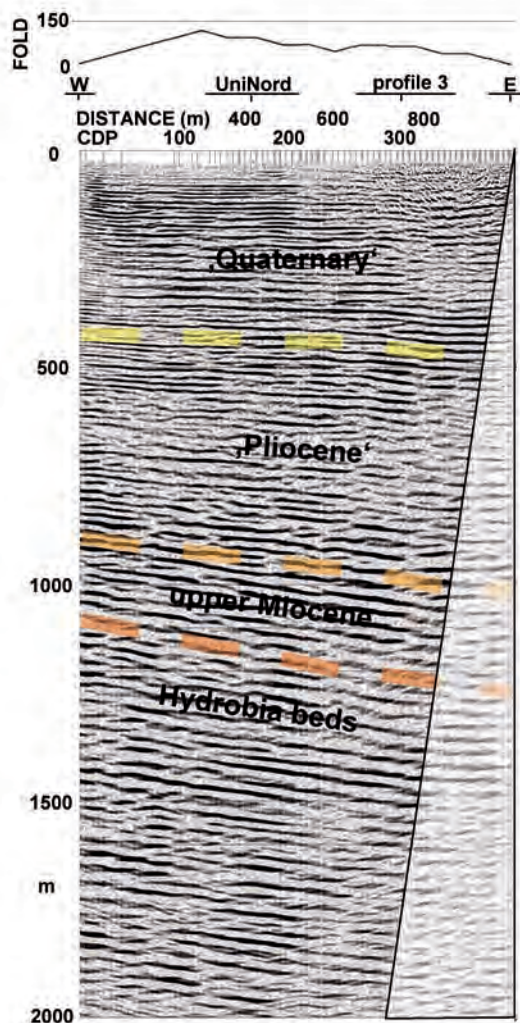


Fig. 13: Migrated seismic reflection profile 5. No seismic information is available inside the transparent triangle for geometric reasons.

Abb. 13: Migriertes reflexionsseismisches Profil 5. Aufgrund der geometrischen Verhältnisse sind innerhalb des transparenten Dreiecks keine seismischen Informationen verfügbar.

In the 'Heidelberg UniNord 1' borehole in 2006 (ELLWANGER et al. 2008), a vertical seismic profile (VSP) was recorded (Fig. 14), as well as numerous other geophysical logging methods (HUNZE & WONIK 2008). To ensure comparability with the reflection seismic profiles, the same vibrator and the same field parameters

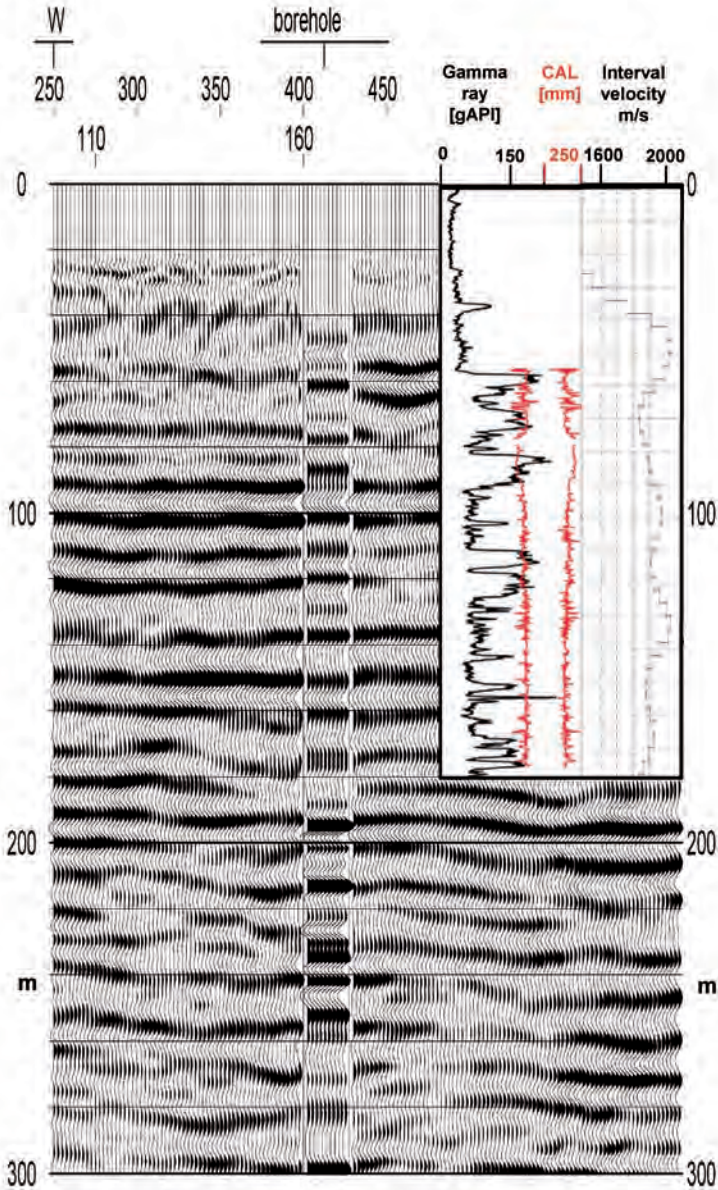


Fig. 14: Detail of seismic profile 5 (cf. Fig. 13) with corridor stack of the VSP (borehole location UniNord 1) inserted. Upper right: gamma ray log, calliper log (after HUNZE & WONIK 2008), and interval velocities deduced from the VSP. Reference level is surface level at the borehole location (107 m). Depth conversion is based on VSP-derived velocities down to 180 m depth.

Abb. 14: Ausschnitt aus dem seismischen Profil 5 (vgl. Abb. 13) mit dem Korridor Stack des VSPs (Bohrlokation UniNord 1). Oben rechts: Gamma Log (nach HUNZE & WONIK 2008), Bohrlochdurchmesser sowie die aus dem VSP abgeleiteten Intervallgeschwindigkeiten. Bezugsniveau ist die Ansatzhöhe des Bohrpunktes (107 m). Die Tiefenkonvertierung basiert auf den aus dem VSP bis 180 m Tiefe abgeleiteten Geschwindigkeiten.

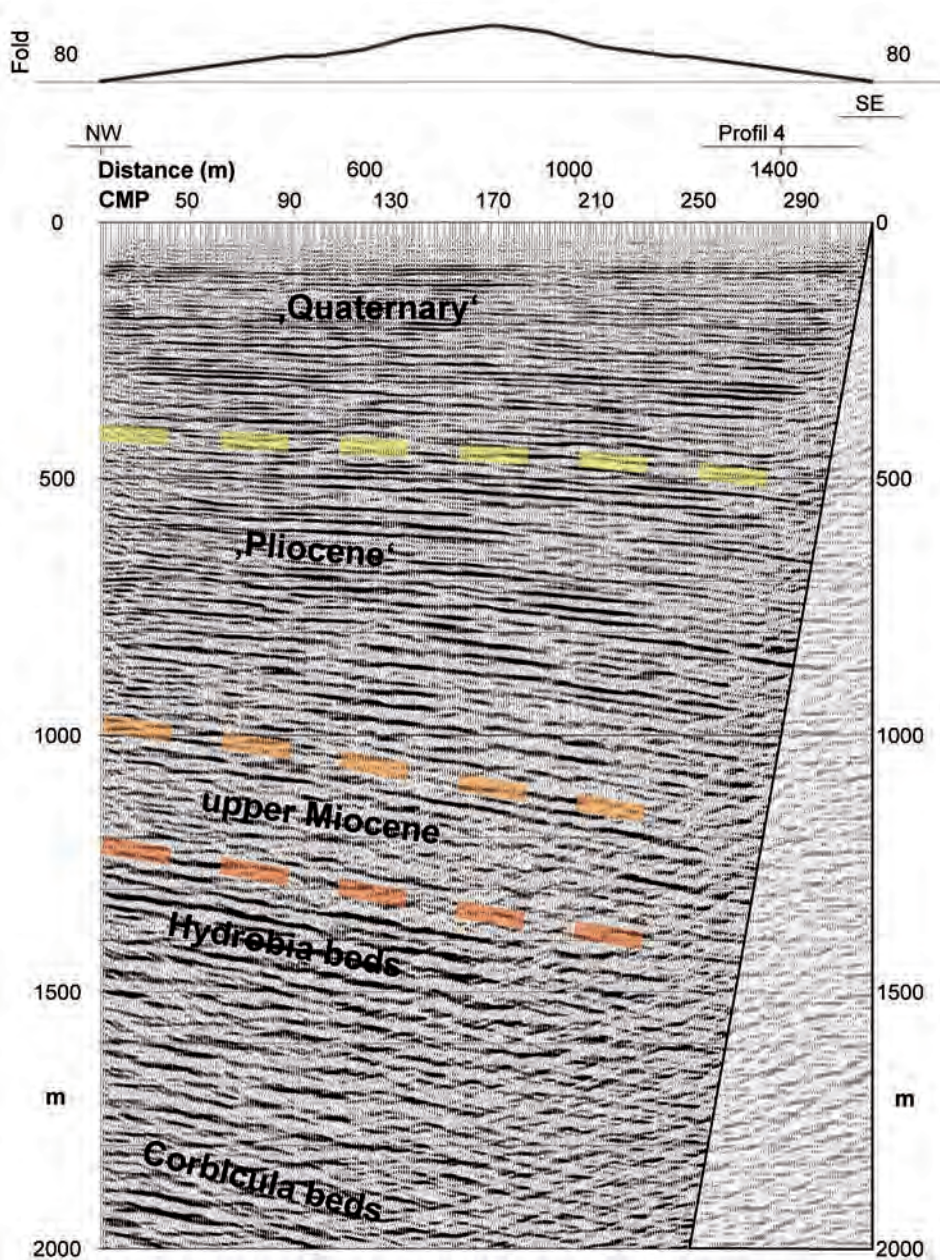


Fig. 15: Migrated seismic reflection profile 1. No seismic information is available inside the transparent triangle for geometric reasons. The strongly northwest-dipping reflectors close to the triangle zone are probably artefacts caused by migration.

Abb. 15: Migriertes reflexionsseismisches Profil 1. Aufgrund der geometrischen Verhältnisse sind innerhalb des transparenten Dreiecks keine seismischen Informationen verfügbar. Die stark nach Nordwesten einfallenden Reflektoren unmittelbar neben diesem Dreieck stellen wahrscheinlich durch die Migration verursachte Artefakte dar.

were used (Table 2). A depth interval from 180 m to 32 m could be measured with a recording distance of 4 m. Several surveys were dependant on the accessible openhole interval. Signal quality is very good and allows an unequivocal determination of traveltimes and the locally-reflected wavefield.

Interval velocities vary between 1850 m/s and 2250 m/s, beside a zone of lower velocity near the surface. A correlation can be observed between the seismic velocity and other petrophysical parameters: low values of the gamma ray log (coarse-clastic material) correlate with high velocities and high values of the gamma ray log (fine-clastic material) with low velocities. The VSP-derived velocities resemble very closely the smoothed stacking velocities derived from profile 5.

The VSP corridor stack represents the locally-reflected wavefield and corresponds with the surface reflection profiling results. Small discrepancies, as seen in Fig. 14, can be explained by different field geometries and recorded frequencies. The corridor stack was used to calibrate the total static of the reflection seismic lines, since the depths of its reflections are determined directly.

Heidelberg – the region south of the Neckar River

The new reflection seismic profiles 1 and 4 (Figs. 15, 16) are located south of the Neckar River (see Fig. 7). Therefore a tie to existing deep wells is hardly possible. The observed reflection patterns cannot be easily correlated with those of the hydrocarbon seismic lines, as assumed prior to the surveys. Therefore, the information from the Schriesheim well was correlated along several seismic lines and transferred to the nearest seismic profile recorded by the hydrocarbon industry, which is about 1,5 km south of profile 4. But the interpretation of the youngest sediments, e.g. Quaternary, remains especially uncertain, because these were not imaged well in the industrial seismic lines. Both profiles 1 and 4 intersect each other at an angle of about 50°. Again, on both profiles the

apparent dip of the sediment fill increases with depth. For Base 'Quaternary' it amounts to 3°, for Base Pliocene it amounts to 9° (profile 1) and 11° (profile 4). Therefore, real dip is about 4° for the Base 'Quaternary' and 11° for the Base Pliocene, in each case towards east. This is about twice as much as these beds north of the Neckar River have.

Similar to profile 3, Quaternary deposits show quasi-continuous reflections (e.g. profile 1 in 300 m depth), alternating with weak and partly sub-parallel reflections. Within the *Hydrobia* beds again a strong reflector occurs that is also visible on the other seismic sections.

Viernheim

In contrast to the seismic profiles recorded in the Heidelberg area the seismic lines recorded in the vicinity of the Viernheim borehole (Figs. 17, 18) allow a distinct classification of the sediment fill. Tops of zones of high reflectivity are visible at both depths of 220 m and 570 m, whereas especially the depth interval between 450 m and 570 m shows low reflectivity.

Considering the results of the research borehole Viernheim (HOSELMANN 2008) the reflector at a depth of 228 m can be assigned to the transition between the 'Quaternary' and the underlying material of local provenance (reference height of the seismic line is 100 m above sea level, the drilling site is 97 m above sea level). The limnic-fluviatile deposits of local provenance cause the observed high reflectivity.

Due to the high quality of the Viernheim data a complex fault pattern can be identified. Especially on the E-W oriented profile 6 (Fig. 17), a fault zone is observed that runs through all prominent reflection horizons and penetrates deep into the Rhenish facies, e.g. into 'Pleistocene' sediments. Originally a drilling location close to the intersection of profiles 6 and 7 was favoured. Based on these seismic results it was shifted by about 500 m towards south where the sediment succession was expected to be less affected by faults.

Due to the acquisition parameters and the processing of the seismic data, reflectors above

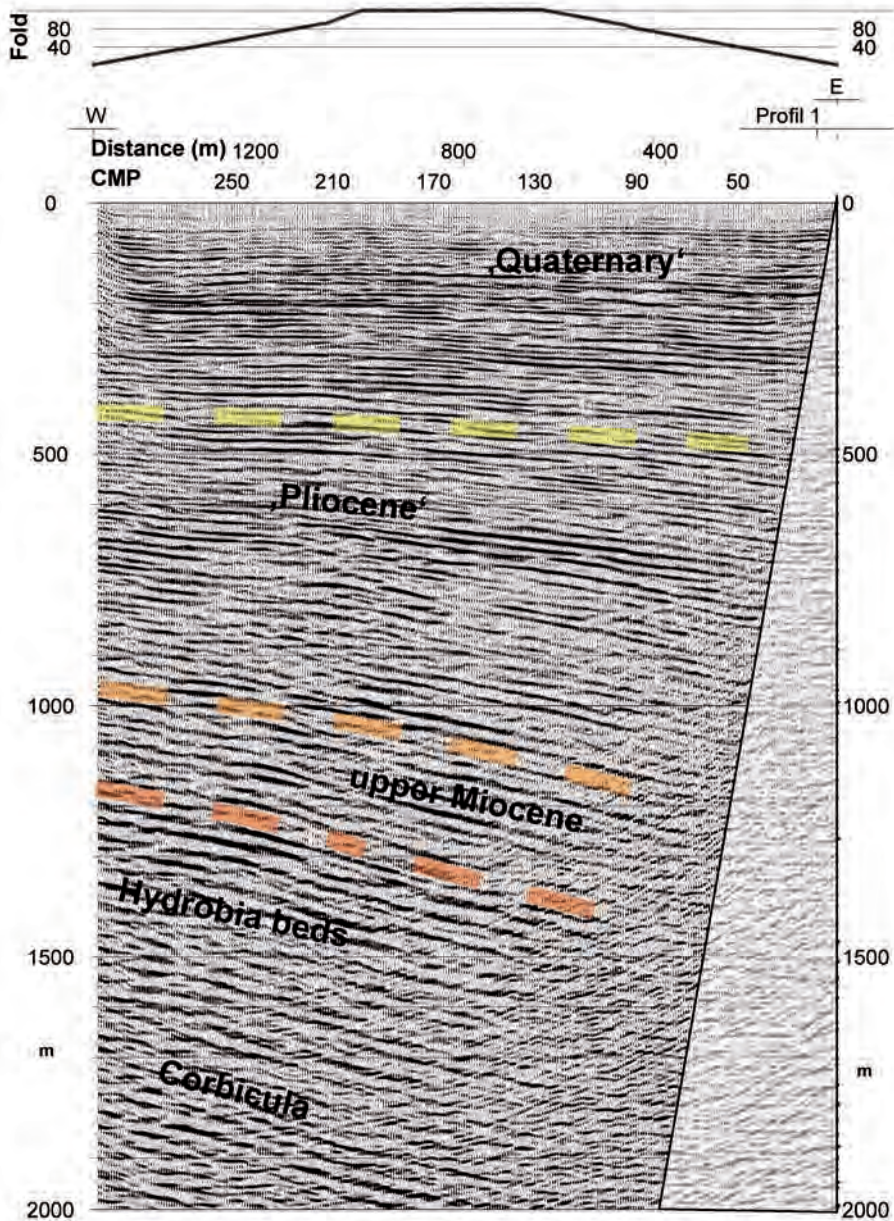


Fig. 16: Migrated seismic reflection profile 4. No seismic information is available inside the transparent triangle for geometric reasons. The strongly west-dipping reflectors close to the triangle zone are probably artefacts caused by migration.

Abb. 16: Migriertes reflexionsseismisches Profil 4. Aufgrund der geometrischen Verhältnisse sind innerhalb des transparenten Dreiecks keine seismischen Informationen verfügbar. Die stark nach Westen einfallenden Reflektoren unmittelbar neben diesem Dreieck stellen wahrscheinlich durch die Migration verursachte Artefakte dar.

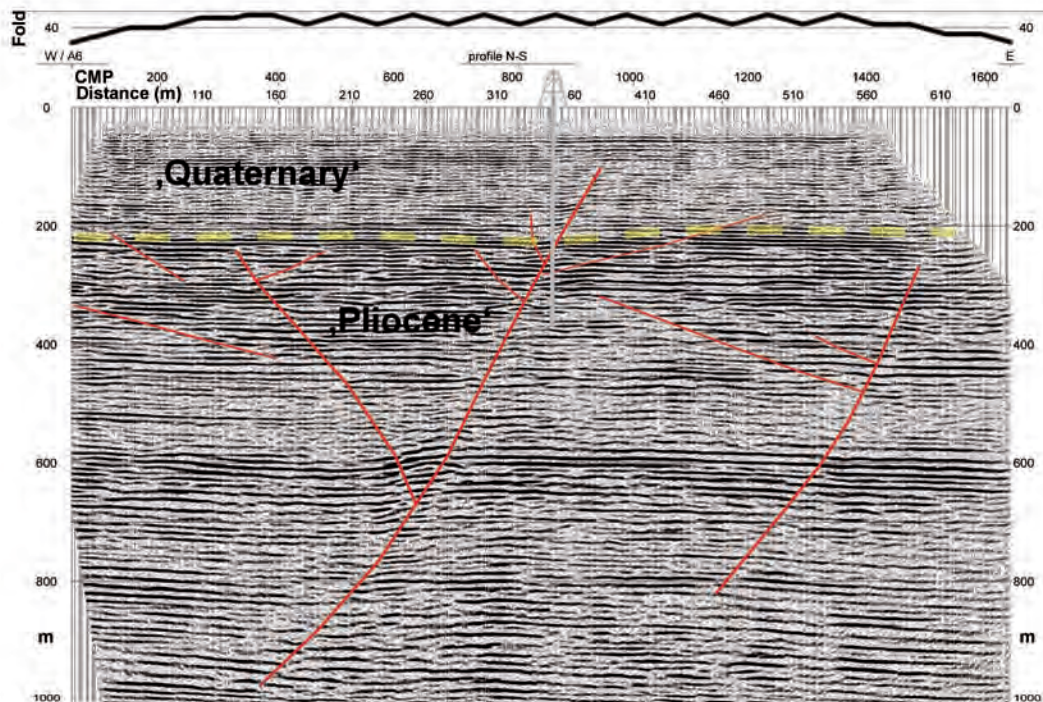


Fig. 17: Migrated seismic reflection profile 6 near Viernheim. The initially-proposed borehole location (marked by a derrick) was abandoned after the seismic became available to avoid the predicted faults. The location was then shifted about 500 m to the south (see Fig. 18).

Abb. 17: Migriertes reflexionsseismisches Profil 6 bei Viernheim. Der ursprünglich vorgeschlagene Bohrpunkt (Bohrturm) wurde aufgegeben, nachdem die Seismik verfügbar war, um ein Durchbohren der prognostizierten Störungen zu vermeiden. Der Bohrpunkt wurde sodann ungefähr 500 m nach Süden verschoben (vgl. Abb. 18).

35 m depth cannot be mapped. The first strong reflection that can be correlated along the entire profile is imaged at 40 m depth, two more are found at 80 m and 180 m depth. The uppermost two reflectors are most probably related to the top and the base of the Ladenburg Horizon, a prominent fine-grained horizon in the Upper Rhine Graben (in hydrostratigraphy named the 'Oberer Zwischenhorizont').

A precise and detailed stratigraphic interpretation of the reflectors below 570 m depth was not possible, because these were not reached by the research borehole. Instead we intend to achieve this by using industrial reflection seismic data, which is much denser than in the Heidelberg area.

6 Discussion and Conclusion

For the Heidelberg Basin thick sediment sequences are apparent, both in gravity and seismic data. Quantitative interpretation remain preliminary, as long as no deep boreholes can be included and 3-D models based on gravimetric and seismic data can be calculated, that additionally consider data from the hydrocarbon industry.

The seismic data at the locations of Viernheim and Heidelberg show quite different images, indicating varying sedimentological processes throughout the Heidelberg Basin. Confirming this, seismic measurements along the Rhine River (HAIMBERGER, HOPPE & SCHÄFER 2005, BERTRAND et al. 2006) show yet other seismic

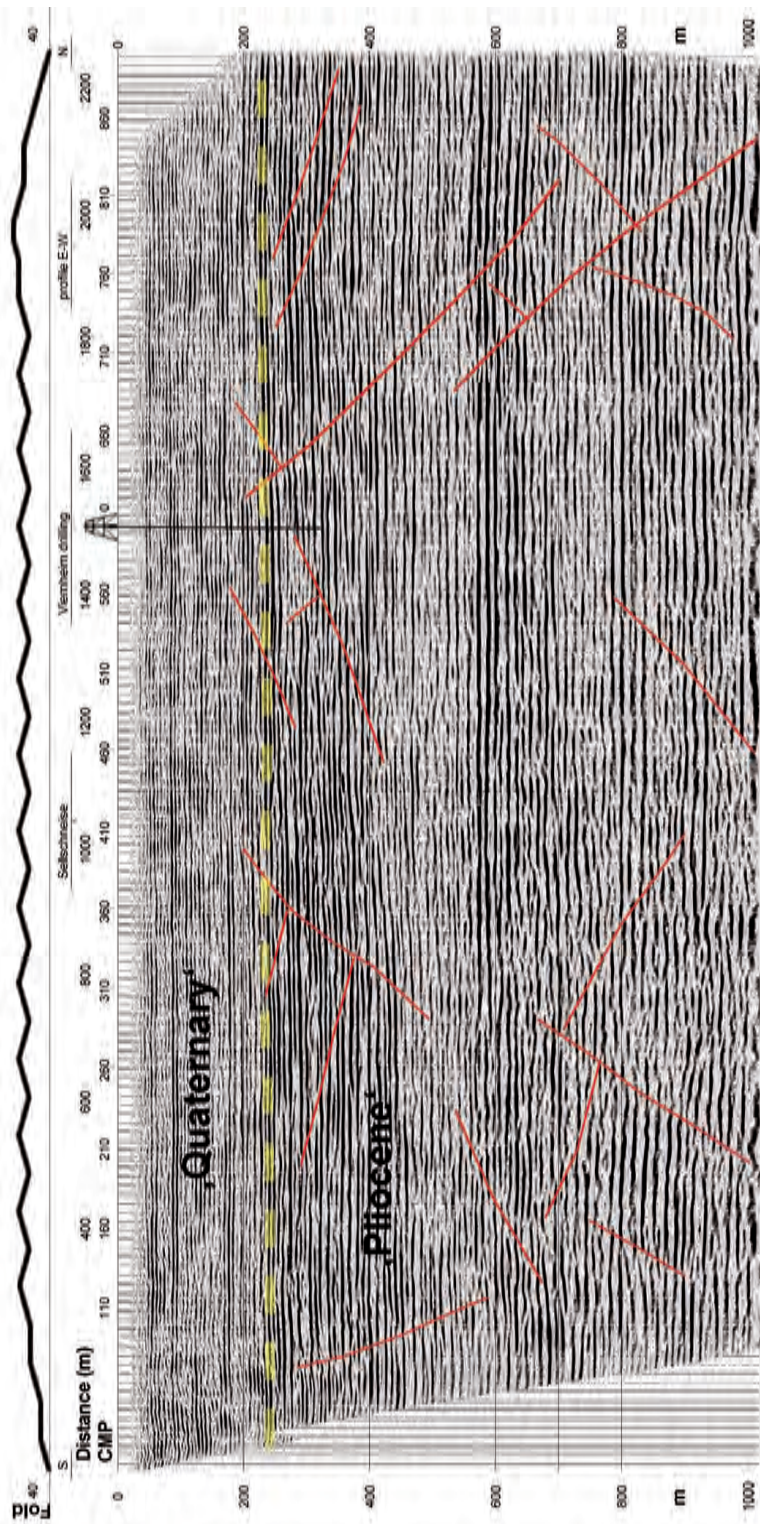


Fig. 18: Migrated seismic reflection profile 7. The Vierheim borehole location is indicated.

Abb. 18: Migriertes reflexionsseismisches Profil 7. Die Bohrlotlokation Vierheim ist eingezeichnet.

characteristics. Therefore a consistent seismic stratigraphy for the Plio-/Pleistocene sediments of the URG is not achievable within the limits of the presently applied inconsistent stratigraphic scenarios.

Concerning the location of the Viernheim boreholes, two new reflection seismic profiles image a sedimentary environment that is affected by tectonics, as documented by several faults. But these faults are mainly restricted to older sediments, e.g. to depths greater than about 225 m (Pliocene and older according to HOSELMANN 2008). In contrast, during the Quaternary the tectonic activity seems to be quiet. Along the entire profile these sediments are horizontal. But on a smaller scale much more detailed information is imaged by the two seismic lines indicating a complex depositional system, especially during Pliocene or even older times. Considering the results of the Viernheim research borehole, the top and the base of a fine-grained horizon, comprising the Ladenburg Horizon (Oberer Zwischenhorizont) at about 40 and 80 m, respectively, depths can be traced in the seismic section. Reflections at a depth of about 180 m can be correlated with a sequence of regional distributed fine-grained sediments (Unterer Zwischenhorizont).

The top of the uppermost section of high reflectivity can be correlated with the transition from the Rhenish facies (alpine) sediments to material with local provenance at a depth of 225 m, as revealed by heavy mineral analysis, carbonate content, and petrography (HOSELMANN 2008). Whether this seismic reflector can be interpreted as the transition Plio-/Pleistocene must be investigated by additional palynological studies that are not yet available.

The most important result derived from the seismic pre-site survey at the Heidelberg UniNord location is the existence of a sub-basin in the Heidelberg Basin close to the eastern margin of the Upper Rhine Graben. The additional subsidence adds up to some hundred meters with respect to deeper strata, as imaged on profile 3, e.g. about 400 m for the top of the *Hydrobia* beds. All recorded profiles do not show any disconformities, faults are

restricted to early Miocene (*Hydrobia* beds or older) units. However, this observation does not exclude potential hiatuses, which cannot be imaged by reflection seismic data.

By tying the new reflection lines to the Schriesheim well, the transition Pliocene to Pleistocene was predicted at depths between 400 and 500 m. This statement was based on the assumption that the extrapolation of information of the industry seismic lines across the gap between the northern end of the seismic profile 3 and the location of the Schriesheim borehole is correct. Furthermore using the interpretation of Base 'Quaternary' from a hydrocarbon well is uncertain because information is not available on the kind of data it is based on. Work done in the framework of hydrocarbon exploration was focused on deep structures rather than on young sediments.

The seismic lines reveal more or less continuous subsidence. The dip of sediments towards east increases with depth, showing a classical rollover anticline structure at the eastern URG master fault. The amount of subsidence increases from north to south: the Base Pliocene dips east by about 4.5 % on profile 2, 6 % on profile 5 and 11 % on profile 1 and 4. These values make a major fault zone beneath the Neckar River plausible, of which indications can be found at the southern end of profile 3. Differential compaction may have played a role by increasing the dip of the Miocene and younger strata. The rollover structure is not found to the north, as well as to the south of the 'Heidelberger Loch', as inferred from industry seismic profiles, which affirms it as the area with the most rapid subsidence.

Due to the thick sequences of Plio-/Pleistocene sediments, the research boreholes Heidelberg UniNord reveals a unique high temporal resolution. From the first Heidelberg borehole in 2006 a time marker of the Waalian stage is reported at 183 m – 210 m depth (HAHNE, ELLWANGER & STRITZKE 2008). As the age of the Waalian is considered to amount to 1.5 Ma, and assuming continuous sedimentation, the Top of the Pliocene at 2.6 Ma has to be expected at only 365 m depth. However, the preliminary interpretation of the new reflection seismic

profiles reveals Base 'Quaternary' first at about 430 m depth. Consequently, concerning the early Quaternary, increased sedimentation rates can be expected.

To aim on a deeper understanding of the basin genesis, a crucial point in the chronostratigraphic interpretation of seismic reflectors will be the proper definition of 'Base Quaternary'. A major step is also expected once the new drillings will be interpreted and correlated using the tools of sequence stratigraphy (ELLWANGER et al. 2008). We are convinced that this will also include a consistent seismic scenario.

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Sediment Input into the Heidelberg Basin as determined from Downhole Logs

SABINE HUNZE & THOMAS WONIK*

Abstract: The Upper Rhine Graben, and the Heidelberg Basin in particular, play an important role in the investigation of climate change and tectonic activity during the Tertiary and Quaternary periods. Several research boreholes were recently drilled to acquire data for a new interpretation of the geology of the northern Upper Rhine Graben. This paper investigates in detail the boreholes at Heidelberg, Viernheim and Ludwigshafen-Parkinsel, as well as the shallower boreholes at Pfungstadt, Stadtwerke Viernheim and Hüttenfeld, in terms of their geophysical parameters. The physical properties of the lithologies described in the cores are characterised on the basis of borehole logging data. A hole-to-hole correlation between adjacent boreholes is then conducted, using the characteristic changes in the 'natural radioactivity' parameter to acquire information on changes in sediment provenance (Rhine, Neckar, Pfälzerwald and Odenwald). An interpretation applying the statistical method of cluster analysis allows identification of sections with homogenous physical properties from downhole measurements and thus the determination of possible sediment provenance.

[Interpretation des Sedimentationsgeschehens im Heidelberger Becken anhand von Bohrlochmessungen]

Kurzfassung: Der Oberrheingraben und insbesondere das Heidelberger Becken spielen eine Schlüsselrolle bei der Untersuchung der Änderungen im Klima und der tektonischen Aktivitäten im Tertiär und Quartär. In den letzten Jahren wurden einige Forschungsbohrungen abgeteuft, um Daten für eine neue Interpretation der Geologie des nördlichen Oberrheingrabens zu erhalten. Im Rahmen dieser Arbeit werden die Bohrungen Heidelberg, Viernheim und Ludwigshafen-Parkinsel sowie die flacheren Bohrungen Pfungstadt, Stadtwerke Viernheim und Hüttenfeld anhand der dort durchgeführten geophysikalischen Bohrlochmessungen näher untersucht. Die physikalischen Eigenschaften der einzelnen an den Kernen beschriebenen Lithologien werden mit Hilfe der Daten der Bohrlochmessungen charakterisiert. Anschließend erfolgt eine Korrelation zwischen benachbarten Bohrlöchern, um aus den charakteristischen Änderungen im Parameter 'natürliche Radioaktivität' Aussagen zur Änderung der sedimentären Liefergebiete (Rhein, Neckar, Pfälzerwald und Odenwald) zu treffen. Eine Auswertung mit der statistischen Methode der Clusteranalyse ermöglicht es, aus den Bohrlochmessungen Bereiche mit einheitlichen physikalischen Eigenschaften zu finden und damit die möglichen sedimentären Liefergebiete einzugrenzen.

Keywords: Heidelberg Basin, downhole logging, hole-to-hole correlation, sediment provenance

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1 Introduction

1.1 Geology

One of Europe's largest river systems, the River Rhine, provides a unique geoscientific data set with the potential of bridging the gap between glaciated alpine areas and the inland ice-sheet advances of Northern Europe (WESTERHOFF 2008). The Upper Rhine Graben extends approximately 300 km from Basel (Switzerland) to Frankfurt (Germany) and is 35–45 km wide on average. This graben is a north-northeast-trending rift of Tertiary age (ELLWANGER et al. 2005). As a subsidence structure it forms an element of the Oligocene and Neogene rifting of the Upper Rhine Graben, representing 40 Ma of active graben tectonics (BEHRMANN et al. 2003). The Oberrhein (Upper Rhine) Valley, as a graben structure, forms part of the rifting system that began to develop during the mid-Tertiary (PREUSSER 2008). During the Quaternary, the subsiding part of the northern Upper Rhine Graben acted as a distal and final accommodation space for coarser alpine material (ELLWANGER et al. 2005).

Rhine sediments have recorded changes in both climate and tectonic activity (PREUSSER, 2008). During the Pliocene-Quaternary, the major subsidence centre shifted towards the eastern part of the graben (Heidelberg Basin), with a zone of maximum subsidence located in the central-eastern part of the basin, around the city of Heidelberg (ELLWANGER et al. 2005). The Pliocene-Quaternary infill of the Heidelberg Basin was mainly deposited by the River Rhine. The most distal signals of alpine climate dynamics associated with the major events of alpine glaciation can still be identified as sediment bodies within the Pleistocene succession. They are embedded in relatively fine alpine material and coarser local material. Coarser layers are also present, which are related to sediment input from the River Neckar (ELLWANGER et al. 2005). Towards the north, the character of the graben sediments change as a result of sorting processes during fluvial transport and admixture with local material derived from

the graben margins. In the northern part of the Upper Rhine Graben the graben sediments are generally finer grained, better sorted and mixed with local sediment input from the graben margins (HAGEDORN & BOENIGK 2008).

1.2 Boreholes

Recently, numerous boreholes have been sunk to acquire new data for reinterpreting the geology of the northern Upper Rhine Graben. These research boreholes are at Heidelberg, Viernheim and Ludwigshafen-Parkinsel (hereinafter called Ludwigshafen for short); shallower local boreholes are located at Pfungstadt, Stadtwerke Viernheim and Hüttenfeld (Figure 1 and Table 1). The borehole at Viernheim documents the conditions at the centre of the basin and those in Ludwigshafen and Heidelberg the western and eastern margins of the basin facies. Ludwigshafen and Viernheim represent the succession of Rhine sediments, but both also include signals from non-rhine sedimentation: small rivers from Pfälzerwald in Ludwigshafen, Neckar sediments in Viernheim. The borehole in Pfungstadt is located at the northern margin of the Heidelberg Basin.

The most significant parameters of the investigated sites are given in Table 1. The geological surveys of Hessen, Baden-Württemberg and Rheinland Pfalz, respectively, are in charge of the investigated boreholes. The drilling depth varies significantly between the sites: the deepest boreholes are at Viernheim and Ludwigshafen (350 m and 300 m respectively) and the shallowest at Hüttenfeld (99 m). Overall core recovery is very high (81–99 %). The only exception is the borehole at Hüttenfeld, where only cuttings were analysed and documented. The depth reached by downhole measurements differs from the total depth due to borehole and technical problems.

The position of the Pliocene/Pleistocene boundary within these deposits, as well as the entire stratigraphy of the Upper Rhine Graben, is rather controversial (cf. ELLWANGER et al. 1995; FETZER et al. 1995; GIBBARD 2004; GIBBARD et al. 2005; CLAGUE 2006). The boundary

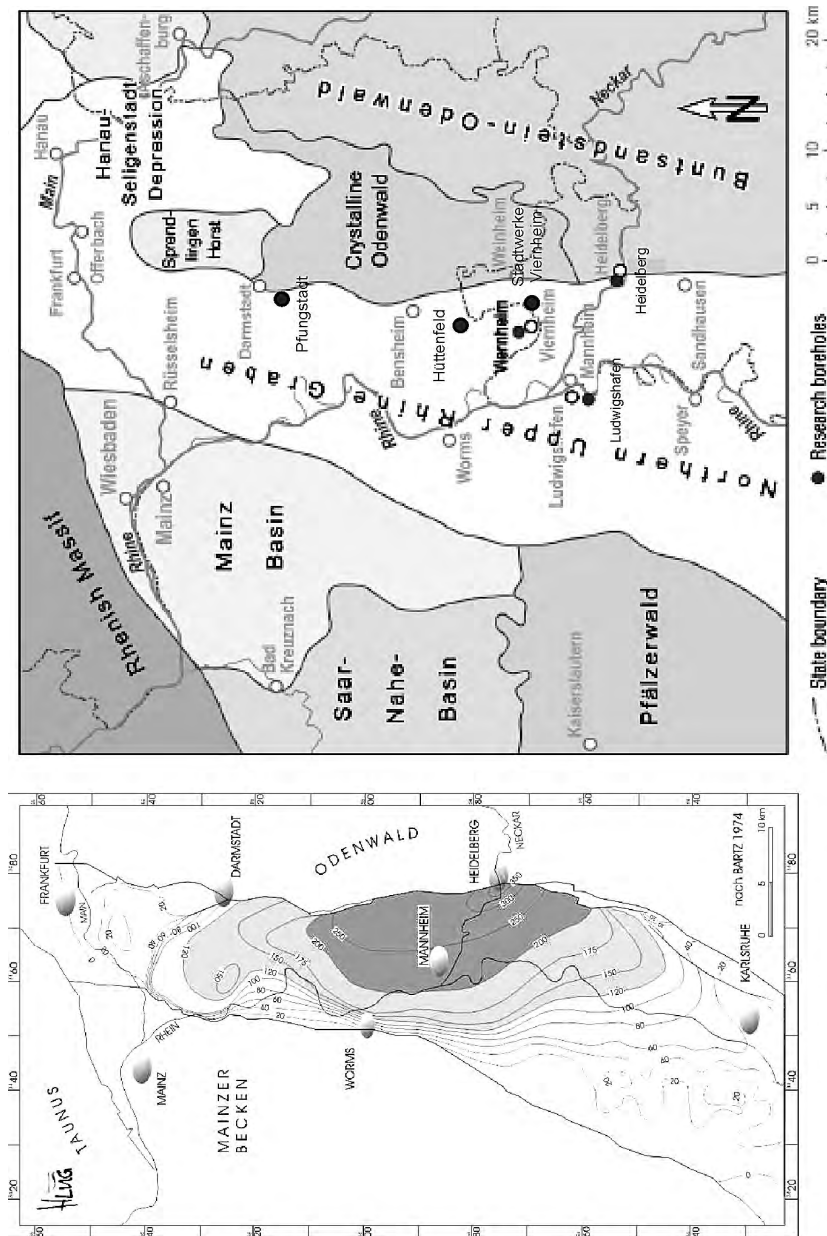


Fig. 1: Location of drill sites in the Heidelberg Basin. The main geological areas (Upper Rhine Graben, Odenwald, Pfälzerwald) and the main rivers (Rhine and Neckar) are marked (after HOSELMANN 2008). The small rectangle (left) comprises the sediment thicknesses of Quaternary age in the northern Upper Rhine Graben (after BARTZ 1974).

Abb. 1: Lage der Bohrlokationen im Heidelberger Becken (rechts). Die wichtigsten geologischen Gebiete (Oberrheinischer Graben, Odenwald, Pfälzerwald) und die wichtigsten Flüsse (Rhein und Neckar) sind markiert (nach HOSELMANN 2008). Das Rechteck (links) enthält eine Darstellung der Sedimentmächtigkeit im Quartär im nördlichen Oberrheinischen Graben (nach BARTZ 1974).

Table 1: Basic parameters of the investigated boreholes.

Tab. 1: Grundlegende Parameter der untersuchten Bohrungen.

	Pfungstadt	Hüttenfeld	Viernheim	Stadtwerke Viernheim	Heidelberg	Ludwigs- hafen
Responsible operating institution	Geological Survey (HLUG) Hessen	Geological Survey (HLUG) Hessen	Geological Survey (HLUG) Hessen	Geological Survey (HLUG) Hessen	Geological Survey (LGRB) Baden-Württemberg	Geological Survey (LGB-RLP) Rheinland-Pfalz
Drilling depth	200 m	99 m	350 m	110 m	180 m	300 m
Core recovery	98 %	Drill cuttings	97 %	99 %	81 %	99 %
Basis of Quaternary	At 132 m	Not recovered	At 225 m	Not recovered	Not recovered	At 177 m
Downhole logging	To 180 m	To 75 m, only GR	To 238 m	To 108 m, only GR	To 180 m	To 300 m, only GR

between Pliocene and Quaternary strata has been defined by the first occurrence of alpine input into the graben as indicated by the carbonate content within the sediments, as well as by the changing distribution of heavy minerals. The Pliocene sediments in the graben zone are predominantly fine-grained. Sandy clays alternate with fine to medium sands and some peat layers. Gravel layers occur only sporadically (HAGEDORN & BOENIGK 2008).

2 Methods

2.1 Downhole Logging

In the majority of boreholes downhole logging was conducted by the Leibniz Institute for Applied Geophysics (LIAG), Hannover; an exception is Hüttenfeld, where downhole logging was performed by the Geological Survey of Hessen (HLUG). The number of measured geophysical parameters at each site differs significantly: the most complete borehole measurement data sets were obtained at the Heidelberg and Viernheim sites, but Pfungstadt comprises a slightly reduced number of downhole measurements. At

the Ludwigshafen, Stadtwerke Viernheim and Hüttenfeld sites only gamma ray logs (GR) were run.

The downhole logging in Heidelberg, Viernheim and Pfungstadt was conducted in several sections because of problems within these less lithified sediments during drilling. This means that up to five logging campaigns were necessary to complete downhole logging. The borehole diameter generally decreases in stages (e.g. from 244 mm to 200 mm to 150 mm) with increasing depth. The steps coincide with each logging campaign.

The downhole measurements can be differentiated into radioactive methods (density, neutron porosity, spectral gamma ray including gamma ray, potassium, thorium and uranium); acoustic methods (sonic velocity and acoustic borehole televiewer); electrical methods (dual laterolog resistivity, dipmeter); magnetic methods (magnetic susceptibility); and other methods (borehole diameter, temperature and salt content of the drilling mud).

Only those logs yielding information on the physical properties of the sediments, in particular on their grain size, were utilised for further

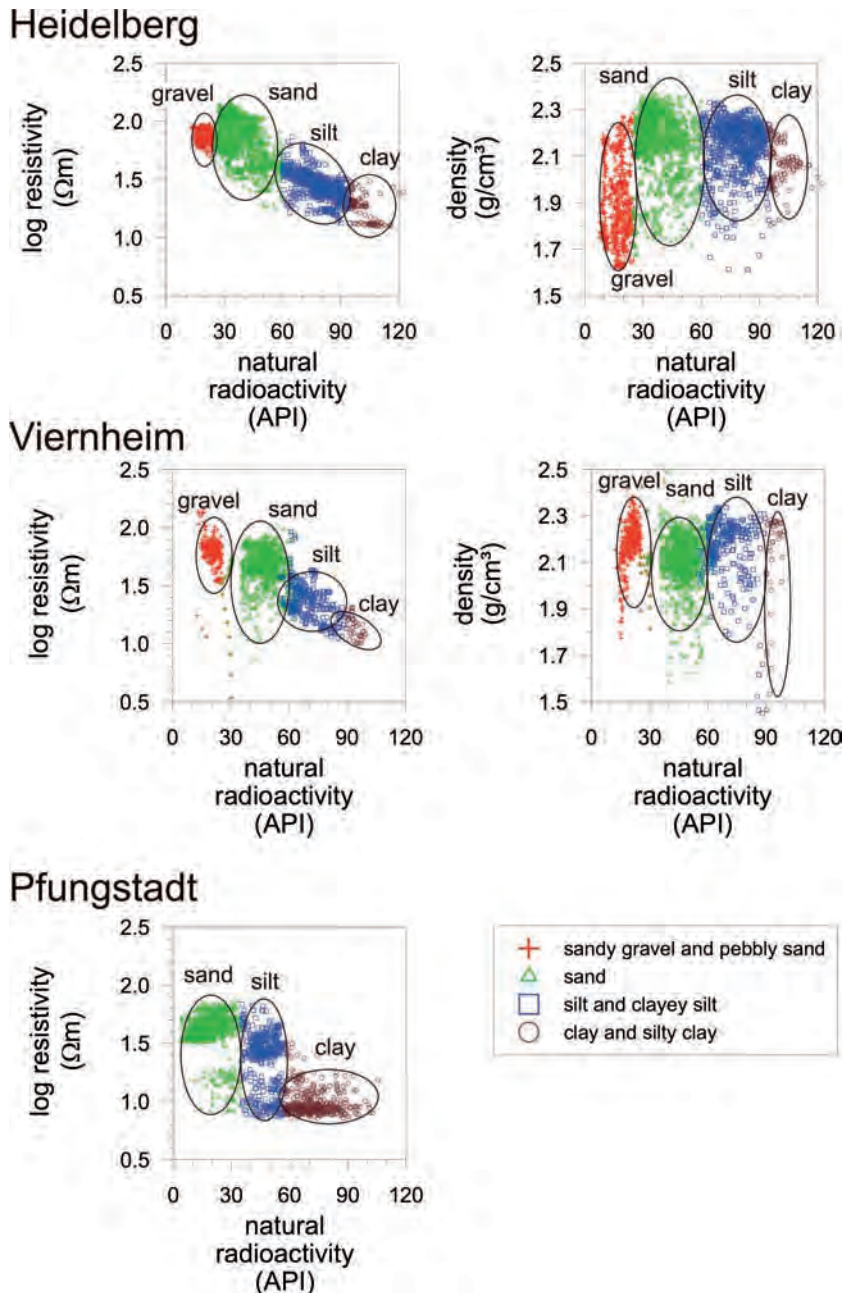


Fig. 2: Crossplots of the parameters natural radioactivity vs. resistivity and vs. bulk density for the drill sites Heidelberg, Viernheim and Pfungstadt. The four lithologies differentiated from the cores (sandy gravel; pebbly sand; sand, silt and clayey silt; clay and silty clay) are marked.

Abb. 2: Crossplots der Parameter natürliche Radioaktivität gegen Widerstand bzw. natürliche Radioaktivität gegen Dichte für die Bohrlokationen Heidelberg, Viernheim und Pfungstadt. Darin eingetragen ist die Differenzierung der vier Lithologien der Kerne (sandiger Kies und kiesiger Sand, Sand, Schluff und toniger Schluff sowie Ton und schluffiger Ton).

interpretation. These logs are GR, density, neutron porosity, resistivity, susceptibility, and caliper (borehole diameter). The caliper reflects the actual borehole diameter during logging as compared to the drilling diameter. Any sections with high differences in borehole diameter must be interpreted with caution. The neutron porosity log measures only relative porosity values; no absolute porosity values can be given. Therefore, the unit a.u. is used below.

2.2 Physical Properties

The main objective of analysing physical properties is to characterise each lithology by its specific physical properties. For better comparison the mean value and the standard deviation for each lithology has been calculated (Table 2 and Appendix). The log data sets were corrected prior to interpretation; this includes deleting erroneous and negative values. Those sections preferably with homogeneous lithology as determined by core descriptions (HOSELMANN 2008; WEIDENFELLER & KNIPPING 2008) are then selected and corrected using crossplots. Cross-plotting logs is usually done to quantify lithology and empirical relationships often become evident (RIDER, 1996). Finally, the physical properties of each lithology are calculated from the most prominent downhole logs, which are GR, density, porosity, resistivity (far) and susceptibility.

2.3 Hole-to-hole Correlation

Changes in sediment thickness, and therefore in sediment input, can be quantified by hole-to-hole correlation. This method comprises the synoptical comparison and connection of similar characteristic peaks and downhole log trends from adjacent sites. The correlation was performed using several logs (such as GR, susceptibility, porosity, density and resistivity), but most logs, except the GR log, were not measured at all sites and/or data variation is high. The correlation was therefore carried out on the GR log (Figures 3, 4 and 5), because of its low data scatter and characteristic log

trends. RIDER (1996) pointed out that the GR log is used for correlation, because the 'character' of the gamma ray log is repeatable, is not affected by compaction with depth, and gives some indication of lithology.

2.4 Cluster Analysis

The lithologies and resultant possible sediment provenances were determined using the statistical method of cluster analysis (Ward method of complete hierarchical linkage, Figure 5). The WINSTAT Statistic for Windows (Version 3.1) software application was employed for this purpose.

Cluster analysis is a multivariate statistical technique, which assesses the similarities between units based on the occurrence or absence of specific components within them. This analysis creates homogeneous (members are similar to one another) groups of variables, the clusters. The elements in a cluster have relatively small distances from each other and relatively larger distances from elements outside a cluster (DAVIS 1986; BACKHAUS et al. 1996). Cluster analysis differentiates groups characterised by their physical properties. The number of clusters is determined using a tree-like structure, the dendrogram, which visualises the similarity of clusters (MOLINE et al. 1992, FRICKE & SCHÖN 1999). The dendrogram is cut horizontally at any level to create groups, which are compared to core data, and the most appropriate grouping level is chosen (RIDER 1996).

The cluster analysis is performed for four locations simultaneously, allowing a comparison of the resulting clusters between the drill sites. The maximum number of parameters used for calculating the clusters in Heidelberg and Viernheim is six (Table 2). However, the number of logging parameters is reduced at the two other locations: in Pfungstadt four parameters were measured and in Ludwigshafen only one. Cluster analysis was not performed for the Hüttenfeld and Stadtwerke Viernheim boreholes, because of an insufficient number of measured geophysical parameters.

Table 2: Overview of the logged parameters at each drill site. x: this parameter was logged at this site. -: this parameter was not logged.

Tab. 2: Überblick über die gemessenen Parameter an jeder Bohrlokation. x: der Parameter wurde an dieser Bohrlokation gemessen. -: der Parameter wurde nicht gemessen.

Drill site	SGR (API)	Density (g/cm ³)	Neutron porosity (a.u.)	Resistivity (Ωm)	Susceptibility (10^{-4} SI)	Borehole diameter (mm)
Pfungstadt	x	-	x	-	x	x
Viernheim	x	x	x	x	x	x
Ludwigshafen	x	-	-	-	-	-
Heidelberg	x	x	x	x	x	x

3 Results

3.1 Physical Properties

3.1.1 Crossplots

The crossplots of the most significant parameters characterising the physical properties of the cored lithologies are described (Figure 2): the most useful logs for this purpose are GR, resistivity, and density in Heidelberg and Viernheim, and GR and resistivity in Pfungstadt. The other locations did not use these logs and, hence, crossplots cannot be plotted. Four groups of lithologies are differentiated based on the description of core lithologies: (1) sandy gravel and pebbly sand, (2) sand, (3) silt and clayey silt and (4) clay and silty clay.

The natural radioactivity (GR) vs. resistivity crossplots from Heidelberg, Viernheim and Pfungstadt (Figure 2, left column) are compared. The crossplots of the three locations show almost the same log trends: a strong negative correlation between GR and log resistivity. There, (a) sandy gravel and pebbly sand and (b) sand are characterised by low GR values (0-55 API) and high log resistivity values (0.8-2.3 Ωm). However, the log resistivity values of sandy gravel and pebbly sand differ between boreholes: in Heidelberg the values are

slightly higher (1.3-2.3 Ωm) than in Viernheim (1.1-2.1 Ωm) and considerably higher than in Pfungstadt (0.8-1.9 Ωm). At all sites (a) silt and clayey silt and (b) clay and silty clay are characterised by high GR values (55-130 API) and low log resistivity values (0.8-1.8 Ωm). It is important to note that in Pfungstadt the downhole logs were measured within the liner and, as a result, the downhole logs, especially the GR values, are significantly lower. If this is corrected, the boundary values for each lithology are also corrected. However, the clay resistivity values for Pfungstadt are much lower than at the other sites.

The crossplots GR vs. density for Heidelberg and Viernheim (Figure 2, right column) are plotted. In Heidelberg, the density data differ between the sandy gravel and pebbly sand (1.6-2.2 g/cm³) and the sand, silt and clayey silt, and clay and silty clay lithologies (1.8-2.4 g/cm³). In contrast, almost all Viernheim lithologies display similar density data (1.8-2.4 g/cm³). An exception is sandy gravel and pebbly sand, which are characterised by slightly enhanced values (1.9-2.4 g/cm³). Unfortunately, no density data were recorded in Pfungstadt. One possible reason why sandy gravel and pebbly sand display lower density values in Heidelberg than in Viernheim may be a lesser degree of compaction in Heidelberg due to rapid sedimentation from the River Neckar. This conclusion coincides

Appendix: Physical properties (mean value and standard deviation) of the six investigated boreholes within the Heidelberg basin. The boreholes are sorted according to distance from the margin towards the centre of the Heidelberg Basin. The number of data points varies as a function of the logging parameter.

Pfungstadt	Sandy gravel, pebbly sand	Sand	Silt, clayey silt	Clay, silty clay
Number of data points	0	957-1000	197-522	420-428
GR (API)	-	20±7	47±6	72±10
Log resistivity (Ωm)	-	1.61±0.16	1.36±0.24	0.98±0.09
Log susceptibility (10^{-4} SI)	-	0.65±0.06	0.62±0.05	0.63±0.06

Ludwigshafen	Sandy gravel, pebbly sand	Sand	Silt, clayey silt	Clay, silty clay
Number of data points	828	4108	263	806
GR (API)	20±3	39±8	68±6	102±9

Hüttenfeld	Sandy gravel, pebbly sand	Sand	Silt, clayey silt	Clay, silty clay
Number of data points	737	450	163	47
GR (API)	24±5	37±4	59±6	77±7

Viernheim	Sandy gravel, pebbly sand	Sand	Silt, clayey silt	Clay, silty clay
Number of data points	325-466	1114-1165	261-324	38-110
GR (API)	20±3	47±7	70±8	109±15
Density (g/cm^3)	2.20±0.12	2.12±0.13	2.16±0.14	2.11±0.23
Neutron porosity (a.u.)	35±11	38±11	42±12	46±14
Log resistivity (Ωm)	1.75±0.29	1.60±0.36	1.37±0.16	1.15±0.09
Log susceptibility (10^{-4} SI)	0.65±0.26	0.80±0.43	0.87±0.67	0.71±0.45

Stadtwerke Viernheim	Sandy gravel, pebbly sand	Sand	Silt, clayey silt	Clay, silty clay
Number of data points	774	677	552	132
GR (API)	27±5	42±4	58±5	77±6

Heidelberg	Sandy gravel, pebbly sand	Sand	Silt, clayey silt	Clay, silty clay
Number of data points	409-1034	1584-1661	775-808	99-110
GR (API)	15±5	38±9	78±9	102±6
Density (g/cm^3)	1.90±0.17	2.16±0.13	2.14±0.11	2.06±0.09
Neutron porosity (a.u.)	41±18	33±5	38±5	43±8
Log resistivity (Ωm)	1.87±0.06	1.86±0.19	1.44±0.11	1.25±0.11
Log susceptibility (10^{-4} SI)	0.66±0.05	0.57±0.06	0.65±0.11	0.67±0.06

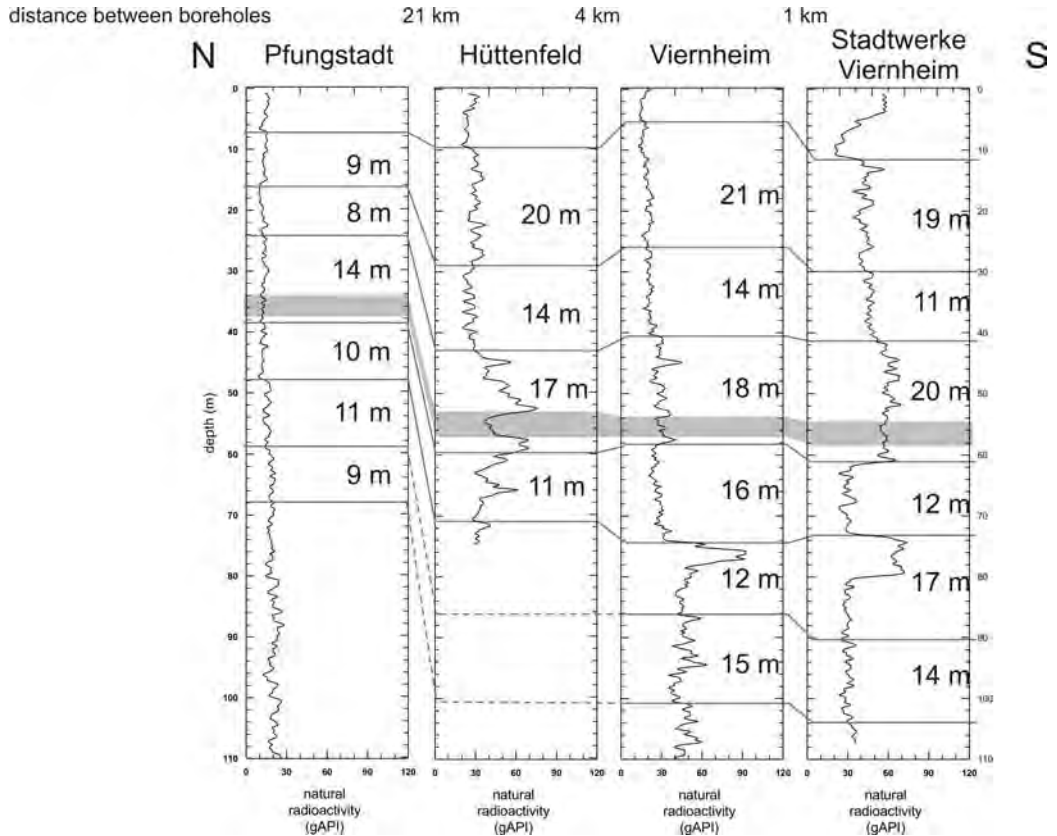


Fig. 3: Hole-to-hole correlation using the natural radioactivity log (smoothed) of the drill sites around Viernheim (Viernheim, Stadtwerke Viernheim and Hüttenfeld) and the Pfungstadt borehole. The grey horizontal lines mark the sediment package, characterised by its specific log trends and peaks. The grey boxes mark the detailed correlation of this randomly selected sediment package.

Abb. 3: Bohrloch-Bohrloch Korrelation unter Verwendung des Logs der natürlichen Radioaktivität (geglättet) der Bohrlokationen um Viernheim (Viernheim, Stadtwerke Viernheim und Hüttenfeld) sowie Pfungstadt. Die graue horizontale Linie markiert das Sedimentpaket, das charakterisiert ist durch seine besonderen Log-trends und Spitzen. Die grauen Kästchen markieren die detaillierte Korrelation dieser zufällig ausgesuchten Sedimentpakete.

with the highest sediment thickness at the basin centre. Another reason may be the mineralogy of the sediments at the different drill sites.

Comparing the results of both crossplots shows that the GR vs. resistivity crossplot implies that the fine-grained sediments are characterised by low resistivity values, which suggests high conductivity possibly caused by high water content, salinity and/or porosity. In the GR vs. density crossplot the coarser-grained sediments

are characterised by both low and high density values and the finer-grained ones by high density values. This suggests that the high density values in fine-grained sediments may be caused by mineralogy and/or a higher degree of compaction.

3.1.2 Statistics of Physical Properties

The physical properties of the Upper Rhine

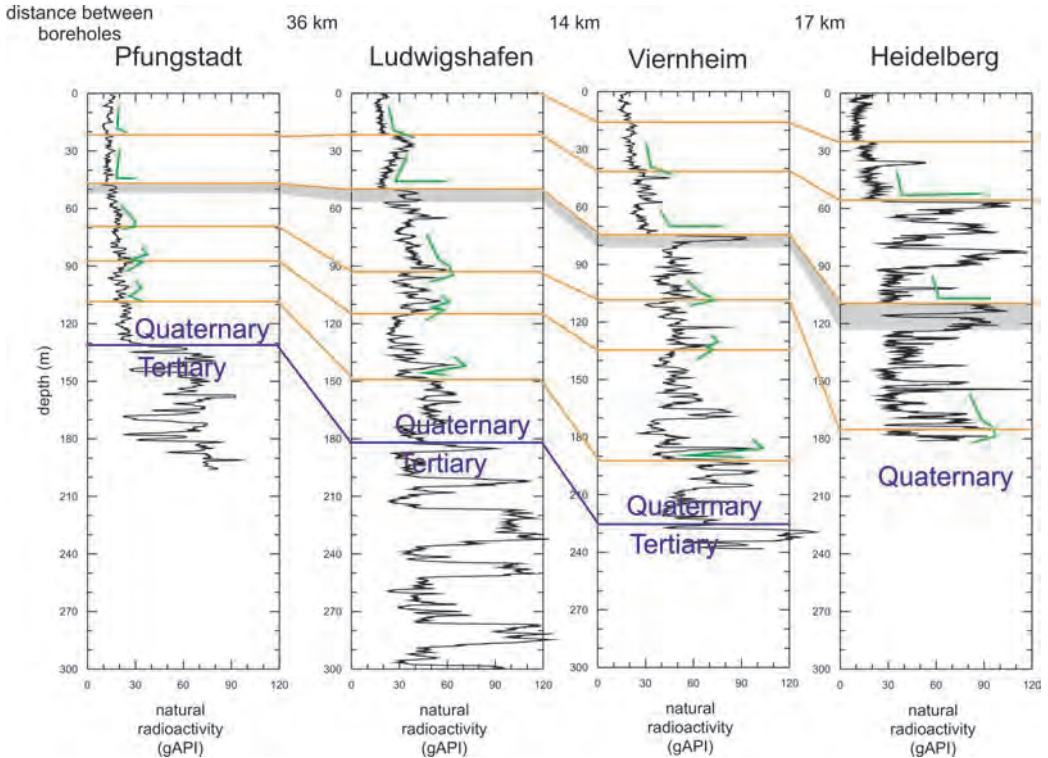


Fig. 4: Hole-to-hole correlation using the GR log from the drill sites within the Heidelberg Basin (Pfungstadt, Ludwigshafen, Viernheim, and Heidelberg). The lowermost blue horizontal lines mark the Pliocene-Pleistocene boundary as described in the cores. The red horizontal lines mark the sediment package, characterised by specific log trends (marked with vertical line) and peaks. The green lines mark the characteristic log trends. The grey boxes mark the detailed correlation of one randomly selected sediment package.

Abb. 4: Bohrloch-Bohrloch Korrelation unter Verwendung des GR Logs der Bohrlokationen im Heidelberger Becken (Pfungstadt, Ludwigshafen, Viernheim und Heidelberg). Die unterste blaue, horizontale Linie markiert die Pliozän-Pleistozän Grenze wie sie in den Kernen beschrieben wurde. Die anderen roten, horizontalen Linien markieren jeweils das Sedimentpaket, das charakterisiert ist durch seine besonderen Logtrends (markiert mit vertikaler Linie) und Spitzen. Die grünen Linien markieren die charakteristischen Logtrends. Die grauen Kästchen markieren die detaillierte Korrelation dieser zufällig ausgesuchten Sedimentpakete.

Graben were computed to determine both their characteristics and their changes according to location within the Heidelberg Basin (see Appendix). The physical properties are described using Viernheim as an example (Table 3). Principally, natural radioactivity reflects the grain size: the higher the natural radioactivity values, the higher the clay content of the sediment. The density and log resistivity values decrease and the neutron porosity values increase with increasing clay content (from sandy gravel to clay). Again, this may imply a decreasing degree of

compaction from the coarser-grained to the finer-grained sediments. The log susceptibility values vary with grain size, because this parameter mainly reflects an overall change in sediment input.

A number of important trends are observed by comparing the physical properties of all lithologies within the Heidelberg Basin (see Appendix). Overall, it must be kept in mind that the lithologies were defined for each site separately. This leads to more or less significant differences in GR values between

Table 3: Physical properties of the Viernheim sediments derived from logging data. The number of data points is given for quality control.

Tab. 3: Die physikalischen Eigenschaften der Sedimente in Viernheim, die aus den Bohrlochmessungen abgeleitet wurden. Die Anzahl der Messdaten wird zur Qualitätskontrolle angegeben.

Viernheim	Sandy gravel, pebbly sand	Sand	Silt, clayey silt	Clay, silty clay
Number of data points	325-466	1114-1165	261-324	38-110
Natural radioactivity (API)	20±3	47±7	70±8	109±15
Density (g/cm ³)	2.20±0.12	2.12±0.13	2.16±0.14	2.11±0.23
Neutron porosity (a.u.)	35±11	38±11	42±12	46±14
Log resistivity (Ωm)	1.75±0.29	1.60±0.36	1.37±0.16	1.15±0.09
Log susceptibility (10 ⁻⁴ SI)	0.65±0.26	0.80±0.43	0.87±0.67	0.71±0.45

the boreholes and the composition of each lithology varies among the drill sites. Sand is taken as an example: varying GR values at the different drill sites imply different amounts of fine-, medium- and coarse-grained sand and a varying amount of clay within the sediment matrix. This is valid for all four lithologies. However, the bulk density values of all drill sites are comparable, but the GR values differ significantly between Heidelberg and Viernheim. Reasons may be different mineralogical compositions caused by different sediment provenances.

3.1.3 Sediment Thickness

The crossplots facilitate a differentiation of lithologies. The thickness of the sediment in each lithology is determined from this (Table 4). The main objective is to quantify the changes in sediment composition from margin to centre in the Heidelberg Basin and from south to north in the Upper Rhine Graben. In Pfungstadt (at the northern margin of the basin) the drilled and logged sediments are mainly composed of sand (sediment thickness 100.0 m). Pfungstadt is located furthest north of all boreholes in the Upper Rhine Graben. Thus, input from the

Odenwald is high and less influenced by the River Rhine due to its distance from the Rhine Graben. This borehole contains the highest amount of clay and silty clay (45 m), which may be a result of its location at the northern margin of the basin with its fine-grained sediment input. In comparison, there are only small differences to the other drill site in the basin margin at Ludwigshafen. The lithology with the largest sediment thickness is sand (205 m), and the smallest is silt and clayey silt (13 m). Finally, the borehole at Heidelberg (at the centre of the Heidelberg Basin) is characterised by very thick sandy sediments (82 m) and much lesser clay and silty clay (4 m). These observations reflect the input of coarser-grained sediments from the River Neckar and almost no influence by the River Rhine.

Overall, it must be kept in mind that the total drilling depth and total logging depths differ locally due to borehole collapse. The most significant example is Hüttenfeld, where the total drilling depth amounts to 99 m and, in contrast, the total logging depth is only 75 m. This leads to incomplete analysis of lithologic composition, which underestimates the less compacted lithologies such as sand and gravel.

Table 4: Overview of sediment thicknesses in all boreholes differentiated according to the four cored lithologies. The boreholes are sorted according to distance from the margin to the centre of the Heidelberg Basin. The bold numbers are the highest and lowest values of each drill site, respectively.

Tab. 4: Überblick über die Sedimentmächtigkeiten an allen Bohrlokalationen, die entsprechend den vier in den Kernen erbohrten Lithologien differenziert wurden. Die Bohrlokalationen sind nach ihrer Entfernung vom Beckenrand bis zum Zentrum des Heidelberger Beckens sortiert. Die fett gedruckten Zahlen stellen die jeweils höchsten und tiefsten Werte für jede Bohrlokalation dar.

	Logging depth (m)	Sediment thickness (m)			
		Sandy gravel and pebbly sand	Sand	Silt and clayey silt	Clay and silty clay
Pfungstadt	198	0	100	53	45
Ludwigshafen	300	41	205	13	40
Hüttenfeld	75	38	24	9	4
Viernheim	238	53	133	38	15
Stadtwerke Viernheim	108	39	34	28	6.9
Heidelberg	175	50	82	39	4.1

3.2 Sediment Provenance

3.2.1 Viernheim

3.2.1.1 Hole-to-hole Correlation

Although Hüttenfeld and Pfungstadt are separated by 21 km, the resulting correlation between all four drill sites around Viernheim, as determined by hole-to-hole correlation, is good (Figure 3). The thicknesses of the line-marked sediment sections at the drill sites around Viernheim do not vary significantly, but decrease slightly towards Pfungstadt.

As an example, one randomly selected sediment package is picked and marked grey (Figure 3). Surprisingly, the thickness of this sediment section increases from Stadtwerke Viernheim and Viernheim to Hüttenfeld and Pfungstadt. The selected sediment section is composed of sand to silt and clayey silt and is about 2 m thick in Viernheim and Stadtwerke Viernheim. This increases to 2.5 m in Hüttenfeld, which may be a result of local sediment accumulation. On the

other hand, the two drill sites around Viernheim (Viernheim and Stadtwerke Viernheim) may be influenced by erosion or sediment compaction. The sediment thickness in Pfungstadt decreases slightly to 2 m, due to different, possibly local sediment input from Odenwald.

3.2.1.2 Cluster Analysis

The cluster analysis for the drill sites around Viernheim could not be performed due to the absence of several geophysical parameters measured downhole.

3.2.2 Heidelberg Basin

3.2.2.1 Hole-to-hole Correlation

Overall, there is good correlation between all four drill sites determined from hole-to-hole measurements (Figure 4), despite the large distances between the investigated sites (36 km between Pfungstadt and Ludwigshafen). As anticipated, the thickness of correlated sediment

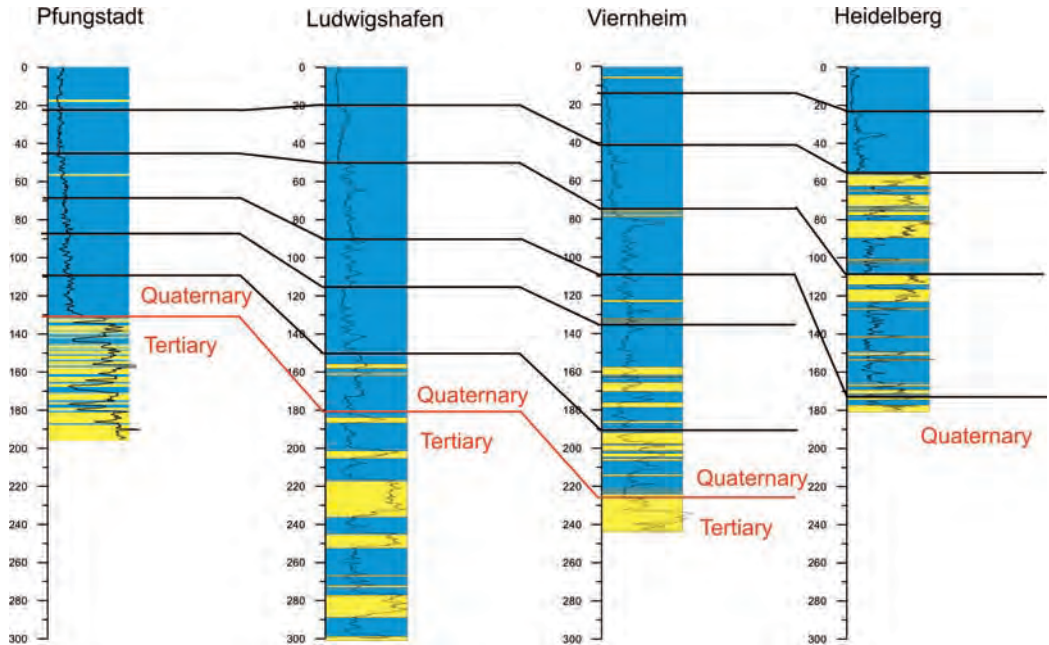


Fig. 5: Results of the cluster analysis of the Ludwigshafen, Viernheim, Pfungstadt, and Heidelberg boreholes. The characteristic GR log and the Pliocene-Pleistocene boundary for each site are plotted.

Abb. 5: Ergebnisse der Clusteranalyse der Bohrlokationen Ludwigshafen, Viernheim, Pfungstadt und Heidelberg. Darin sind das charakteristische GR Log und die Pliozän-Pleistozän Grenze für jede Bohrlokation dargestellt.

layers is greatest in Heidelberg, which is located in the subsidence centre of the Heidelberg Basin, and lowest in Pfungstadt located at the northern margin of the basin.

Again, one sediment package is chosen at random to determine the changes in sediment provenance (Figure 4). The following description of the sediment package is based on a profile from the two margins of the basin (Pfungstadt and Ludwigshafen) to its centre (Heidelberg). The log trends and the thickness of the chosen sediment package are quite similar in Pfungstadt and Ludwigshafen (sediment composition: sandy gravel and pebbly sand). In Viernheim, the sediments are fine-grained in the upper part and coarse-grained in the lower part. Thus, additional fine-grained input did occur. Finally, in Heidelberg, the sediment composition changes again: the sediments are mainly composed of clay and only small amounts of sand occur. Surprisingly, the log characteristics are quite si-

milar to the curve trends at Ludwigshafen, but with enhanced clay content. Sediment transport to Viernheim is influenced by a different sediment provenance (such as the River Rhine) or is blocked by a local sediment barrier.

3.2.2.2 Cluster Analysis

The clusters are computed for each drill site, separately defining the number of clusters (Figure 5). Overall, this number was defined as two, because the other clusters contained only a few data points or reflect erroneous values (e.g., at the top or the bottom of the borehole). The determined clusters were interpreted as different lithologies reflecting two different sediment provenances: The first cluster (dark coloured) with low GR (34 ± 13 API), high log resistivity ($1.60 \pm 0.33 \Omega\text{m}$) and low log susceptibility ($0.67 \pm 0.21 \cdot 10^{-4}$ SI) values reflects sediments rich in sandy gravel and pebbly sand to sand,

which probably originate from the River Neckar (proximal deposition from Odenwald). The second cluster (light coloured) with high GR (81 ± 20 API), low log resistivity ($1.29 \pm 0.45 \Omega\text{m}$) and high log susceptibility ($0.77 \pm 0.52 \cdot 10^{-4}$ SI) values mainly contains sediments rich in clay, which were probably deposited from the River Rhine (distal deposition from the Alps, Black Forest and Vogues). The other parameters (density, borehole diameter, and neutron porosity) have similar values for both clusters. Transferring the hole-to-hole correlation to the results of the cluster analysis shows clear differences in sediment provenances. These observations lead to the assumption that overall sediment depositions are similar, but differ in detail influenced by two different sediment provenances River Neckar (Cluster 1) and River Rhine (Cluster 2).

4 Conclusions and Discussion

The interpretation of downhole logs leads to the following conclusions: the physical properties of sediments derived from logs and cores can be quantified and the results compared. Four groups of lithologies can be differentiated using the natural radioactivity (GR) vs. resistivity and GR vs. density crossplots: (1) sandy gravel and pebbly sand, (2) sand, (3) silt and clayey silt and (4) clay and silty clay. The results of the GR vs. resistivity crossplots imply high conductivity possibly caused by high water content, salinity and/or porosity. In the GR vs. density crossplot, the coarser-grained sediments are characterised by both low and high density values and the finer-grained sediments (silt, clayey silt, clay and silty clay) by high density values. This suggests that the high density values in fine-grained sediments may be caused by mineralogy and/or a higher degree of compaction.

Interpretation of the hole-to-hole correlation displays a coincidence of sediment provenances around Viernheim (close to the centre of the Heidelberg Basin) and only small changes towards Pfungstadt (located at the northern margin of the basin). Furthermore, this correlation shows the anticipated changes in sediment

layer thickness from Pfungstadt (smallest thickness) to Heidelberg (greatest thickness at the basin centre). Additionally, the statistical method of cluster analysis confirms two main sediment provenances interpreted as deposits from the Rivers Rhine and Neckar. However, the results of the hole-to-hole correlation and the cluster analysis differ. This implies different contributions of a single sediment package to one of the two sediment provenances. The problem posed here is that both methods are based on assumptions and more interpretation is necessary: hole-to-hole correlation is a visual method, very detailed and based on the assumption that the GR log is the most significant log, because it mainly describes the sediment composition. On the other hand, the cluster analysis needs a predefined number of clusters and must be interpreted carefully.

More investigations are required to determine absolute ages in the investigated boreholes in order to confirm and/or critically test our visual and statistics-based hole-to-hole correlation and the interpreted sediment provenances. Sedimentological interpretations should be incorporated in our small-scale observations and expansion to a basin-scale analysis should follow.

Additionally, data acquired after deepening of the Heidelberg drill site in spring 2008 to an end depth of 500 m should be integrated in further studies, which are important in terms of the Tertiary-Quaternary boundary and the changes in sediment thickness at the drill sites.

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Pleistocene molluscs from research boreholes in the Heidelberg Basin

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Abstract: Cores cut in the research boreholes at Viernheim and Parkinsel P34 and P35 in Ludwigshafen were analysed to investigate their fossil content, and particularly the remains of molluscs. The selected material was suitable for reconstructing the palaeoclimatic conditions and simplifies the chronostratigraphic classification of individual beds. Two mollusc species and one rodent species from the Lower Pleistocene (Lower Biharium) were identified in the northern Upper Rhine Graben for the first time (in the Viernheim borehole). The fossils from the Lower Pleistocene sections of the Viernheim borehole are clearly related to the Uhlenberg fauna from Bavarian Swabia dated as Upper Villanium/Tegelen.

[Pleistozäne Mollusken aus Forschungsbohrungen im Heidelberger Becken]

Kurzfassung: Bohrkerne der Forschungsbohrungen Viernheim und Parkinsel P34 und P35 aus Ludwigshafen wurden auf ihren fossilen Inhalt, besonders auf Molluskenreste, untersucht. Das ausgelesene Material ist geeignet die paläoklimatischen Verhältnisse zu rekonstruieren und erleichtert die chronostratigraphische Einstufung einzelner Schichten. Zwei Molluskenarten und eine Nagetierart wurden erstmalig aus dem Altpleistozän (Altbiharium) der Bohrung Viernheim für den nördlichen Oberrheingraben nachgewiesen. Die aus den altpleistozänen Abschnitten der Bohrung Viernheim vorliegenden Fossilien weisen deutliche Beziehungen zu der in das Obere Villanium/Tegelen datierten Uhlenberg-Fauna aus Bayerisch-Schwaben auf.

Keywords: Upper Rhine Graben, Quaternary, Pleistocene, Arvicolidae, Mollusca, Stylommatophora, Pupilloidea, Gastrocoptinae

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1 Introduction

The mollusc fauna in the Upper Rhine Graben is very important for palaeoclimatology because most of the mollusc species are still present today in the flood plains of large valleys, and their habitats and lifestyles are known. They provide information on hydrological and climatic change, and landscape history, and can therefore provide “valuable pieces in the dating puzzle” (ENGESSER & MÜNZING 1991). Most endemic mollusc species already colonised our landscapes in the Upper Pliocene. In addition, the northern Upper Rhine Graben in the Pleistocene lay in an optimum development zone according to the description by LOŽEK (1969) of the climatically favourable zones of the Czech Republic and Southwest Germany. During the interglacials, the climate here meant that the mollusc fauna was much more habitat sensitive than that of northern Central Europe. During the periods of glaciation, the glacial ice did not extend into the Upper Rhine Graben area, which meant that the area had favourable conditions for the survival of animals, as well as molluscs. Loess steppes exist in the Upper Rhine area during the high glacial periods.

2 Research boreholes in the Heidelberg basin

2.1 Fossil material and depositional conditions

The thanatocoenosis of molluscs from a core only reflects a small piece of the overall landscape complex. It is therefore also important to look at the sediments in more detail. Fossils are often found in the argillaceous layers of the “Zwischenhorizonte” (intermediate horizons) as well as in the fine and medium-grained sands of the “Kieslager” (gravel beds). The grey sand horizons of the “Rhenish Facies” (HOSELMANN 2008) in the Lower Quaternary may contain open steppe fauna of the glacials and interglacials. The coarser the sediment, the poorer the preservation of the mollusc shells. The coarser gravels also contain few species because they

are formed during glacial conditions. An exception are the loess horizons which are, however, usually only found at the boundary to slope deposits. Loess horizons contain mollusc fauna which although species poor, contain characteristic high glacial species. These horizons are not found in the cores from the fluvial Rhine Graben. However, molluscs do occur in some sandy beds, which indicate high glacial conditions. The work of BARTZ (1959) is recommended for a short, though usually still valid subdivision of the Pleistocene in the northern Upper Rhine Graben, and for the description of the most important associated fossil localities – although the middle sandy sequence described in this paper is today classified as Middle Pleistocene (ENGESSER & MÜNZING 1991).

2.1.1 Condition of preservation of fossil molluscs

Complete gastropods and molluscs are only rarely preserved. This is possible in some of the clay lenses of the intermediate horizons as well as in a few hard “Terra Rossa beds” from Cromerian horizons. This also applies to small specimens in sand, finer gravel beds and flood sediments. In other cases, identification of species is based on the characteristic properties of the aperture armatures and the surface structures. The shells are sometimes corroded, polished or seriously fragmented by transport and the influence of water. We now know that the thickness of the shells does not provide any information on the climatic conditions: thick-shelled gastropods were also present during interglacials and are therefore not necessarily an indicator of a colder climate (GEISSERT 1967a).

2.1.2 The Oberer Zwischenhorizont (OZH)

The Oberer Zwischenhorizont (Upper Intermediate Horizon) is undoubtedly the most interesting level in all of the investigated boreholes because of the numerous mollusc remains and also because it contains sensitive interglacial species whose presence can be correlated with palynological analysis. Exceptions are the ar-

gillaceous peat beds from the low peat bogs of the Upper Pleistocene which mostly originated in periglacial periods. They are present in thin beds, e.g. in boreholes from around Darmstadt and Riedstadt (SCHWEISS 1988). At the west edge of the Rhine Graben, the minor thickness of the Obere Kieslager (Upper Gravel Horizon) of the OZH means that the OZH is already encountered at a depth of around 30 m. In addition, this OZH originated in the Middle Pleistocene as deduced from pollen analysis (KNIPPING 2004) and from the molluscs. The further to the east one looks at the Quaternary sequences, the thicker and more detailed they become. The Viernheim research borehole therefore includes several argillaceous intermediate horizons. The Lower Pleistocene in Viernheim is reached in the third, lower horizon. The Oberer Zwischenhorizont is characterised by beds of sandy, silty and argillaceous sediments. The thickness and nature of the OZH can fluctuate widely (ENGESSER & MÜNZING 1991).



Fig. 1: *Perforatella bidentata* (GMELIN, 1788), Gernsheim C00-BK1 at 88.4 m.

Abb. 1: *Perforatella bidentata* (GMELIN, 1788), Gernsheim C00-BK1 bei 88,4 m.

2.1.3 The Pliocene clays

Because of the absence of molluscs, the Pliocene samples provided no results in the Ludwigshafen boreholes or in the Viernheim borehole. Only a few rare shell remains were found which could not be identified because of the frequent strong corrosion of the fragments. Plant residues were however frequently found. Only very rare molluscs have been found previously in boreholes within Pliocene sediments of the southern Rhine Graben. (GEISSERT 1964, 1967b, 1980; NORDSIECK 1974; SCHLICKUM & GEISSERT 1980; WEDEL unpublished).

2.2 The Ludwigshafen boreholes (Rheinland-Pfalz)

The filling of the Rhine Graben with Quaternary sediments increases considerably to the east towards the edge of the Odenwald, so that the



Fig. 2: *Gastrocopta moravica oligodonta* (KROLOPP, 1979), Viernheim research borehole, 132.6-132.7 m.

Abb. 2: *Gastrocopta moravica oligodonta* (KROLOPP, 1979), Forschungsbohrung Viernheim, 132,6-132,7 m.

thickest Quaternary deposits are encountered around Heidelberg. The thickness is much thinner at the western margin of the Rhine Graben so that in borehole P35, the Tertiary boundary is already found at a depth of 183 m. The Quaternary is divided up into sequences of terraces built up by gravel horizons and intermediate horizons. Similar to palynological investigations, sampling concentrates on the dark, argillaceous intermediate horizons representing interglacial deposits.

2.2.1 Borehole P34

Borehole P34 (Ludwigshafen-Parkinsel, Tab. 1) has much less investigatable material than the other boreholes. The only malacological sample with any usable material comes from the first sample taken from the OZH at 21 m depth. The dark beds contain numerous mollusc remains. The high proportion of *Valvata cristata* (O.

F. MÜLLER, 1774) and *Gyraulus crista* (LINNAEUS, 1758) indicate swampy stagnant water conditions on the flood plains. 98 per cent of the species found are water molluscs. Of the few terrestrial gastropods found, they are all of hydrophilic species with the exception of *Clausilia* fragments transported in by water, and *Aegopinella* sp.. The thanatocoenosis is very similar to that of early Holocene flood plains in which flood plain clay is formed and Tschernozems and para brown earths can develop to maturity (LOŽEK 1964). The terrestrial molluscs found in the samples are of climate-independent species and therefore often not very useful for determining the temperature fluctuations. *Vertigo substriata* (JEFFREYS, 1833) and *Oxyloma elegans* (RISSE, 1826) are typical swamp and peat bog species and not necessarily restricted to calcareous soils, although the charophytes present (calcalgae) indicate such an environment. Pollen analysis of the pollen from the Oberer



Fig. 3: *Gastrocopta* n. sp. from the Viernheim research borehole, 174.3-174.4 m.

Abb. 3: *Gastrocopta* n. sp. aus der Forschungsbohrung Viernheim, 174,3-174,4 m.



Fig. 4: *Parafossarulus crassitesta* (BRÖMME, 1883), Ludwigshafen Maudach A36 borehole, 27.5-28.0 m OZH.

Abb. 4: *Parafossarulus crassitesta* (BRÖMME, 1883), Bohrung Ludwigshafen Maudach A 36 27,5-28,0 m OZH.

Zwischenhorizont in P34 analysed by KNIPPING (2004) confirmed its attribution to the Cromerian Complex (interglacial III or IV). This also matches the results reported by RÄHLE (2005) and the author for the boreholes in Mannheim and Ludwigshafen described below.

2.2.2 Borehole P35

The Quaternary is divided up into sequences of terraces built up by gravel horizons and intermediate horizons, whereby the uppermost “Oberer Zwischenhorizont” of borehole P35 begins at 19 m and does not end until 34 m. 5 samples were taken from this zone. It is followed by an 8-metre-thick “Unteres Kieslager”. This is underlain by sandy sequences containing organic material (peat) at six positions. Mollusc samples were extracted from 50-50.3 m and at 88.7 m. A “Unterer Zwischenhorizont” lies between 98 and 109 m. The “Unterer sandig-schluffigen Ab-

folgen” (Lower sandy-silty sequences) continue on to the Pliocene boundary. A sample from 178.9m was selected from this zone. Another sample was collected in the Pliocene clays at 236.1 m in P35. Two additional samples from the Pliocene at 276.2 and 287.5-287.9 m were investigated from borehole P35a which was drilled directly adjacent to P35.

The most concentrated mollusc remains in borehole P35 are also found in the uppermost layer in the OZH at 21.9 m, and in the “Oberer sandige Folge” (Upper Sandy Suite) at 50 m. Both horizons contain gastropod fauna primarily belonging to interglacial species. The samples differ in the spectrum of species they contain. The sample at 21.9 m primarily contains terrestrial species pointing to the presence of a flood plain forest (*Perforatella bidentata*). The fauna from the sample at 50 m is aquatic consisting of stagnant plant-rich waters (*Gyraulus crista*, *Lymnaea stagnalis*, *Bathym-*



Fig. 5: *Borysthenia naticina* (MENKE, 1845) from the Viernheim research borehole, 164.0-164.2 m.

Abb. 5: *Borysthenia naticina* (MENKE, 1845) Forschungsbohrung Viernheim, 164,0-164,2 m.

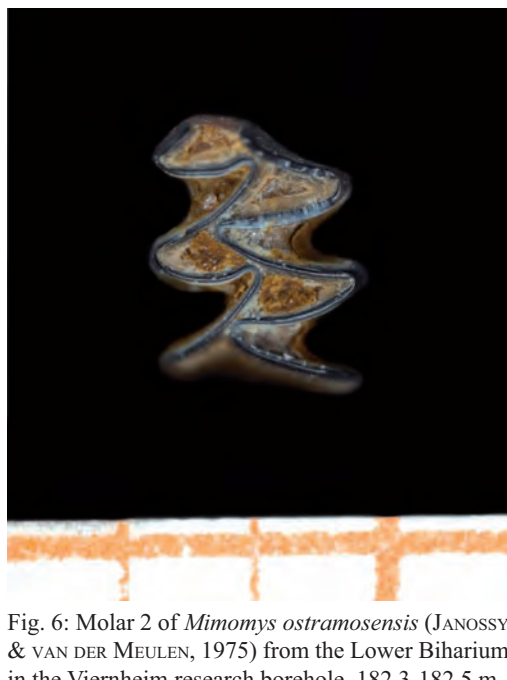


Fig. 6: Molar 2 of *Mimomys ostromosensis* (JANOSSY & VAN DER MEULEN, 1975) from the Lower Biharium in the Viernheim research borehole, 182.3-182.5 m.

Abb. 6: Molar 2 von *Mimomys ostromosensis* (JANOSSY & VAN DER MEULEN, 1975) aus dem unteren Biharium der Forschungsbohrung Viernheim, 182,3-182,5 m

phalus contortus and *Planorbarius corneus*) to slow flowing waters. Unfortunately, the material at 50 m has been compacted within a silty-sandy sediment which has become broken up as a result of elutriation. Some juvenile examples are still preserved. The composition of the thanatocoenosis at 50 m indicates the Mosbacher Sande 2 (GEISSERT 1970) by way of *Borysthenia naticina* (MENKE, 1845) (Fig. 5), because these sands also contain this species.

The terrestrial molluscs from the sample at 21.9 m are mostly hydrophilic (e.g. *Vertigo substriata*, *Vallonia enniensis*, *Carychium minimum* as well as *Carychium tridentatum*). The sample from the OZH at approx. 21 m in borehole P34 also contains a rich mollusc fauna, although dominated in this case by freshwater species. Terrestrial snails typical of warmer conditions, as still found today under leaf mould in shady flood plain forests, are the waxy glass snails, e.g. *Aegopinella nitens* (MICHAUD, 1831).

The two completely preserved valves of the pea clam *Pisidium amnicum* (O. F. MÜLLER, 1774) from 27.8 – 28.0 m are very interesting. This species occurs again at 178 m although it is primarily associated in the sample with species which tended to live in glacial, open landscapes. However, this mollusc is generally more frequent in interglacial deposits.

The molluscs in both boreholes indicate interglacial conditions. There is still some doubt, however, whether we are dealing here with the Cromerian or the Holsteinian interglacial because important type fossils are absent. The situation in the other boreholes described below is different because the molluscs in these boreholes can be confidently assigned to the Cromerian Complex.

2.3 Boreholes in Mannheim-Lindenhof (Baden-Württemberg) and Ludwigshafen – A36 Maudach (Rheinland-Pfalz)

The boreholes drilled in Mannheim-Lindenhof (RÄHLE 2005) and Ludwigshafen – A 36 Maudach (WEDEL unpublished), are particularly interesting. They were drilled in the vicinity of the two boreholes described above and were

sunk in advance of the research boreholes and investigated by LGB Rheinland-Pfalz. The boreholes contain notable fossil gastropod species from the Oberer Zwischenhorizont (Tab. 3). The first of these is *Clausilia rugosa antiquitatis* (NORDSIECK, 1990). This form has been identified in the younger Tegelen up to the older Middle Pleistocene. It is replaced in the Middle Pleistocene by *Clausilia rugosa parvula* (FÉRRUSAC, 1807). The second interesting gastropod is *Parafossarulus crassitesta* (BRÖMME, 1885) (Fig. 4).

Two particularly well preserved examples with numerous characteristic opercula of this species were found in the Maudach borehole between 27 and 28 m depth. The genus *Parafossarulus* is no longer present in Europe (GLOER 2002). In addition, the boreholes in Mannheim and Ludwigshafen-Maudach also contained remains of *Azeza goodalli* (A. FÉRUSSAC, 1821).

According to RÄHLE (2005), this “*crassitesta*-horizon” is not younger than Cromerian interglacial IV and not older than Cromerian interglacial III. This investigation looked at the Oberer Zwischenhorizont in eight different boreholes in Mannheim-Lindenhof, and specifically at the depth zone between 29 and 33 m. Analogous to Mannheim-Lindenhof, the Maudach borehole also contained tooth remains of early forms of the water vole *Arvicola terrestris* (LINNE, 1758). The genus *Arvicola* appears in Central Europe at the earliest in Cromerian interglacial III (OIS 15) according to KOENIGSWALD & VAN KOLFSCHOTEN (1996). The fauna encountered in Mannheim-Lindenhof is therefore of a very similar age to Mosbach 2 (Middle Mosbach) and Mauer, where *Arvicola* is also found (RÄHLE 2005). The results from other investigations of the OZH are also reported by ENGESSER & MÜNZING (1991) from boreholes that are also from the Mannheim area.

2.4 Viernheim research borehole (Hessen)

The conclusions drawn from the Viernheim borehole are that the three intermediate horizons encountered in the well reflect differentiated climatic conditions. Unlike the Ludwigs-

hafen boreholes, the Oberer Zwischenhorizont (Section Xa according to HOSELMANN 2008) is marked by climatically cooler species (Tab. 4). The second intermediate horizon from 89 m contains species preferring warmer conditions. This may possibly be the same horizon as in the Ludwigshafen-Mannheim area (“*crassitesta*” – horizon), although the type species is missing. The 5 m-thick third intermediate horizon between 164.73 and 169.9 m (Section Vb according to HOSELMANN 2008) and the underlying sandy-gravelly beds, are assigned a Lower Pleistocene age. The Lower Pleistocene (Tegelen) is confirmed at 132 m by the gastropod *Gastrocopta moravica oligodonta* (KROLOPP, 1979) (Fig. 2), and at 174 m by the previously unknown *Gastrocopta n. sp.* (Fig. 3), and at 182,5 by the mollusc *Corbicula cf. fluminalis* (O. F. MÜLLER, 1774) and the Arvicolidae *Mimomys ostramosensis* (JANOSSY & VAN DER MEULEN, 1975) (Fig. 8). The transition to the Pliocene contains plant remains but no molluscs, as in all of the samples looked at. The drill site was selected to penetrate the most undisturbed possible sequence of Pleistocene sediments (HOSELMANN 2008).

2.4.1 Upper Middle Pleistocene

The upper part of the borehole between 39.79 and 58.55 m depth, contains an almost 20 m thick bed of the Oberer Zwischenhorizont. Typical forest-type molluscs are not present. The molluscs are mainly climate-insensitive species of wet flood plain habitats of which *Vitreola crystallina* (O. F. MÜLLER, 1774) is the most common. This indicates that the climate was rather cool (LOŽEK 1964). The picture is dominated by species common to periglacial, open landscapes and sparse forest steppes. *Trochulus hispidus* (LINNAEUS, 1758) (“*Trichia hispida*” in older references) as well as amber snail species *Succinella oblonga* (DRAPARNAUD, 1801) and *Succinea putris* (LINNAEUS, 1758) are frequent. This thick intermediate horizon is obviously not the same as that in Ludwigshafen. These may be the pre-Eemian or Early Holsteinian interbeds described by SCHWEISS

(1988) from the Gernsheim and Groß-Rohrheim area. The chronostratigraphic interpretation is based on pollen analysis which indicates the pre-sence of shallow peat bogs and sparse birch and spruce growth.

The “*crassitesta*- intermediate horizon” must therefore lie much deeper because of the much thicker deposition in the eastern part of the Upper Rhine Graben. ENGESSER & MÜNZING (1991) also subdivide the OZH into three horizons with different climatic conditions. Investigations in the Karlsruhe and Mannheim areas led to a subdivision into a lower zone of interglacial Cromerian Complex sediments, followed by a middle and upper zone both marked by glacial environments, of which the upper is classified as Rissian.

2.4.2 Lower Middle Pleistocene

The species diversity in the Kieslager increases beneath the Oberer Zwischenhorizont. A mixed fauna is present at 70 m consisting of a *Fruticola fruticum* fauna, indicating a forest steppe environment, as well as a *Succinella oblonga* fauna with a high proportion of *Pupilla muscorum* (LINNAEUS, 1758), which indicates cooler steppe conditions. In addition, there is also the pea clam (*Pisidium casertanum ponderosum* STELFOX, 1918) which is adapted to cold and extreme environmental conditions.

The *fruticum* fauna which had its optimum at 89 m because it is joined by climatically more sensitive species such as *Pomatias elegans* (O. F. MÜLLER, 1774), *Ena montana* (DRAPARNAUD, 1801), *Discus perspectives* (MEGERLE VON MÜHLFELD, 1816), *Vertigo pygmaea* (DRAPARNAUD, 1801) and *Monachoides incarnatus* (O. F. MÜLLER, 1774). These species here indicate a much warmer period. This is the “*crassitesta* intermediate horizon” in the Maudach and Lindenhof boreholes. Seven metres deeper at 97 m, cooler steppe conditions dominate again and the molluscs are largely dominated by *Pupilla muscorum*, *Succinella oblonga elongata* SANDBERGER and *Trochulus hispidus* (cf. Chap. 2.3). There are also frequent *Clausilia* remains. Particularly interesting are five well preserved

apertures of *Neostyriaca corynodes*. This is the subspecies *ornatula* (ANDREAE 1884), which has been identified in the Lower Pleistocene to the Lower Middle Pleistocene in Central Europe (NORDSIECK 2007). It is characteristic of the “Upper Biharium” (Late Biharium, cf. KOENIGSWALD 2007) in Cromer. Associated species are *Clausilia cruciata* (STUDER, 1820) and *Clausilia dubia* (DRAPARNAUD, 1805). This species seems to be indifferent to the climate, analogous to its associated species (NORDSIECK 2006).

2.4.3 The Lower Pleistocene

The Kieslager is underlain between 131.23 and 133.18 m (Section VIIb after HOSELMANN 2008) by another thin intermediate horizon with calcareous loam sand. A small snail belonging to the genus *Gastrocopta*, now extinct in Central Europe, clearly indicates a Lower Pleistocene assemblage from 132.5 m. The specimen is a well preserved shell of *Gastrocopta moravica oligodonta* (KROLOPP 1979) (Fig. 2). Whilst the sediment one metre higher contains a mixed fauna indicative of a cooler “*Chondrula tridens*–steppe fauna” and a cold-moist flood plain forest fauna with a high proportion of *Perforatella bidentata* (Fig. 1) typical of open, glacial conditions, this intermediate horizon contains more thermophilic species representative of a flood plain forest. Some of the freshwater molluscs and a large number of amber snails indicate the existence of stagnant to slowly flowing waters with highly vegetated banks.

A strong increase in steppe elements, particularly *Granaria frumentum* (DRAPARNAUD, 1801), is found in an approximately 5 m thick intermediate horizon between 164.7 and 169.9 m (Section IVb after HOSELMANN 2008). This is the zone in which the bi-toothed snail *Perforatella bidentata* (GMELIN, 1791) which lives in flood plains reaches its greatest concentration in the Viernheim research boreholes. Two fragments may come from the larger, related species *Perforatella dibothrion* (M. v. KIMAKOWICZ, 1884). There are also remains of *Clausilia rugosa antiquitatis* (NORDSIECK, 1990) and the forest species *Azeca goodalli*

(A. FÉRUSAC, 1821). Kieslager with sandy horizons and silty clays form alternating interbeds below the intermediate horizon and are part of an alternating sequence of interglacial and glacial sediments found in the section down to 182 m. This is indicated by the different mollusc fauna which suggest a landscape consisting of sparsely wooded forests and open steppe (*fruticum* and *tridens* fauna).

More evidence for the Lower Pleistocene is found at 174 m in the form of a previously unknown species of the genus *Gastrocopta* (WOLLASTON, 1878). The special feature of the shell is the absent basal tooth in the aperture armature. This *Gastrocopta* may be linked to that described from the Lower Pleistocene by Kielniki (Poland). This is found in a fauna with *G. serotina* (LOŽEK, 1964) and is related by STWORZEWICZ (1981) to *Gastrocopta theeli* (WESTERLUND, 1877). However, the basal tooth found in *Gastrocopta theeli* is absent in the figure on Table 1 (Fig. 3). The first classification as *Gastrocopta turgida quadruplicata* SANDBERGER was not confirmed.

An approx. 2.60 m thick gravel-bearing fluvial sand horizon starts at 182.5 m and is followed by an approx. 2 m thick intermediate horizon (Section IVb after HOSELMANN 2008). The shell remains of molluscs in the gravel-bearing horizon are strongly corroded or polished which indicates strong transport of the sand and gravel. Half of the aquatic species found consist of small molluscs such as *Sphaerium corneum* (LINNAEUS, 1758), *Musculium lacustre* (O. F. MÜLLER, 1774) and *Corbicula fluminalis* (O. F. MÜLLER, 1774).

The sample contained two shell remains of *C. fluminalis* with the characteristic hinge. *C. fluminalis* is only known in Germany from the Holsteinian and the Tegelen. This species appears in Europe in the Late Pliocene and has its widest distribution during the Tegelen (MEIJER & PREECE 2000).

Another important stratigraphic discovery is the well preserved molar (M2) of a water vole (Arvicolidae), found in the sample from a depth of 182.4 m. Dr. Lutz Christian Maul (Senckenberg Institute, Weimar) identified the

specimen as a molar from *Mimomys ostramosensis* (JANOSSY & VAN DER MEULEN, 1975) (Fig. 9). Although not unusual, it is fairly rare to find identifiable remains of small rodent fossils in material collected from boreholes (WEDEL 1999). The distribution of Arvicolidae species is now very well known in Europe and tied to chronostratigraphic tables. Analogous to *Corbicula fluminalis*, this water vole lived in the “Lower Biharium” (*Mimomys pusillus* – *Mimomys savini*- zone, KOENIGSWALD 2007), and the “Villanyium” approx. 1.6 to 2.0 million years ago. This species has previously only been found at a few locations in Europe (MAUL 2002). This is only the second identification of this species in the Upper Rhine area in addition to Neuleinigen near Grünstadt (MALEC & TOBIEN 1976). HEIDTKE (1979) dated the Neuleinigen fauna, also on the basis of large mammal fossils, as Lower Pleistocene with a dry-warm to subtropical climate. The fossils were found in the material filling a fissure (Neuleinigen 11) in a limestone quarry. HEIDTKE climatically compared Neuleinigen with the fauna from Hohensülzen (STORCH et al. 1973) reporting that the older Hohensülzen fauna represented the moist-warm river valleys, and Neuleinigen the topographically higher dry steppe.

The smaller two-metre-thick intermediate horizon in the Viernheim research borehole is underlain by an approx. 3.80 m thick, carbonate-rich, gravely sand bed which gradually grades into the sandy-silty series of the Lowest Quaternary. This sequence of interbedded rocks extends down to 224 m where they are underlain by limnic-fluviatile Pliocene sediments (HOSELMANN 2008). This sandy-silty suite again documents fluctuating climatic conditions where the climate optimum at 196.5 m is indicated by fewer forest-living molluscs (*Fruticicola fruticum*, *Vitrinobrachium breve*, *Discus rotundatus*). Forest species are absent at 194 m. Open landscape species such as *Pupilla muscorum* are dominant. These molluscs may indicate the earliest glacial period of the Pre-Tegelen in the Lower Quaternary. There is now a significant general decline downwards in the number of species and individuals. The last

mollusc remains are found at 223 m comprising only three freshwater species and one terrestrial species. In contrast, the samples now contain large quantities of plant remains.

3 Comments on biostratigraphically important species

Perforatella bidentata (GMELIN, 1791) and *Perforatella dibothrion* (M. v. KIMAKOWICZ, 1884)

P. bidentata (fig. 1) is often found in the Pleistocene sands and fine-clastic intermediate horizons of the Upper Rhine Graben. It is particularly common in the Lower Pleistocene deposits in the Viernheim research borehole. Occurrence are known into the Late Eemian (MÜNZING 1999). The species can be easily identified on the basis of the two teeth (basal and palatal) in the lower aperture area. This is a pure flood plain snail (alder marsh) which requires a wet habitat. It is found in both interglacial as well as glacial contexts. It still lives in eastern Central Europe (Thuringia, Bavaria, Saxony, Austria, Czech Republic, Slovakia, Poland and Hungary) and at scattered localities in south-eastern Scandinavia. The species was found far to the west in the Pleistocene. This is confirmed by specimens collected near Paris by GEISSERT (pers. comm.)

The related species *Perforatella dibothrion* is a type fossil for the interglacial habitats of the Rhine valley in the Lower Pleistocene and Lower Middle Pleistocene. GEISSERT (1970) found a complete example in the Mosbacher Sande 2 and reports several discoveries in gravel pits in the Upper Rhine (GEISSERT 1969). Two fragments were found in the sample from 167.35 – 167.45 m in the Viernheim borehole, which may belong to this species. RÄHLE (2005) also mentions *Perforatella* remains which he assigns to this species, from the Oberer Zwischenhorizont in borehole P18 Mannheim-Lindenhof. The main differences to *P. bidentata* are a much higher aperture and two stronger teeth, although the space between the two teeth is narrower than in *P. bidentata*. The shell has clear ribs, and the first whorls have a surface structure with small

scales. This species is only found alive today in eastern Slovakia and in the Carpathians where it lives in the sub-mountainous zone in moderately wet deciduous forests (KERNEY et al. 1979). Its distribution in the Late Pleistocene in Europe was continental-Atlantic, i.e. much broader than today.

***Gastrocopta (Vertigopsis) moravica oligodonta* (KROLOPP 1979) (Fig. 2)**

This is the first confirmation of this species in the Upper Rhine Graben, and comes from the Viernheim research borehole. The shell is less conical than the nominate form *G. moravica* (PETRBOK, 1959). The intraparietal tooth of the aperture is reduced to a thickening at one point. KROLOPP (1979) found it together with *Gastrocopta serotina* (LOŽEK, 1964) in Hungary near Szabádhidvég (comm. Feyér). Both these species had previously only been identified in the Lower Pleistocene (Upper Villanyum – Lower Biharium). These two species are therefore very chronostratigraphically significant as type fossils for the Late Tegelen (KROLOPP 1986). The *Gastrocopta* genus is extinct in Europe. The youngest representative of the genus in the Pleistocene is the species *Gastrocopta theeli* (WESTERLUND, 1877).

RÄHLE (1995) found *G. moravica oligodonta* in argillaceous high flood deposits in Uhlenberg (Iller-Lech Plate, Bavarian Swabia). The Uhlenberg fauna contains molluscs which were also encountered in Viernheim in samples from 131 m and 182 m, and are characteristic species for the Lower Pleistocene. In addition to *G. m. oligodonta*, another interesting find was a fragment possibly belonging to *Cochlostoma salomoni* (GEYER, 1914). GEISSERT (1983) mentions this species from Rhine sediments at Gamsheim and La Wanzenu, which are classified as Tegelen. Further interesting species are *Clausilia rugosa antiquitatis*, *Azeca godalli* and *Ena montana*. All of these species are assigned to the interglacial fauna elements, whereby the two *Gastrocopta* species were already part of Pliocene faunas and are unknown beyond the Lower Pleistocene.

Chondrula tridens (O. F. MÜLLER, 1774) and *Granaria frumentum* (DRAPARNAUD, 1801) are possible indicators of steppe landscapes at the edge of the flood plain forests. Both species have recently become much rarer in Europe because their habitats are virtually no longer existent.

***Gastrocopta (Vertigopsis) sp.* (Fig. 3)**

The small conical shell is 1.9 mm high and 1.0 mm wide. The five arched whorls increase uniformly in size up to the penultimate whorl – the last whorl becomes smaller towards the aperture. The apertural border with the lip is well formed and slightly folded over towards the spindle. The shell is shiny, slightly stripy and has a weakly developed palatine torus. It has three strong teeth, one columellar, one palatal and one parietal, although the adjacent palatine wall of the specimen is damaged and partially missing. The teeth are almost the same distance apart and their tips almost point towards one another. A characteristic feature is the complete absence of a basal tooth. This form has not previously been identified in the Pleistocene.

4 Conclusions

Fossil molluscs were investigated from three research boreholes drilled in the Heidelberg Basin. Special attention was given to the Oberer Zwischenhorizont (OZH) because, particularly at the western margin of the Upper Rhine Graben, it stands for the Middle Pleistocene interglacials which are assigned to the Cromerian Complex. Whilst the OZH is relatively thick at Ludwigshafen and already encountered at a depth of around 21 m, the similar or slightly thinner horizon is first encountered in Viernheim at a depth of around 39 m. A lower horizon in the Viernheim borehole lying at around 50 m depth contains a mollusc fauna probably derived from the earlier Holsteinian period. A much thicker intermediate horizon is found as the deepest horizon between 195 and 223 m at the boundary to the Tertiary. It is divided up by several small

Kieslager (gravel horizons). Evidence of Lower Pleistocene mollusc assemblages have already been found above this horizon from 132 m. The findings support the assumption proposed by ENGESSER & MÜNZING (1991), that the Oberer Zwischenhorizont covers two to three climatically different epochs, whereby the deepest horizon in the Rhine Graben can be assigned to the Lower Pleistocene on the basis of its fossils, whilst the upper horizons have Pre-Eemian or Holsteinian character.

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Table 1: Research borehole Ludwigshafen Parkinsel P34.

Tab. 1: Forschungsbohrung Ludwigshafen Parkinsel P34.

Classification Parkinsel P34	qp(a)				tpl			
	OZH	OZH	UZH	UZH	Reuver?			
Art:	20.50-21.00	36.8-37.5	147.8-148.0	160.3-160.4	176.0-176.5	207.8-208	232.2-232.4	290.7-290.8
Aegopinella sp.	1							
Clausiliidae indet.	1							
Trochulus sp.	1							
Vertigo sp.	3							
Vertigo substriata	3							
Oxyloma elegans	1							
Succinea putris	1							
Pisidium amnicum	1							
Musculium lacustre	2							
Valvata cristata	125							
Planorbis planorbis	5							
Lymnaeidae indet.	200							
Stagnicola aff. palustris	2							
Valvata macrostoma	1							
Acroloxus lacustris	2							
Gyraulus crista	30							
Hippeutis complanatus	3							
Planorbarius corneus	6							
Sphaeriidae indet.	90	2						
Galba truncatula	6							
Bithynia tentaculata (Opercula)	85					1		
Bithynia tentaculata	4							
Valvata piscinalis	6							
Viviparus sp. (Opercula)	1							
Pisidium pulchellum	10							
undetermined fragments of molluscs	2			1				1
Number:	592							
Piece of plants	x	x	x	x	x	x	x	
Charophyts	x							
Small mammals	x							

OZH = Oberer Zwischenhorizont; UZH = Unterer Zwischenhorizont; qp(a) = Older Pleistocene; tpl = Pliocene

Table 2: Research borehole of Ludwigshafen Parkinsel P35/P35a (first part).

Tab. 2: Forschungsbohrung Ludwigshafen Parkinsel P35/35a (erster Teil).

Classification: Parkinsel P35 / P35a	qp						OSF		USF	tpl	
	26.33- 26.37	27.2- 27.24	27.8- 28.0	30.26- 30.3	50.0- 50.3	88.70	178.00	236.10		276.20 (P35a)	287.5- n. F.
<i>Clausiliidae</i> indet.	21.90							1	n. F.	n. F.	
<i>Vitrobrachium</i> breve	1										
<i>Aegopinella</i> sp.			1								
<i>Aegopinella nitens</i>			1								
<i>Monachoides incarnatus</i>			1								
<i>Arianta arbustorum</i>	4			1				2			
<i>Vitrea crystallina</i>	1										
<i>Perforatella bidentata</i>	8		1								
<i>Vallonia excentrica</i>	3										
<i>Vallonia pulchella</i>	1										
<i>Vertigo pygmaea</i>	2			2							
<i>Pupilla muscorum</i>	2							1			
<i>Vertigo</i> sp.	2			3							
<i>Trochulus</i> sp.	2	1		4				1			
<i>Cochlicopa lubrica</i> Komplex	3										
<i>Punctum pygmaeum</i>	4										
<i>Deroceras laeve</i>	1										
<i>Vertigo angustior</i>	3										
<i>Succinella oblonga</i>				4				1			
<i>Vertigo substriata</i>	1										
<i>Carychium tridentatum</i>	4										
<i>Carychium minimum</i>	2										
<i>Oxyloma elegans</i>	3										
<i>Succinea</i> sp.	2										
<i>Vallonia emnensis</i>	1										
<i>Succinea putris</i>								1			
<i>Unionidae</i> indet.								4			
<i>Pisidium amnicum</i>			2					5			
<i>Borysthenia naticina</i>				2							

Table 2: Research borehole of Ludwigshafen Parkinsel P35/P35a (second part).

Tab. 2: Forschungsbohrung Ludwigshafen Parkinsel P35/35a (zweiter Teil).

Classification:	qp						OSF		USF	tpl		
	21.90	26.33-26.37	27.2-27.24	27.8-28.0	30.26-30.3	50.0-50.3	88.70	178.00		236.10	276.20 (P35a)	287.5-287.9
Species:												
<i>Musculium lacustre</i>												
<i>Segmentina nitida</i>	1											
<i>Valvata cristata</i>	3											
<i>Planorbis planorbis</i>	7							1				
Lymnaeidae indet.	4							4				
<i>Anisus spirobis</i>	4							5				
<i>Gyraulus albus</i>			1						1			
<i>Gyraulus crista</i>	3							17				
<i>Lymnaea stagnalis</i>								1				
<i>Pisidium</i> sp.					1							
<i>Planorbarius corneus</i>	2							3				
Sphaeriidae indet.	2							4				
<i>Galba truncatula</i>	4							5				
<i>Bithynia tentaculata</i> (Opercula)	4				12			16				
<i>Bithynia tentaculata</i>								2				
<i>Valvata piscinalis</i>								1				
<i>Bathynphalus contortus</i>								2				
Number:	90	1	1	6	13	102	0	23	0	0	0	0
Piece of plants	x	x	x	x	x	x	x	x	x	x	x	x
Ostracods												
Piece of fishs	x											
Small mammals												
Piece of insects		x	x									

n. F. = no Fauna; OZH = Oberer Zwischenhorizont; OSF= Obere sandige Folge; USF= Untere sandig-schluffige Folge
 pl = Pleistocene; tpl = Pliocene (only borehole P35a)

Table 3: Borehole Ludwigshafen-Maudach A-36.

Tab. 3: Bohrung Ludwigshafen-Maudach A-36.

Classification:	Middle-Pleistocene			
Lu-Maudach A 36	OZH			
Art:	23.10 - 23.40	26.40- 26.80	27.50- 28.00	number:
Clausiliidae indet.	7	1	7	15
Clausilia bidentata	2	1	2	5
Discus ratus rude			1	1
Vitrinobrachium breve			5	5
Aegopinella la nitidu			1	1
Ena na monta			3	3
Helicodonta obvolvata		2	2	4
Macrogastra lineolata		2	1	3
Monachoides incarnatus		1	1	2
Azeca lli gooda			2	2
Arianta arbustorum	2	1		3
Vitrea crystallina	3	3	4	10
Discus rotundatus	1	2		3
Limax inereoniger c		1		1
Cepaea hortensis	2	1	1	4
Clausilia pumila	3	5	6	14
Perforatella bidentata	7	8	29	44
Chondrula ns tride			3	3
Pupilla terri s			1	1
Pupilla nsegyrata de		2		2
Vallonia excentrica		3	1	4
Vallonia pulchella	6	2	9	17
Vallonia declivis			1	1
Vallonia costata	5		5	10
Pupilla muscorum		3	17	20
Columella olumella c		1		1
Vallonia tenuilabris			1	1
Cochlicopa ella lubric			1	1
Trochulus sp.	2	1	1	4
Vertigo sp.	8	3		11
Clausilia rugosa antiquitatis			2	2
Cochlicopa lubrica Komplex	2	3	4	9
Euconullus fulvus	1		1	2
Nesovitrea hammonis		2	11	13
Trochulus hispidus	1		3	4
Clausilia dubia		1	3	4
Helicigona lapicida	2	1	1	4
Columella dentula e			3	3
Deroceras leave	2		1	3

<i>Vertigo substriata</i>	3			3
<i>Succinella oblonga</i>	21	26	290	337
<i>Succinella elongata</i>		1	3	4
<i>Carychium um minim</i>	1			1
<i>Oxyloma legans e</i>			3	3
<i>Vallonia enniensis</i>	1	1	3	5
<i>Vertigo genesii</i>	1	6	44	51
<i>Vertigo geyeri</i>			1	1
<i>Pseudotrichia ginosa rubi</i>	1			1
<i>Zonitoides nitidus</i>			2	2
<i>Ancylus fluviatilis</i>	3	2	3	8
<i>Borysthenia naticina</i>		3	1	4
<i>Valvata cristata</i>			8	8
<i>Valvata macrostoma</i>		1	10	11
<i>Valvata piscinalis</i>	9	2	21	32
<i>Bithynia sp.</i>	26	100		126
<i>Parafossarulus crassitesta</i>	6	5	20	31
<i>Bithynia tentaculata</i>	1	1	4	6
<i>Bithynia leachii</i>		3	2	5
<i>Planorbis planorbis</i>	5	4	9	18
<i>Planorbarius corneus</i>	2	1	2	5
<i>Planorbis arinatus c</i>	1			1
<i>Anisus leucostoma</i>	1	3	9	13
<i>Bathyomphalus contortus</i>	1		2	3
<i>Hippeutis complanatus</i>			3	3
<i>Segmentina nitida</i>			2	2
<i>Gyraulus albus</i>	1		4	5
<i>Gyraulus laevis</i>		1	1	2
<i>Lymnaeidae indet.</i>	2		2	4
<i>Stagnicola agg. palustris</i>			2	2
<i>Radix balthica</i>		1	12	13
<i>Radix balthica aff. ampla</i>			5	5
<i>Lymnaea stagnalis</i>			4	4
<i>Galba truncatula</i>	6	8	5	19
<i>Sphaeriidae t. inde</i>		1		1
<i>Sphaerium orneum c</i>	2			2
<i>Pisidium sp.</i>		1		1
<i>Pisidium amnicum</i>	4	4	30	38
<i>Pisidium upinum s</i>			4	4
<i>Pisidium obtusale lapponicum</i>			9	9
<i>Pisidium ssieranummoite</i>		2		2
gesamt:	154	227	654	1035
Charophyts-Oogonie	x	x	x	
Piece of fishes	x	x	x	
Small mammals	x	x	x	

OZH = Oberer Zwischenhorizont

Evidence for a Waalian thermomer pollen record from the research borehole Heidelberg UniNord, Upper Rhine Graben, Baden-Württemberg

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Abstract: A pollen record from the borehole Heidelberg UniNord, in the northern Upper Rhine Graben shows evidence for one of the rare Waalian thermomers. The record may not comprise the complete thermomer it certainly reflects a sort of succession, initiated by sequences with high values of pine and spruce, followed by a dominance of pine, hemlock-spruce and spruce and finished by a dominance of hemlock-spruce, oak and hornbeam becomes visible. Pollen percentage values for *Tsuga* reach 20 % indicating that this taxon is an important forest element. There is a continuous presence of *Carya*, *Pterocarya*, *Eucommia*, *Celtis* and *Ostrya*-type. Neighbouring pollen records reveal similar patterns. The pollen diagram is closely related with the Waalian profiles Leerdam and Eindhoven from the Netherlands.

[Nachweis der Waal-Warmzeit anhand pollenanalytischer Untersuchungen an der Forschungsbohrung Heidelberg UniNord, Oberrheingraben, Baden-Württemberg]

Kurzfassung: Eine Pollensukzession aus der Bohrung Heidelberg UniNord im Oberrheingraben kann mit der bisher selten nachgewiesenen Waal-Warmzeit verknüpft werden. Das wohl nicht vollständig erfasste Thermomer reflektiert eine angedeutete Sukzession, die von einer basalen Fichten-Kiefernzeit über einen Abschnitt mit Kiefer, Hemlocktanne und Fichte, hin zu einer abschließenden Waldzeit verläuft, in der Hemlocktanne (*Tsuga*), Eiche und Hainbuche dominieren. *Tsuga* erreicht im Gesamtdiagramm Werte bis zu 20 % und repräsentiert ein wichtiges Waldelement. Ständig vorhanden sind in geringen Anteilen ferner *Carya*, *Pterocarya*, *Celtis*, *Eucommia* und *Ostrya*-Typ. In Nachbarbohrungen deutet sich dasselbe Muster an. Das Pollendiagramm wird korreliert mit den Waal-Profilen aus Leerdam und Eindhoven (Niederlande).

Keywords: Upper Rhine Graben, pollen analysis, Waalian thermomer, biostratigraphy

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1 Introduction

In order to develop a better understanding of the Pleistocene geological processes associated with the evolution of the Upper Rhine Graben (URG), a scientific core drilling has been brought down at the campus of Heidelberg University. In this location, one of the thickest successions of continental Quaternary deposits has been expected according to various pre-site surveys (BUNESS, GABRIEL & ELLWANGER 2008). For technical reasons, a first borehole (Heidelberg UniNord1) has been cored down to 190 m in 2006, followed by a second borehole (UniNord2) down to 500 m in 2008. For a first summary of geological results, cf. ELLWANGER et al. (2008); for an outline of the Heidelberg project cf. GABRIEL et al. (2008).

In this paper, only results from the first borehole (0 – 190 m; UniNord1) have been evaluated. According to ELLWANGER et al. (2008), only about one third of the succession consists of fine-grained i.e. silt-dominated deposits. Out of these, only three intervals, altogether amounting to less than five meters of sediment core, turned out to be suitable for a detailed pollen analysis (57 – 58 m; 81 – 87 m and 180 – 181.70 m). The remaining 65 m of silt-dominated sediments were poor in pollen-content. This may be due to oxidation and differential preservation of the pollen, e.g. concentration of thick-walled grains such as conifers and *Tilia*, but also of poor core quality.

The biostratigraphic review maybe summarized as follows:

- (1) At 57 – 58 m and 82 – 85 m depth a pine-spruce forest indicating cool climate;
- (2) At 81 – 82 m and at 87 m depth a middle Pleistocene pollen spectrum without *Fagus* indicates an interglacial environment is indicated;
- (3) At 180 – 181.70 m depth some very pollen rich samples were found in peaty sediments with more or less silt. The higher pollen percentage values of the genus *Tsuga* (Hemlock spruce) indicates a temperate “interglacial” resembling an early Pleistocene age.

This study is focused on the interval (3).

2 Methods

Peaty samples were taken at 10 cm intervals. In total, 17 pollen samples were analysed. Samples were subjected to HF (70 %) treatment followed by ultrasonic sieving (mesh size 6 x 8 µm) then acetolysis. With the exception of the two oldest samples, arboreal pollen counts of at least 500 were attained. The basis for representation on the pollen diagram was the total pollen sum (all pollen types excluding aquatics and ferns). The pollen diagram (Fig. 2) was prepared using the plotting program *Tilia* (GRIMM 1991). Non-arboreal taxa that were only encountered in few samples and in low numbers are not shown on the diagram. Values below 1 % are also represented (symbol “•”).

3 Results

The pollen diagram (Fig. 1) is divided into three local pollen zones (A, B and C):

- A: 181.70 – 181.15 m: *Pinus* – *Picea* zone (with ferns)
- B: 181.15 – 180.55 m: *Pinus* – *Tsuga* – *Picea* zone
- C: 180.55 – 180.00 m: *Tsuga* – *Quercus* – *Carpinus* zone (with *Alnus* and *Betula*)

The local pollen assemblage zone A is characterized by a dominance of *Pinus* and *Picea*, ferns are of great importance. The arboreal taxa such as *Tsuga*, *Ulmus*, *Corylus*, *Carpinus* and *Eucommia* are present with rates up to 2 %, *Quercus* ranges between 2-5 %.

In zone B *Tsuga* (and *Alnus*) is increasing and represents an important forest element.

Zone C reflects a dominance of *Tsuga* (10-15 %), *Quercus* (10-15 %) and *Carpinus* (10-25 %) (together with *Alnus* and *Betula*). *Pinus* and *Picea* are of less importance. Typical elements of Older- and Middle Pleistocene such as *Pterocarya*, *Eucommia*, *Carya*, *Celtis* and *Ostrya*-type are continuously present in low rates. *Fagus* is limited to sample 180,10 m where two pollen grains are recorded.

Altogether, the succession is clearly dominated by arboreal taxa representing a thermomere stage i.e. warm climate. Non arboreal pollen

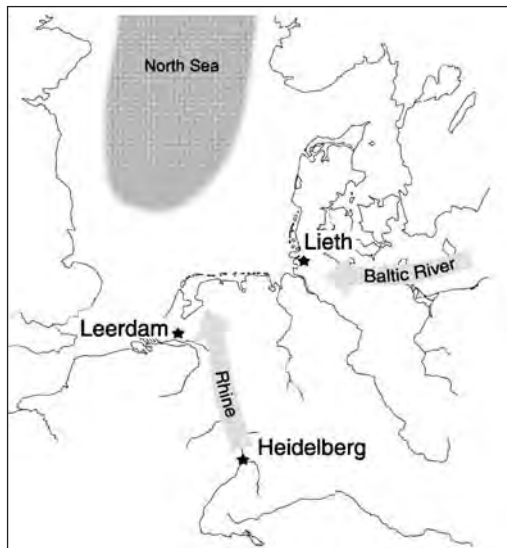


Fig. 2: Geographic position of Waalian sequences in middle Europe, Leerdam, Lieth, and Heidelberg. Present topography and palaeogeographical setting (GIBBARD 1988). Palaeo-shoreline uncertain, varying according to high and low sea level.

Abb. 2: Geographische Lage der Waal-Vorkommen in Mitteleuropa, Leerdam, Lieth und Heidelberg. Heutige Geographie und Waal-zeitliche Paläogeographie (GIBBARD 1988). Die Paläo-Küstenlinie ist unsicher, sie schwankt je nach Höhe des Meeresspiegels.

types are of less importance and mostly below 10 %, with the exception of two samples with *Filipendula* (up to 30 %) and an unknown, very small *Liliaceae*-type. Both elements represent the influence of local swampy vegetation.

4 Discussion

According to the implied succession of the significant arboreal taxa, the pollen diagram (Fig. 1) has been divided in three pollen assemblage zones. There are, however, no taxa, which are immigrating in the course of the sequence. All are present right from the beginning. Complete sequences of the immigration of tree species during an interglacial cycle (e.g. shrubs – birch - pine - broad leaf trees – climax zone – conifers) is well known from the Holocene, from the Eemian, and from middle Pleistocene

thermomers (Holsteinian, Cromerian). These characteristics are also reported from Bavelian sequences (ZAGWIJN & DE JONG 1984). However, as indicated above, this type of succession is not recorded in the Waalian thermomer, the older Pleistocene (Tiglian thermomers) and the Reuverian.

The decrease of arboreal taxa during the Pleistocene is known from various studies. *Pterocarya* is ultimately recorded at the end of the Holsteinian (e.g. BEAULIEU & MONJUVENT 1985).

Eucommia and *Celtis* barely occupy interglacial I and II of the Cromerian-Complex, e.g. HOMANN & LEPPER (1994), HAHNE (1996a,b). VAN DER HAMMEN et al. (1971) record *Carya* and *Ostrya*-type restricted to the Older Pleistocene thermomers Waalian and Tiglian. ZAGWIJN & DE JONG (1984) report from the cores from Bavel, Netherlands, i.e. the type region of the Bavelian stage, also *Carya*, *Ostrya*-type and *Tsuga*.

Tsuga was the first taxon recognised as an element of the Older Pleistocene (e.g. by VAN DER HAMMEN et al., 1971). According to ZAGWIJN (1957, 1960) and HAHNE (1996a,b), this taxon is present in low levels in the Reuverian and in the Pretiglian as well as in the Tiglian A, B and C. KNIPPING (2004) recorded single pollen grains in sediments of Bavelian age, whereas ZAGWIJN & DE JONG (1984) report values up to 30 % in a profile in Bavel revealing also a distinct succession of the tree species. Quite recently, KNIPPING (2008) even stated a single pollen grain of *Tsuga* in a thermomer, which she assumed to belong to the Cromerian Complex.

On the other hand, Pliocene elements such as *Liquidambar*, *Nyssa*, *Sciadopitys*, *Taxodium*- and *Sequoia*-habitus are not present in the pollen diagram (Fig. 1). All those are frequent in the Reuverian and, with the exception of *Sciadopitys*, very sparsely during Tiglian (ZAGWIJN 1960, 1963, 1989). Some very first results of the lower parts of the Heidelberg UniNord borehole confirm this trend (ELLWANGER et al. 2008, Fig. 3).

5 Conclusions

The pollen diagram (Fig. 1) reflects a thermomer revealing a closed forest vegetation in which pine, spruce, Hemlock-spruce, oak, hornbeam and elder were dominant. Records of Older Pleistocene elements (*Pterocarya*, *Eucommia*, *Celtis*, *Carya*, and *Ostrya*-type) are continuously present but only in low numbers. A successive immigration is not visible.

All these characteristics exclude an assignment of this thermomer to young or middle Pleistocene stages. A correlation with the Reuverian is also not possible due to the missing of Pliocene elements. For the Tiglian the presence of *Sciadopitys*, *Nyssa*, *Liquidambar*, and *Pinus haploxylon*-Habitus is compulsive. Therefore, only the Waalian and the Bavelian stages are to be taken on closer consideration.

After ZAGWIJN & DE JONG (1984), the Bavelian fills an intermediate position between Older- and Middle Pleistocene. With respect to partly high values of *Tsuga*, the presence of *Ostrya*-type, *Celtis* and *Eucommia* an Older Pleistocene character is evident whereas a succession of the expansion of various trees due to differences in re-immigration emphasizes a strong resemblance with thermomers younger than Tiglian and Waalian.

The pollen diagram (Fig. 1) shows significant similarities with the Leerdam-Profile (Fig. 2) which is the type locality for Waalian (ZAGWIJN & DE JONG 1984). The similarity is not restricted only to the spectra of the arboreal pollen types, but also in their values (with the exception of *Picea*, which in Leerdam has a smaller representation). Different with Leerdam is also the missing of indications of systematic suc-

	Pliocene		Older Pleistocene			Middle Pleistocene		Younger Pleist.	
	Reuverian	Tiglian	Waalian	Bavelian	Cromerian	Holsteinian	Eemian	Holocene	
<i>Sequoia</i> -Hab.	○	x							
<i>Taxodium</i> -Hab.	○	x							
<i>Sciadopitys</i>	○	○							
<i>Liquidambar</i>	○	x							
<i>Nyssa</i>	○	x							
<i>Carya</i>	○	x	x	x					
<i>Ostrya</i> -type	○	x	x	x					
<i>Tsuga</i>	○	○	○	○	x				
<i>Eucommia</i>	○	○	○	○	○				
<i>Celtis</i>	○	○	○	x	○				
<i>Pterocarya</i>	○	○	○	○	○	○			
<i>Fagus</i>	○	○	x	x	○	○	x	○	

Fig. 3: Presence of important arboreal taxa in Middle European pollen sequences from the Reuverian (Pliocene) to the Holocene. Adapted from LANG (1994), updated according to actual investigations and with regard to new results from the Heidelberg research boreholes. – Symbols: ○ = frequent, x = rare.

Abb. 3: Vorkommen wichtiger Baumpollen in mitteleuropäischen Pollensequenzen vom Reuver (Pliozän) bis ins Holozän. Nach LANG (1994), fortgeschrieben aufgrund neuer Untersuchungen, insbesondere neuen Ergebnissen aus den Forschungsbohrungen Heidelberg. – Symbole: ○ = häufig, x = selten.

cession in immigration and culmination of the individual trees.

In Kronau, about 20 km south of Heidelberg, another borehole revealed cores of peaty sediments at depths around 85 m. Here several pollen samples reveal nearly identical Waalian pollen assemblages as in Heidelberg.

ZAGWIJN (1989) noted that the Waalian generally has the character of an interglacial complex consisting of an interstadial situated between two thermomers. A similar sequence is recorded by MENKE (1976) for the "Tornesch thermomer" (Lieth series) situated in Schleswig-Holstein which is also assigned to the Waalian. Although it reveals a domination of Ericales (pointing toward Atlantic heaths) apart from *Tsuga* all Older Pleistocene elements are present. Running analysis on the cores of the UniNord2 borehole at Heidelberg reveal 26 m underneath the peaty Waalian sediments again typical Waalian pollen sequences which possibly could be assigned to the older Waalian thermomer. The intermediate sediments are fluvial gravels, which, unfortunately, are poor of pollen so that their climatic character is still uncertain. Even a separation within either the upper or the lower thermomer by fluvial gravels, without any difference in climate, may be considered.

The question remains why there are such great similarities in the forest vegetation between the Upper Rhine Graben and the Netherlands, whereas from Lieth (Schleswig-Holstein, Fig. 2) a quite different forest vegetation and maritime influenced different climatic conditions has been reported by MENKE (1969, 1976). In our view, this indicates uniform climate conditions along the Rhine, and different conditions further east. Today the Rhenish Schist highlands act as a barrier between the Netherlands and the Upper Rhine Graben. In the Waalian stage, prior to their major uplift, the Rhenish Schist was an area of some low hills. In this scenario, there was an almost flat landscape from Heidelberg to the Netherlands, with uniform forest vegetation and climate conditions (Fig. 2).

However, K.-E BEHRE (Wilhelmshaven) has expressed another view (pers. comm.). The Neth-

erlands situated in the lower Rhine area, were an excellent sediment and pollen catching area, including reworked pollen from further south. Their pollen records reflect the complete upper Rhine vegetation combined with the regional elements. On the other hand, the Lieth series (MENKE 1976) reflects the regional vegetation of Holstein, maybe including input from the Baltic river system (Fig. 2).

The chronostratigraphic age of the Waalian amounts to 1.1 -1.3 Ma (ZAGWIJN 1989) or to 1.4 - 1.6 Ma (OGG, OGG & GRADSTEIN 2008), both with duration of approximately 200 ka. Thus, the peaty sequence from Heidelberg (Fig. 1) with a length of 1.7 m does certainly not reflect one of the two thermomers as a whole, but only a shorter period belonging to either of them.

Considering the arboreal pollen types, the climate of the recorded sequence was probably not warmer than today. The present day distribution of the most important *Tsuga* species (*T. canadensis* and *T. diversifolia*) in North-East America and East Asia reflect mountainous climates and moist habitats.

According to DE JONG (1988), many of the so-called Pliocene trees that are today restricted to North America or East Asia, actually do very well in European parks or as forest elements. Thus, the interglacial climates of the Early Pleistocene might not have been more favourable than in the middle or young Pleistocene, but the intermediate stadials were not as extreme as the glacial stages of that period.

Thus, for the identification of a Waalian thermomer in Middle Europe three conditions must be fulfilled:

1. no complete succession of the arboreal genera,
2. *Tsuga* must be an important member of the forest vegetation, and
3. there must be at least single records of so called Older Pleistocene elements such as *Eucommia* and *Pterocarya* and in very low rates also *Carya*, *Celtis* and *Ostrya*-type and possibly even *Fagus*.

Final remark: In summer 2008, a second drilling in Heidelberg ("UniNord2") finally reached

its planned depth of 500 m. First scans of pollen content down to 499.94 m include continuous findings of *Fagus* and *Carya* as well as such of *Sciadopitys*, *Nyssa*, *Pinus haploxyylon*, Cupressaceae, *Sequoia*- and *Taxodium*-habitus. The spectra are clearly different from the Waalian and represent most likely the Tiglian interval. By now, we also know that the thickness of the Quaternary at the UniNord location will amount to more than 500 m (well exceeding the originally assumed thickness of ~ 400 m). The Reuverian is expected well below 500 m.

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Timing of Medieval Fluvial Aggradation at Bremgarten in the Southern Upper Rhine Graben – a Test for Luminescence Dating

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Abstract: The Holocene flood plain of the River Rhine is a complex dynamic sedimentary system. A series of geochronological results for the Bremgarten section including optically stimulated luminescence (OSL) and radiocarbon dating was determined to improve the understanding of part of the Holocene evolution of the River Rhine. The applied single aliquot regenerative (SAR) protocols and the applied experimental studies to find the best luminescence behaviour leave us with confidence that OSL dating is a suitable method for dating fluvial sediments from large river systems. Insufficient bleaching of the sediments from Bremgarten prior to deposition seems to be not as dramatic as previously thought. OSL and radiocarbon dating results give evidence for a short period of major erosion and re-sedimentation of fluvial sediments from the “Tiefgestade” at the Bremgarten section between 500 and 600 years before present. This time period correlates with the beginning of the Little Ice Age at about AD 1450. Several severe floods occurred in Southern Germany between AD 1500 and 1750; all those floods correlate to the period of the Little Ice Age, including the destruction of the village of Neuenburg AD 1525.

[Die zeitliche Stellung einer mittelalterlichen fluvialen Sedimentakkumulation im südlichen Oberrheingraben am Beispiel Bremgarten – ein Test für Lumineszenz-Datierungen]

Kurzfassung: Die holozäne Überflutungsebene des Rheins ist ein komplexes sedimentäres System. Die geochronologischen Untersuchungen mittels Optisch Stimulierter Lumineszenz (OSL) und Radiokohlenstoff-Datierungsmethode verbessern das Verständnis der holozänen Entwicklung des Rheinsystems und ermöglichen quantitative Studien zu Aggradationsphasen von Flüssen. Die angewandten Single-Aliquot-Regenerierung (SAR)-Protokolle und die angewandten experimentellen Untersuchungen zur Absicherung der Messparameter lassen erkennen, dass die OSL-Datierungstechnik eine geeignete Datierungsmethode für fluviale Sedimente aus großen Flusssystemen ist. Eine unvollständige Bleichung der fluvialen Sedimente vor der Ablagerung scheint für die Sedimente aus Bremgarten nicht so dramatisch zu sein wie ursprünglich vermutet. OSL- und Radiokohlenstoff-Datierungsergebnisse bestätigen eine kurze Periode starker Erosion und Akkumulation im Bereich des Tiefgestades von Bremgarten zwischen 500 und 600 Jahren vor heute. Dieses Zeitfenster korreliert mit dem Beginn der Kleinen Eiszeit um ca. AD 1450. Mehrere schwere Flussüberschwemmungen geschahen in Süddeutschland zwischen AD 1500 und 1750. Diese Überschwemmungsereignisse korrelieren mit dem Zeitfenster der Kleinen Eiszeit, darunter die Zerstörung von Neuenburg im Jahre 1525.

Keywords: Upper Rhine Graben, river aggradation, OSL chronology, Little Ice Age, Quaternary

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1 Introduction

Investigating terrestrial sediment archives of the past is imperative to learning and understanding the future impact of climate and environmental changes. The Medieval climate optimum and the Little Ice Age are two historically recorded periods during which society was considerably influenced by climate and environment change. Fluvial landscapes in modern floodplains and lower terraces are particularly sensitive to climate change. Extreme weather events such as the millenium flood of the year AD 1342 had a catastrophic impact on Man and the society and indeed caused a significant alteration of landscape, such as up to 15 m deeply incised canyons and reworking of up to 8 m thick fluvial sediments in southern Germany (BORK 1989). The extent of the fluvial dynamics and the temporal succession of such events are not yet understood in either large or small rivers.

Extensive research has been carried out in the catchment area of the River Rhine on Pliocene and Pleistocene terrace formation (e.g. BOENIGK & FRECHEN 2006; HAGEDORN & BOENIGK 2008, WESTERHOFF 2008; and references within) demonstrating the complexity of this major fluvial sediment archive. The large increase in terrigenous sediment supply during the Quaternary is a result of continuous disequilibrium between weathering, erosion, sediment transport and aggradation triggered by climatic perturbations (HINDERER 2001). Increased aggradation in the southern Upper Rhine Graben (URG) postdates the filling up of the Lake Constance (Bodensee) Basin and occurred synchronous with periods of major ice melting (WESSELS 1998). During these highly dynamic periods, including the glacial overdeepening of Alpine valleys, the peri-alpine basins were deeply eroded. Late Glacial and Holocene fluvial aggradation and terrace formation are triggered by climate change and tectonic processes as well as human-induced adjustment of river systems (SCHIRMER et al. 2005; BOS et al. 2008; ERKENS et al. 2009; LÄMMERMANN-BARTHEL et al. in press). During the Little Ice Age, signifi-

cant erosion and aggradation occurred along the Rhine.

Little is known about the timing of the Holocene fluvial activity of the River Rhine in the southern Upper Rhine Graben (URG). Historical data is available for major flood events during Medieval times. Numerical dating of fluvial sediments is problematical, as described in detail for the Middle and Lower Rhine area by BOENIGK & FRECHEN (1998, 2006) and the northern Upper Rhine Graben by ERKENS et al. (2009) and FRECHEN et al. (2006). During the past 15 years, much attention has been paid on the investigation of the timing of fluvial activity in the Rhine system. Fluvial sediments are particularly suitable for the application of optically stimulated luminescence (OSL) dating techniques (BUSSCHERS 2008; JAIN et al. 2003; WALLINGA 2002). In this study, OSL dating on fine-sand and radiocarbon dating on wood was carried out on Holocene material from the gravel pit at Bremgarten in the southern part of the Upper Rhine Graben.

The aim of this study is to test the suitability of fluvial sediments for OSL dating and to give a more reliable chronological interpretation for the Medieval fluvial activity in the southern part of the URG. This is part of an ongoing study investigating fluvial sediments from the Rhine system in the frame of the Heidelberg Drilling Project.

2 Geological setting

The Upper Rhine Graben is located between the cities of Basel and Mainz. Its extension has a length of about 300 km and a width of up to 36 km and about 20 km in the southern part and in the northern part, respectively. The altitude of the River Rhine decreases from about 280 m above sea level (asl) in the south near Basel to about 80 m asl in the north near Mainz. The River Rhine flows in numerous meanders northwards, which were partly regulated in the years between 1817 and 1874.

The maximum subsidence of the Quaternary is located in the Heidelberg Basin near to the city of Heidelberg. BARTZ (1951, 1967, 1974)

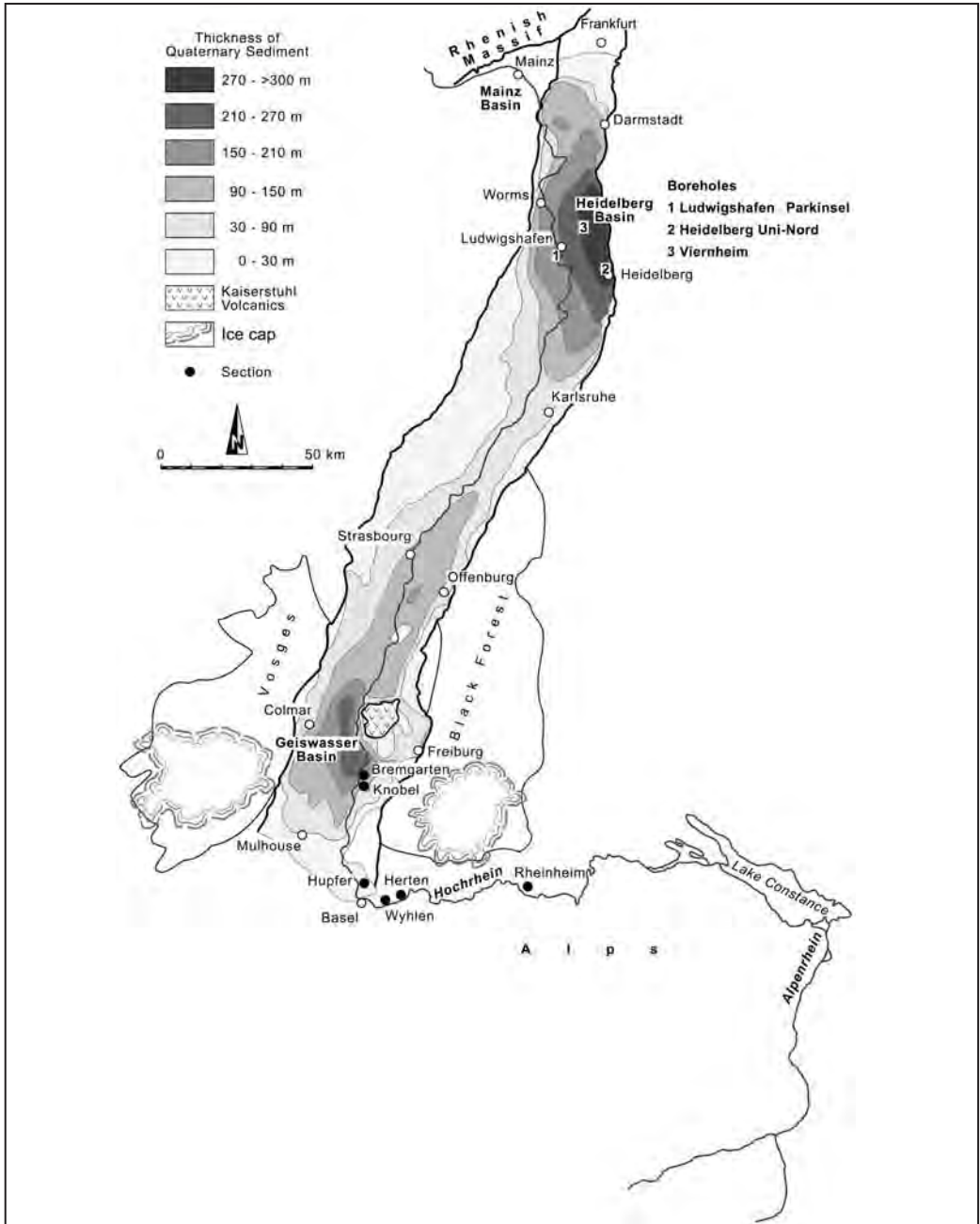


Fig. 1: Map showing the location of the Bremgarten section and the thickness of Quaternary sediments and the position of boreholes of the Heidelberg Drilling Project in the Upper Rhine Graben, modified after HAGEDORN (2004) and BARTZ (1974).

Abb. 1: Geographische Lage des Aufschlusses Bremgarten und der Bohrlokalitäten des Bohrprojektes Heidelberger Becken sowie Mächtigkeit der quartären Sedimente im Oberrheingraben modifiziert nach HAGEDORN (2004) und BARTZ (1974).

reported a thickness of more than 300 m for the Quaternary sequence and about 680 m for the Pliocene record. The Heidelberg Drilling Project including the up to 500 m long sediment cores from the sections at Heidelberg-UniNord, at Ludwigshafen-Parkinsel (WEIDENFELLER & KÄRCHER 2008) and at Viernheim, is currently aiming to reconstruct in detail the sediment archive of the basin fill (Fig. 1) (ELLWANGER et al. 2005). A zone of elevated seismic activity is evidenced by the historical earthquakes of Basel Anno Domini (AD) 1021 and AD 1356. In the southern URG, tectonically active faults have caused offsets of up to 20 m during the Holocene (HÜTTNER 1991) resulting in a distinct terrace step east of the river Rhine. The higher level is called "Hochgestade" and the lower part between the step and the river is called "Tiefgestade". The difference in altitude between "Hochgestade" and "Tiefgestade" reaches several metres and has formed by a tectonically active fault propagating parallel to the present river course (BRAM et al. 2005). NIVIÈRE et al. (2008) calculated maximum vertical movements along the faults not exceeding 1.0 mm/yr since the Middle Pleistocene. The current activity is concentrated along the westernmost faults. The coordinates of the section under investigation are H5309115 and R3394285 of the Gauss-Krüger system used in German topographic maps. The gravel pit Bremgarten GmbH is located about 150 m east of the river Rhine and situated in the lowermost part of the Lower Terrace termed "Tiefgestade" in the vicinity of the villages Bremgarten and Hartheim. About 7 m thick sand and gravel successions are exposed including pebbles originating from the Swiss Alps and the Black Forest. The upper part of the sediment succession is coarser than the lower part. A sand lens is covered by not well-sorted gravel about 2 m thick sand-rich. Up to three horizons can be distinguished and make three deposition events likely (Fig. 2, 3). Wood was collected for radiocarbon dating from the western wall about 3 m below surface. In 2006, two further trunks were exposed in the south wall and sampled for radiocarbon dating. The two trunks were located at the top of a sand layer which correlates to the base of a fining up

cycle. The trunk has a north-south orientation, which is very likely the transport direction in the river at the time of deposition (Fig. 2). The lower part of the tree has still its bark, whereas the upper part of the trunk is bare of bark, most likely owing to erosion by water and sediment immediately after deposition of the trunk.

Sample BRE3 was taken 20 cm above the trunk and sample BRE4 was taken from about 20 cm below the trunk for OSL dating. Samples BRE1 and BRE2 were taken from a sand lens intercalated in light-greyish gravel from the eastern quarry wall in 2003 (Fig. 3). A piece of wood was intercalated in the sand lens, as previously described by LÄMMERMANN-BARTHEL et al. (in press) and used for radiocarbon dating.

Massive Alpine sediment supply into the URG is unlikely for Medieval times, as lake Constance again formed as a sediment trap for the River Rhine (WESSELS 1998). In the geological past, massive Alpine sediment supply probably resulted in a lateral movement of the Rhine over the whole southern URG (LÄMMERMANN-BARTHEL et al. in press).

3 Luminescence dating

Luminescence dating of aeolian and fluvial sediments has proved to be successful where radiocarbon and other dating methods are not applicable (FRECHEN et al. 1997; JAIN et al. 2003; LIAN & ROBERTS 2006; RITTENTOUR 2008). Since the late 1980s, luminescence dating of sediments has significantly been improved by the development of more light sensitive techniques such as green or blue optically stimulated luminescence (OSL) or infrared optically stimulated luminescence (IRSL) for monomineralic quartz or feldspar subsamples, respectively (HUNTLEY et al. 1985; HÜTT et al. 1988). A recent review about the lower and upper dating limit is found in WINTLE (2008).

The basic principle of luminescence dating is solid state dosimetry of ionising radiation (WINTLE 1997; AITKEN 1998; PREUSSER et al. 2008).

The quartz signal saturates at lower doses than the feldspar signal. However, the advantage is often offset by the larger dose rates for the feld-



Fig. 2: Picture showing the position of the samples under study. A trunk is intercalated in sandy sediments (sand lense). The trunk was sampled for radiocarbon dating. Two samples were taken from sand about 20 cm below (LUM1220) and about 20 cm above (LUM1219) the trunk.

Abb. 2: Foto der Position der Probennahmepunkte im Aufschluss Bremgarten. Ein Baumstamm ist in die sandigen Sedimente zwischen geschaltet (Sandlinse). Das Holz wurde zur Radiokohlenstoff-Datierung beprobt. Jeweils eine Probe wurde 20 cm unterhalb (LUM1220) und oberhalb (LUM1219) des Baumstammes für OSL-Datierungen entnommen.

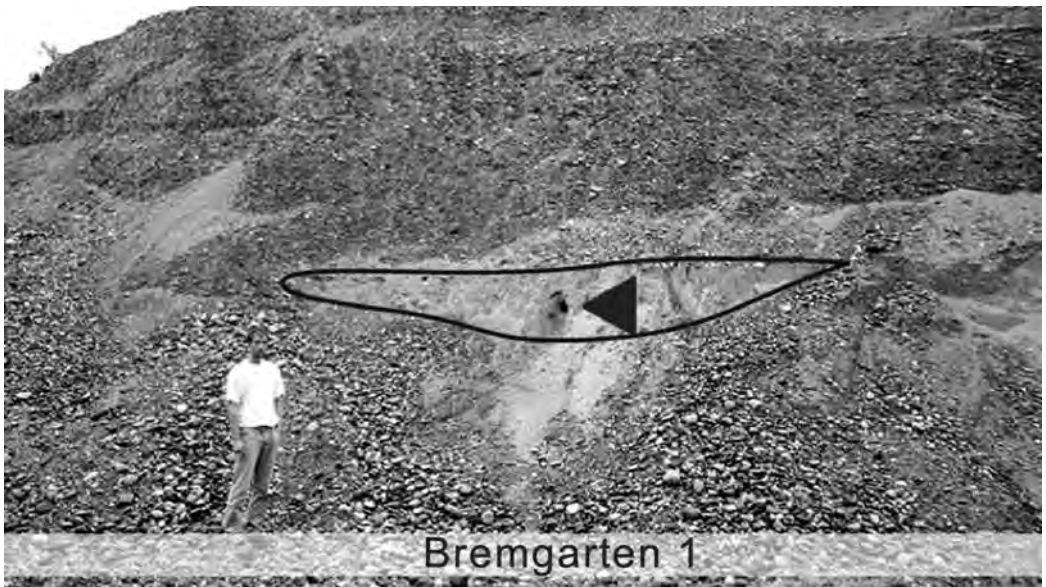


Fig. 3: Picture showing the position of sample BRE1. A second trunk was found 150 m in the north of the profile under study and sampled for radiocarbon dating.

Abb. 3: Foto mit der Position der Probe BRE1. Ein weiterer Baumstamm wurde etwa 150 m nördlich dieses Profils für eine Radiokohlenstoff-Datierung entnommen.

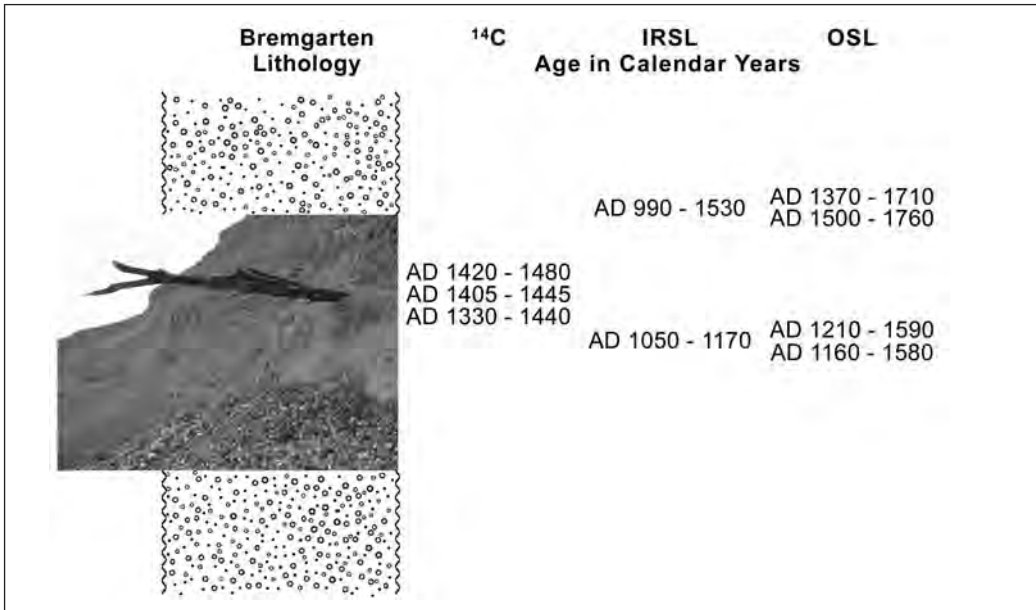


Fig. 4: Idealised sketch of the lithological units and the geological interpretation and position of radiocarbon and luminescence samples.

Abb. 4: Idealisierte Abbildung der Sedimentabfolge in Bremgarten mit geologischer Interpretation und Position der Radiokohlenstoff- und Lumineszenzproben.

spars owing to high potassium content within the feldspar mineral grains and athermal signal instability owing to anomalous fading (WINTLE 1973).

An important assumption of luminescence dating techniques is that the mineral grains were sufficiently long exposed to daylight/sunlight prior to deposition to reset the radiometric clock ("zeroing the luminescence signal"). The level of zeroing depends on the exposure time of the mineral grains to light, the available light intensity and the light spectrum. These parameters are strongly affected by fluvial sedimentary dynamics including water depth, sediment load, turbulence and turbidity, grain-size and transportation distance (RHODES & POWNALL 1994; FRECHEN 1995; MURRAY et al. 1995; WALLINGA et al. 2000; JAIN et al. 2003). In many fluvial environments the probability of complete zeroing of all sediment grains is low. JAIN et al. (2003), WALLINGA (2002) and SINGARAYER et al. (2005) pointed out that in contrast to aeolian sediment a distribution of values deter-

mined for fluvial deposits usually results in age overestimation, if equivalent doses are measured for samples containing a large number of grains. FUCHS et al. (2005) reported that quartz based equivalent dose (D_e) values yielded distinctly lower results than feldspar based D_e values for flood sediments giving evidence for better bleaching of quartz minerals extracted from the sediment. However, in large river systems the zeroing has been found to be more complete or nearly complete owing to the very likely multiple recycling of sediment during repeated phases of deposition and erosion (WALLINGA 2002). Most bleaching does occur when the grains are close to the water surface where the light intensity is greater and the light spectrum is more complete. Sediment from overbank deposits and scour pools are more likely to be suitable for luminescence dating. Samples taken from sediment of high-energy depositional environments like mass flow deposits or sediments from kames, are not suitable for luminescence dating (HÜTT & JUNGNER 1992). Partial bleaching of sediments

is also attributed to flashy seasonal river flow (floods) associated with high-amplitude precipitation causing input from bank erosion (JAIN et al. 2003). The existence of scatter in equivalent dose determinations is an indication for incomplete zeroing of the OSL signal. Grain-to-grain variations do produce scatter in single aliquot equivalent dose determinations (LAMOthe et al. 1994; MURRAY & ROBERTS 1997; OLLEY et al. 1999). The detection of partial bleaching was previously investigated by using small single aliquots or single-grain methodology (FUCHS & WAGNER 2003; SINGARAYER et al. 2005; PREUSSER et al. 2007) and different statistical approaches

(LEPPER et al. 2000; BAILEY & ARNOLD 2006; ERKENS et al. 2009). Following these studies, it appears to be mandatory to investigate a large number of single grains per sample from fluvial environments.

The first fluvial sediments from Germany, which were investigated by a dating approach combining IRSL, OSL and thermoluminescence (TL), were taken from sand layers of the Lower Terrace from the River Emscher (FRENCHEN 1995). TL dating results showed large uncertainties most likely owing to incomplete bleaching prior to deposition; IRSL dating of potassium-rich feldspars showed a better

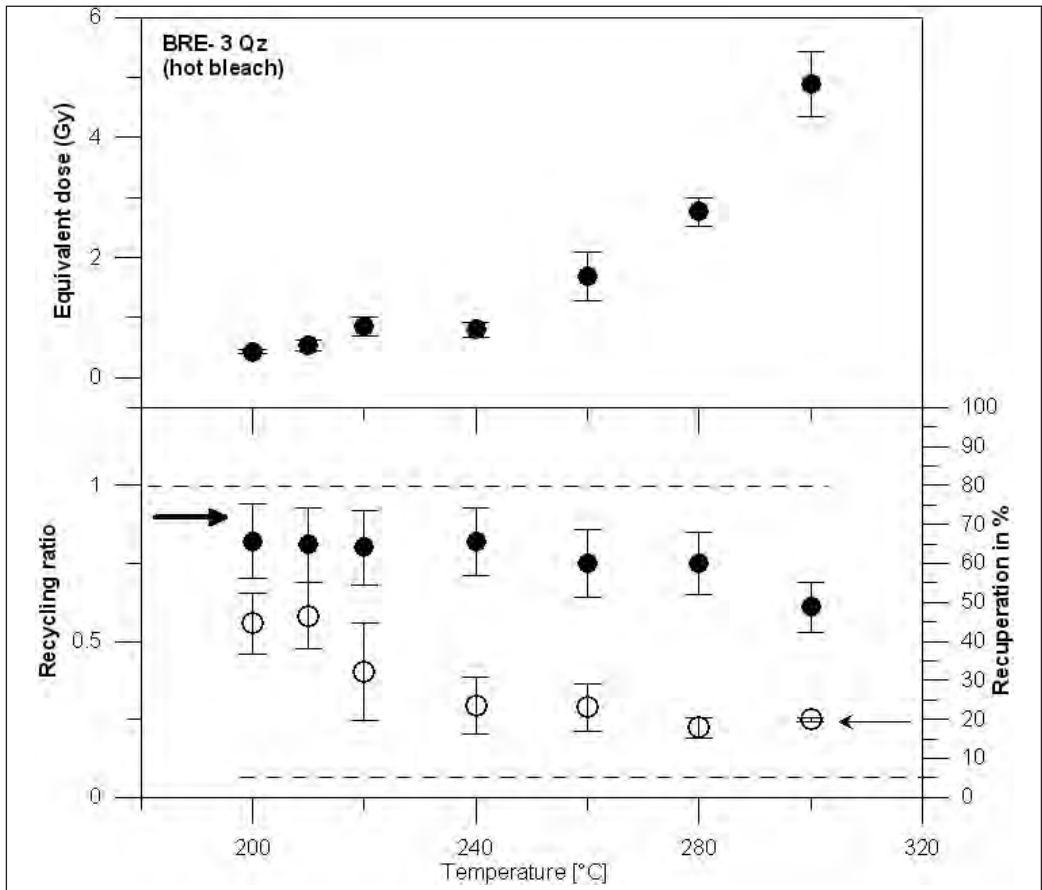


Fig. 5: Preheat plateau, recycling ratio and recuperation for sample BRE3 using quartz extracts and the hot bleach procedure.

Abb. 5: „Preheat plateau“, „Recycling Ratio“ und „Recuperation“ für Quarze der Probe BRE3 unter Anwendung des „Hot Bleach“-Verfahrens.

agreement with the geological age estimates. FRECHEN (1995) suggested to investigate 100-200 μm potassium-rich feldspar extracts to test the bleaching behaviour for different transmission wavelength.

Recent advances in instrumentation and measurement protocols have played a key role in enabling the dating of fluvial sediments. OSL dating of quartz has been significantly improved by single-aliquot regenerative (SAR) protocols (MURRAY & WINTLE 2000; WALLINGA et al. 2001). Sensitivity changes are monitored and corrected in the SAR protocol. This procedure allows the determination of equivalent dose by interpolation with a precision of up to 5 %.

Feldspar IRSL dating can be troubled by anomalous fading (WINTLE 1973) causing age underestimation. Anomalous fading is an

athermal signal loss following irradiation and reflecting the instability of some of the electron traps. HUNTLEY & LAMOTHE (2001) pointed out that it is essential to determine the fading rate precisely and carried out fading experiments to correct the measured IRSL age estimates. However, age correction is problematic and a reliable procedure is still under discussion (AUCLAIR et al. 2003; LAMOTHE et al. 2003; HUOT & LAMOTHE 2003). There is a close relation between fading rate and geological provenance. HUNTLEY & LAMOTHE (2001) determined fading rates for North American sediments ranging from 2 to 10 % per decade (decade means a factor of 10 in time since irradiation). These fading corrections are restricted to the low-dose linear portion of the dose response and are not expected to be applicable to samples

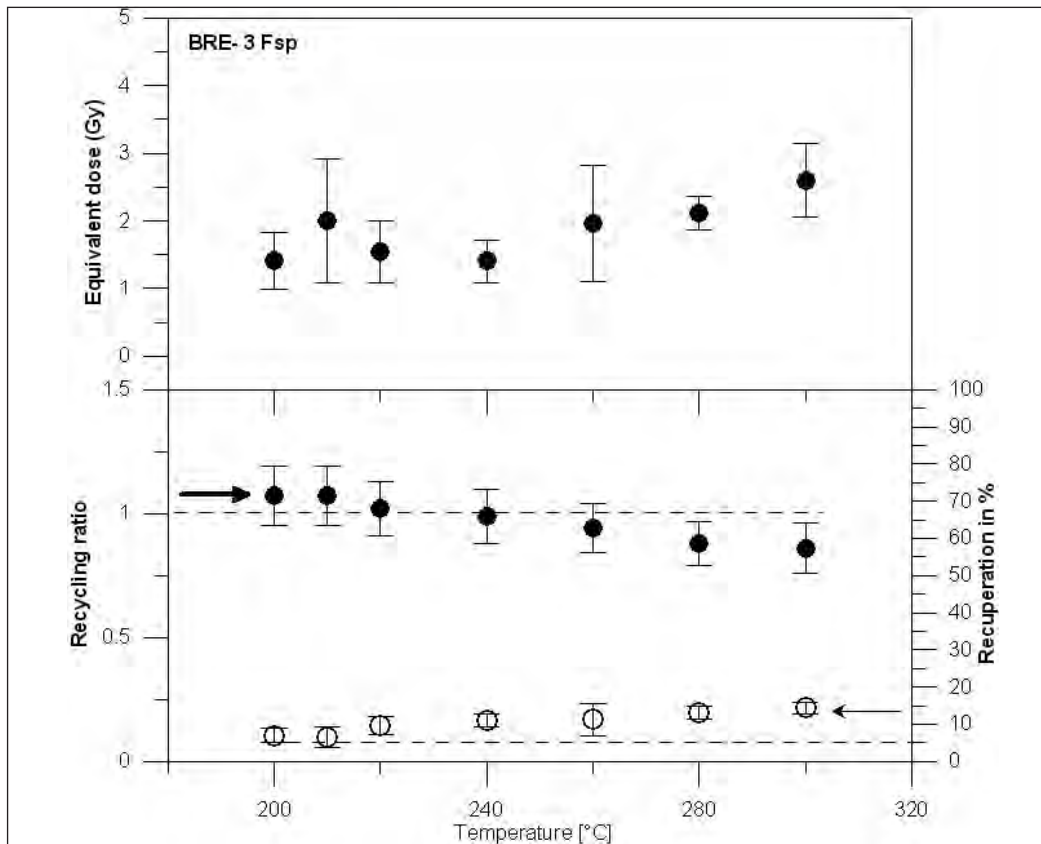


Fig. 6: Preheat plateau, recycling ratio and recuperation for sample BRE3 using feldspar extracts.

Abb. 6: „Preheat plateau“, „Recycling Ratio“ und „Recuperation“ für Feldspäte der Probe BRE3.

older than ~20 - 50 ka. Fading corrections with a potential to be applicable over a much wider range are under discussion (LAMOTHE et al. 2003). Parts of the time-dependent signal from feldspars using pulsed stimulation show less fading than others (TSUKAMOTO et al. 2006) and thus possibly providing another way of dating potassium-rich feldspars. PREUSSER et al. (2007) reported that long-term fading has apparently no effect on potassium-rich feldspar specimens from the Swiss Alpine Foreland.

Natural ionising radiation includes alpha, beta and gamma irradiation released by the radioactive decay of the unstable isotopes of the ^{238}U , ^{235}U and ^{232}Th decay chains, ^{40}K and to a minor content ^{87}Rb and cosmic radiation. The dose rate depends on a number of external factors, some of them are even variable during

burial time, like moisture, sediment thickness, grain-size, geochemical weathering of minerals or salts, mobilisation of clay minerals and radioactive equilibria/disequilibria of the decay chains and internal factors like the amount of ^{40}K in potassium-rich feldspars.

4 Experimental details and luminescence characteristics

Four luminescence samples were taken in light-tight metallic cylinders from sand lenses intercalated in gravel-rich fluvial deposits. The outer light-exposed part of the sediment was removed under subdued light at both ends of the cylinder. The sample preparation included sieving to separate the 100-200 μm or the 150-200 μm grain-size fractions, followed by

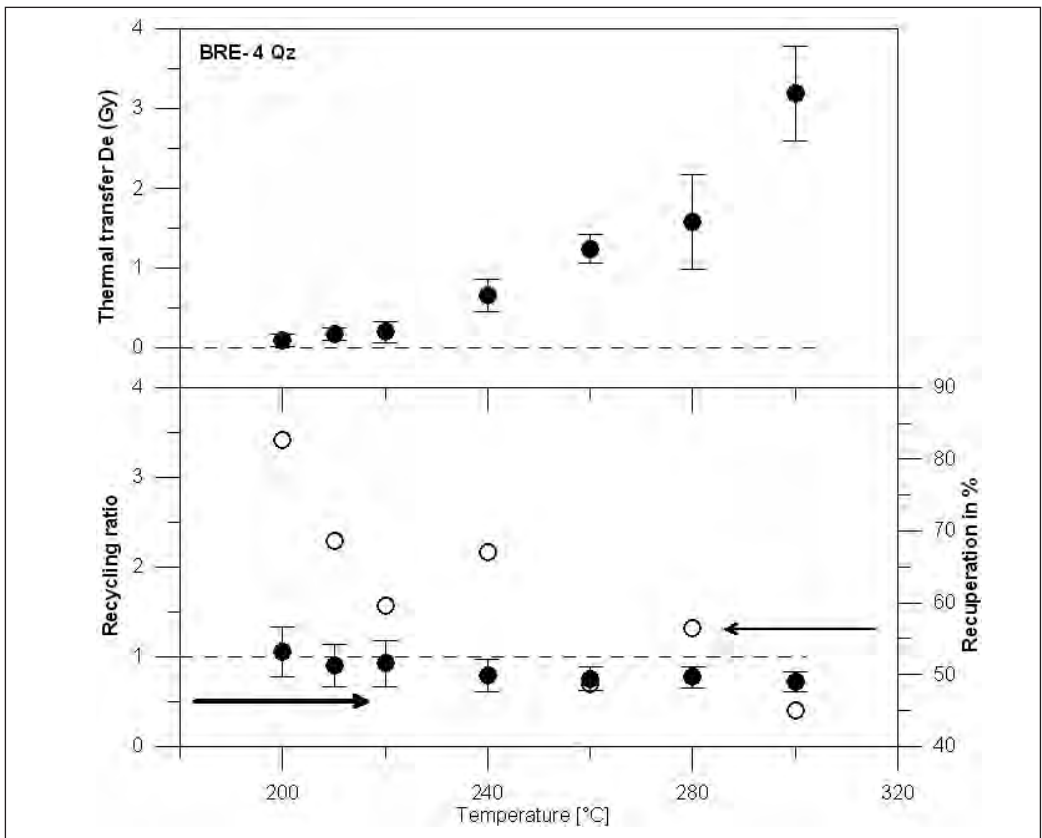


Fig. 7: Preheat plateau, recycling ratio and recuperation for sample BRE4 using quartz extracts.

Abb. 7: Preheat plateau", „Recycling Ratio" und „Recuperation" für Quarze der Probe BRE4.

removing the carbonates in 0,1 N hydrochloric acid, organic matter by 30 % hydrogen peroxide and clay particles by sodium oxalate. The sand-sized feldspar grains with densities lower than 2.58 g/cm^3 were extracted using heavy liquids and the sand-sized quartz was extracted from the remaining fraction using heavy liquids of 2.62 g/cm^3 and 2.70 g/cm^3 densities. The quartz extracts were etched with 40 % hydrofluoric acid for 60 minutes to remove feldspars and sieved again with a $100 \mu\text{m}$ mesh or $150 \mu\text{m}$ mesh. The sand-sized quartz or feldspar grains were brought onto $0,9 \text{ mm}$ steel discs (aliquots). IR stimulation was applied at the end of the quartz SAR protocol to test whether single aliquots show a contamination with feldspar deriving from inclusions after etching with hydrofluoric acid. Single aliquot regenerative-dose protocols

were carried out for monomineralic quartz and feldspar extracts to determine the De values more precisely (MURRAY & WINTLE 2000; WALLINGA et al. 2000, 2001). All growth curves were fitted using a saturating exponential function.

A number of tests were carried out on both quartz and feldspar extracts to investigate the luminescence characteristics and to evaluate the suitability of the applied SAR protocol for the samples under study.

Preheat plateau

To determine appropriate preheat conditions for the determination of De values and to avoid thermal transfer effects, the variation of equivalent dose with preheat temperature was measured for both feldspar and quartz

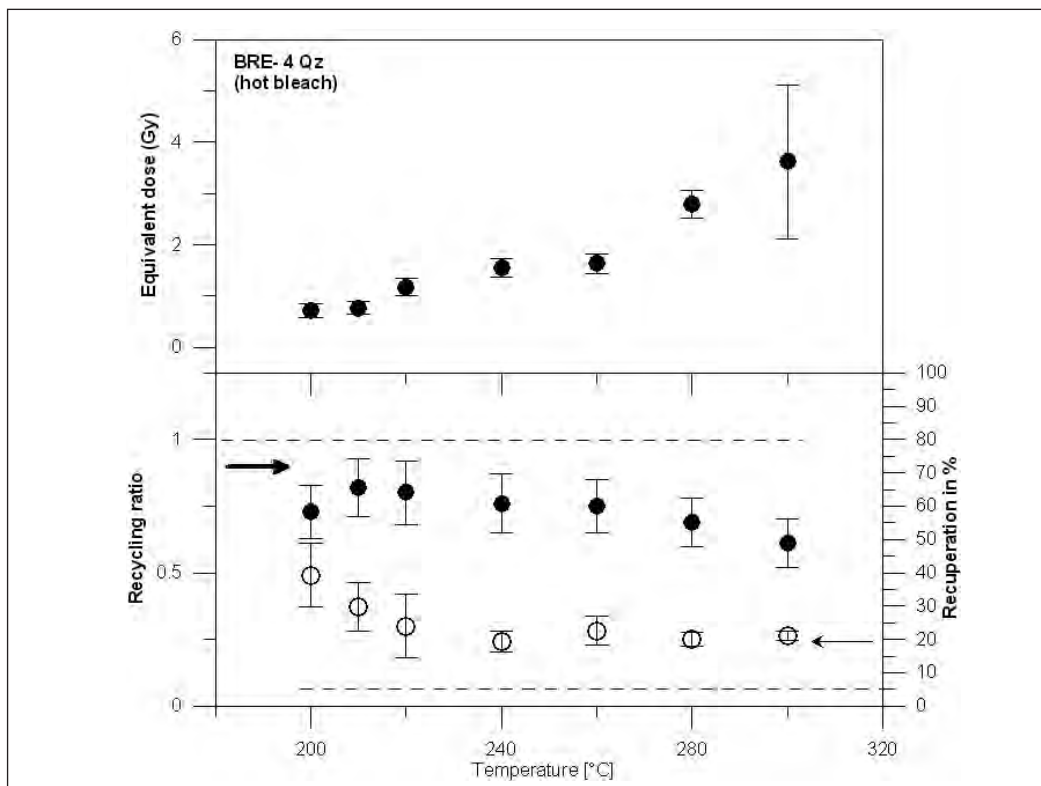


Fig. 8: Preheat plateau, recycling ratio and recuperation for sample BRE4 using quartz extracts. The hot bleach procedure was applied (MURRAY & WINTLE 2003).

Abb. 8: „Preheat plateau“, „Recycling Ratio“ und „Recuperation“ für Quarze der Probe BRE4 unter Anwendung des „Hot Bleach“-Verfahrens (MURRAY & WINTLE 2003).

extracts. A plateau of D_e values against preheat temperature for quartz and feldspar was determined using 10 seconds (s) preheats at 20°C intervals from 200 to 300°C (cp. MURRAY et al. 1997). Results were obtained from all samples. Examples of preheat plateau are given in Figs. 5-11. A preheat temperature of 210°C or 220°C for 10 seconds was used for natural and regenerative doses for quartz extracts and feldspar extracts.

Recycling ratio

Changes in the measured luminescence intensity with dose are known to occur in quartz and feldspar during single aliquot measurement procedures. Therefore a small test dose is given to each aliquot after the measurement of

the natural or regenerated signal to determine sensitivity changes. The sensitivity correction is calculated by dividing the natural or regenerated IRSL or OSL intensity (L_x) by the test dose IRSL or OSL intensity (T_x). The recycling ratio (R_x/R_1) is a test for the effectiveness of the sensitivity correction and determined by repeating the first dose point in the growth curve at the end of the measured cycle. The recycling ratio should be consistent with unity. MURRAY & WINTLE (2000) suggested that aliquots should be rejected if recycling ratios are outside 10 % of unity. However, MURRAY & WINTLE (2000) pointed out that the recycling ratio only tests whether sensitivity changes following laboratory measurements have been accurately corrected for. Figures 5-11 show plots of recycling ratios for samples BRE1-4 show-

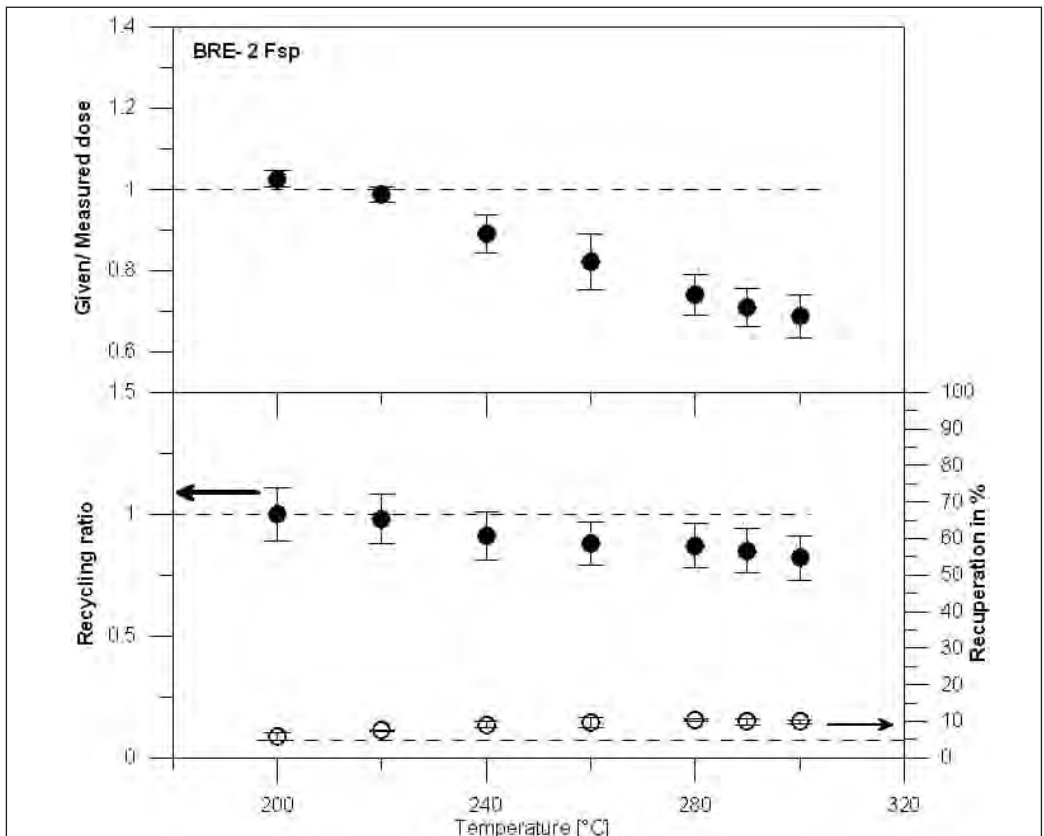


Fig. 9: Dose recovery test, recycling ratio and recuperation for sample BRE2 using feldspar extracts.

Abb. 9: „Dose recovery test“, „Recycling Ratio“ und „Recuperation“ für Feldspäte der Probe BRE2.

ing that for most of the samples the aliquots fall within 10 % of unity for the temperature range 200° to 220°C indicating that the SAR sensitivity correction is appropriate for these samples. Furthermore, a hot bleach treatment was used for the quartz extracts. After measurement of the response to the test dose, the aliquots were exposed to the blue diodes for 40 seconds whilst holding them at 280°C in order to reduce any build up of slow components in the OSL signal (Murray & Wintle, 2003). However, the recycling ratios concerning the hot bleach procedure for samples BRE3-quartz and BRE4-quartz fall only within 35 % of unity indicating that the sensitivity correction is problematic although the final OSL age estimates are within 1-standard deviation in agreement with independent age control. The SAR proto-

col for feldspar yielded recycling ratios within 10 % of unity for the preheat temperature range between 200° and 240°C.

Dose recovery test

A successful dose recovery test indicates that the SAR protocol produces internally consistent results for a sample and evaluates the credibility of the equivalent dose measured from a natural sample and so confirms that the applied SAR protocol enables to recover a known laboratory dose (RHODES 2000). After bleaching the aliquots with a dr. hönle solar simulator, the aliquots were given a known laboratory dose close to the D_e value. The artificially dosed aliquots were then treated as “natural” in the SAR protocol (Table 1) to determine the D_e value (here:

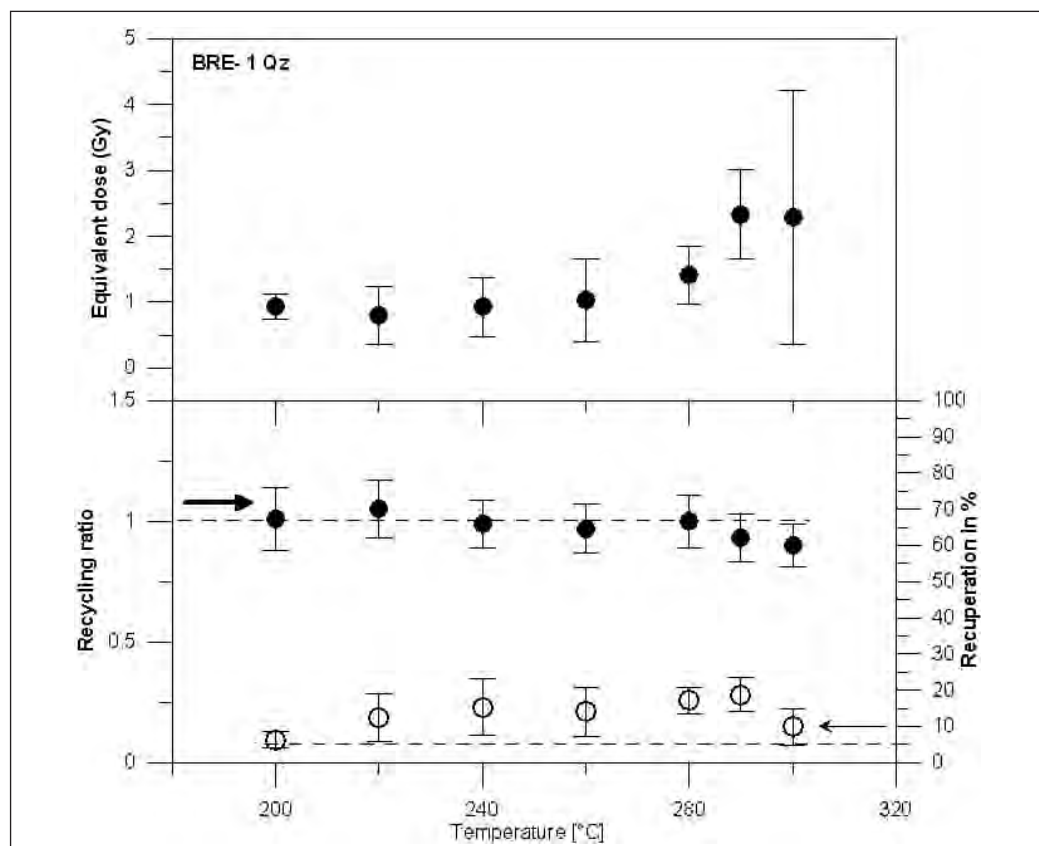


Fig. 10: Preheat plateau, recycling ratio and recuperation for sample BRE1 using quartz extracts.

Abb. 10: „Preheat plateau“, „Recycling Ratio“ und „Recuperation“ für Quarze der Probe BRE1.

dose recovery). An example for dose recovery is given for sample BRE2-feldspar using a test dose of 1.0 Gy (Fig. 9). The dose recovery tests yielded the recovering dose values within 5 % from the given dose in the preheat temperature range 200°-220°C.

An example for thermal transfer is given in Figure 7. Sample BRE4-quartz seems to be stable for the temperature range between 200° and 220°C but shows an increasing amount of thermal transfer above 220°C. Recuperation is insufficient over the whole temperature range for sample BRE4-quartz (Fig. 8), whereas recuperation is less than 10 % and even less than 5 % over the temperature range between 200° and 240°C for sample BRE2-feldspar and BRE1-quartz (Fig. 9, 10). The hot bleach procedure gives a recuperation of >20 % over

the temperature range from 200° to 300°C for sample BRE4-quartz.

Single-aliquot regenerative dose protocol

The single-aliquot regenerative (SAR) dose protocol was applied to determine the De values for feldspar and quartz (MURRAY & WINTLE 2000; WALLINGA et al. 2000, 2001; WINTLE & MURRAY 2000). A dose response curve with typically three dose points is measured on a single aliquot by repeated irradiations, preheats and IRSL/OSL measurements. Sensitivity changes occurring due to laboratory heat treatment are monitored after each OSL measurement and corrected (Table 1). The weighted mean De value and the standard deviation were calculated for most of the samples

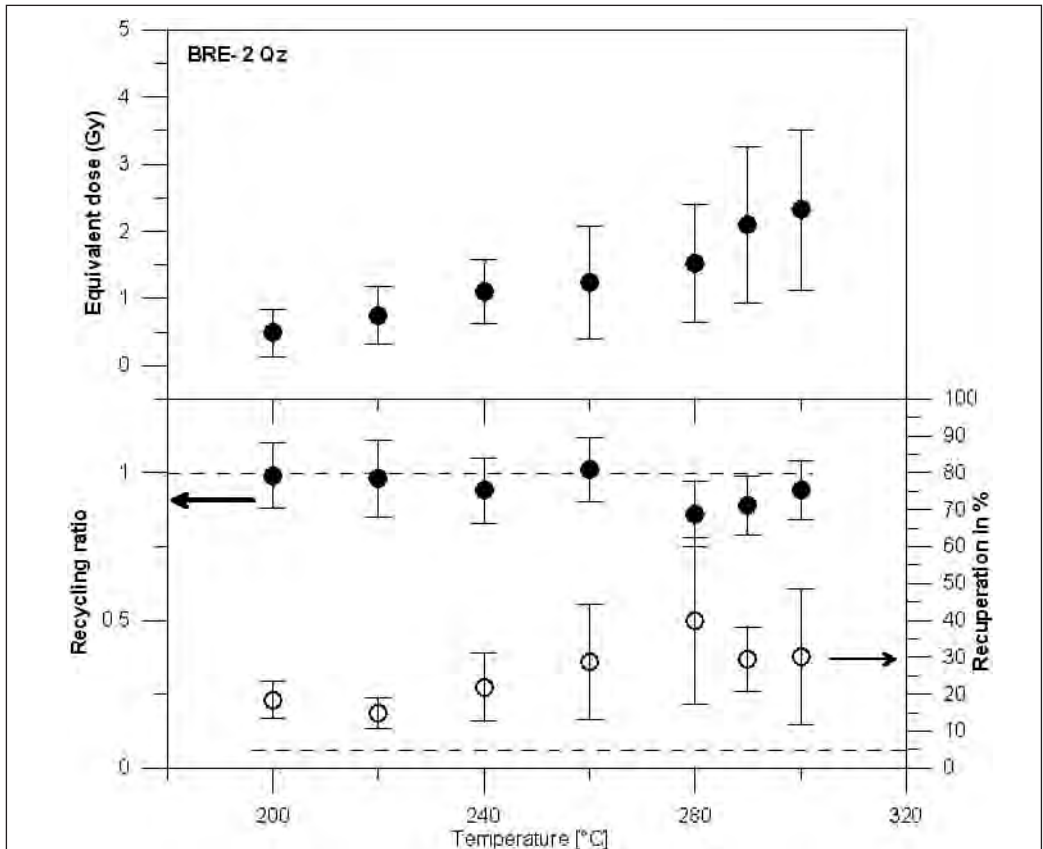


Fig. 11: Preheat plateau, recycling ratio and recuperation for sample BRE2 using quartz extracts.

Abb. 11: „Preheat plateau“, „Recycling Ratio“ und „Recuperation“ für Quarze der Probe BRE2.

from 24 aliquots for both feldspar and quartz grains. In general, aliquots were taken into account within a 3-sigma standard deviation and a recycling ratio between 0.8 and 1.2. Most of the dose points for the natural signal plotted between first and second artificial dose step. The hot bleach procedure resulted in two outliers (samples BRE3 and BRE4) with recycling ratios of 0.87. The IRSL signal was measured with a Schott BG39/Corning 7-59 filter combination between photomultiplier and feldspar extracts. A Schott U-340 filter with a detection window of 290–370 nm was placed for OSL measurements between photomultiplier and quartz extracts. Blue light emitting diodes were used for quartz stimulation.

Table 1: Single Aliquot Regenerative (SAR) protocol, as applied for quartz (Qz) (OSL stimulation) and feldspar (Fsp) grains (IRSL stimulation) (after MURRAY & WINTLE 2000; WALLINGA et al. 2000, 2001).

Tab. 1. SAR-Protokoll für Quarz (Qz) (OSL-Stimulation) - und Feldspatminerale (Fsp) (IRSL Stimulation) (nach MURRAY & WINTLE 2000; WALLINGA et al. 2000, 2001).

1. Preheat of Natural
2. IRSL decay of naturals for 300s at 50°C / OSL decay for 40s at 125°C
3. Test dose (10s)
4. Preheat of test dose of 210°C for 10s (Fsp), and 210°C for 0s (Qz)
5. Measurement of test dose (IRSL decay for 300s at 50°C for Fsp / OSL decay for 40s at 125°C for Qz)
6. Regenerative dose
7. Preheat of regenerative dose for 10s at 290°C (Fsp), 240°C or 260°C (Qz)
8. IRSL decay of regenerative dose for 300s at 50°C / OSL decay for 40s at 125°C
9. Test dose for 10s
10. Preheat of test dose at 210°C for 10s (Fsp), and 160°C or 210°C for 0s (Qz)
11. IRSL decay for 300s at 50°C / OSL decay for 40s at 125°C
12. Repeat step 6-11 for R2, R3, zero point and recycling point
etc.

Equivalent dose determination was carried out using the software Analyst 6.0 (G.A.T. Duller, Aberystwyth, unpublished). Age calculation was applied by the software ADELE (M. Krbetschek, Freiburg, unpublished).

Dosimetry

As the outer shell of the feldspar minerals was not etched by hydrofluoric acid, alpha efficiency was estimated to a mean value of 0.2 ± 0.1 for all feldspar samples (DULLER 1994). Dose rates for all samples were calculated from potassium, uranium and thorium contents, as measured by gamma spectrometry in the laboratory, assuming radioactive equilibrium for the decay chains. The following radioisotopes were measured: ^{234}Th , ^{214}Bi , ^{214}Pb and ^{212}Pb for uranium; ^{228}Ac , ^{208}Tl and ^{212}Pb for thorium and ^{40}K for potassium. An average internal potassium content of $12 \pm 0.5\%$ was applied for all feldspar samples (HUNTLEY & BARRIL 1997). Cosmic dose rate was corrected for the altitude and sediment thickness, as described by PRESCOTT & HUTTON (1994). The natural moisture content of the sediment was estimated between $10 \pm 2\%$ weight % and $15 \pm 5\%$ weight % for the samples.

5 Results

Dosimetric results, D_e values, IRSL and OSL age estimates are shown in Tables 2 and 3. Uncertainties are given in 1-sigma confidence interval. The total dose rates range from 2.21 ± 0.22 to 2.53 ± 0.11 Gy/ka and 1.60 ± 0.05 to 1.70 ± 0.06 Gy/ka for feldspar and quartz, respectively. The D_e values range from 0.6 to 1.1 Gy for quartz grains and from 1.6 to 2.0 Gray for feldspar extracts (Table 3), as determined by the SAR protocol (Table 1). Examples for the quartz and feldspar D_e -distributions of the single aliquot equivalent dose estimates and the radial plots of the same values are shown in figs. 12-15. They give an impression on the complete resetting of the OSL- signal prior to deposition. The radial plot (Fig. 15) displays an expanded D_e - distribution for the feldspar sample BRE4 indicating an insufficient bleaching of these minerals. In

Table 2: Dosimetric results, as determined by gamma spectrometry (ppm=parts per million; H₂O = moisture of sediment in weight %).Tab. 2: Dosimetrische Ergebnisse durch Gamma spektrometrie bestimmt (ppm = part per million; H₂O = moisture of sediment in weight %).

Sample	LUM	Depth [m]	Grain Size [μm]	Uranium [ppm]	Thorium [ppm]
BRE 1	63	3.50	150-200	0.90±0.03	3.08±0.06
BRE 2	62	3.40	150-200	1.17±0.03	3.95±0.08
BRE 3	1219	3.00	150-200	1.13±0.01	3.78±0.03
BRE 4	1220	3.20	150-200	1.03±0.01	3.49±0.03

Potassium [%]	Cosmic [μGy/a]	H ₂ O [%]	Dose rate ¹ [Gy/ka]	Dose rate ² [Gy/ka]
1.29±0.02	120±12	10±2	2.39±0.10	1.60±0.05
1.28±0.03	120±12	10±2	2.53±0.11	1.70±0.06
1.18±0.01	120±12	15±5	2.23±0.22	1.62±0.16
1.21±0.01	120±12	15±5	2.21±0.22	1.61±0.16

contrast the quartz (Fig. 12) of BRE4 (Fig. 14) shows a nearly typical Gaussian distribution indicating that the mean equivalent dose is an appropriate estimate for the burial dose. OSL age estimates are used for chronostratigraphic interpretation only. IRSL age estimates are used for methodological comparison in this study only.

Two samples (BRE1 and BRE2) were taken from a sand lens at a depth of 3.40 m and 3.50 m below surface at the Bremgarten section in 2003. The IRSL age estimates gave 0.9±0.1 ka and 1.0±0.1 ka, whereas OSL age estimates yielded 1.9±0.2 ka and 1.4±0.1 ka. It is likely that the OSL age estimates for the two samples from Bremgarten are overestimated owing to the chosen preheat temperature of 260°C causing thermal transfer. Further experiments were unfortunately not possible owing to the lack of material. The samples BRE3 and BRE4 were taken in 2006 at the eastern wall of the gravel pit.

Sample BRE3 taken above a trunk, which was dated by radiocarbon, gave an OSL age estimate of 460±170 a, whereas the IRSL age estimate on feldspar extracts yielded 740±270 a.

The hot bleach procedure yielded an OSL age estimate of 370±130 a, slightly age underestimated if compared with the radiocarbon ages on wood from the same profile. Age underestimation is also indicated by the dose recovery test. Sample BRE4 taken from below the trunk (Fig. 2) yielded OSL age estimates of 690±160 a and 600±190 a (hot bleach), which are in agreement within 1-sigma standard deviation with the results from sample BRE3. The IRSL age estimate is 890±60 a.

A trunk of an oak tree was sampled from the fluvial deposits at a depth of 3.00 m below surface from the Bremgarten section and studied by radiocarbon dating. The sand most likely correlates to the top of the second fining-up cycle, whereas the trunk postdates the deposition of the second fining-up cycle. The wood is intercalated between the sand layers of samples BRE1 and BRE2. The wood gave a conventional radiocarbon age of 510±45 BP (Hv 25350) resulting in a calibrated age of AD 1330–1440 (Table 4). The chronological results are stratigraphically consistent and in agree-

ment with geological estimates. Two samples from a second trunk of the eastern wall taken in the same stratigraphic position and intercalating the sand layers of samples BRE3 and BRE4 gave radiocarbon ages of 440 ± 45 BP and 485 ± 45 BP resulting in calibrated radiocarbon ages of AD 1420-1480 and AD 1405-1445, respectively. The latter trunk from the eastern wall has about 40 tree-rings. Little variation in the thickness of the annual tree rings made the trunk unsuitable for dendrochronology (H.H. Leuschner, pers. com. 10/03/2008).

6 Discussion and Conclusion

In this study, we are confident that most of the quartz extracts yielded reliable results and uncertainties for D_e values. The resulting OSL age estimates are in excellent agreement with the radiocarbon ages indicating that in large fluvial systems the mineral grains are sufficiently zeroed prior to final deposition owing to several cycles of remobilisation. Furthermore, the results show that OSL methods are suitable for dating fluvial deposits. It is important to note that age overestimation owing to insufficient bleaching prior to deposition does not seem to be dramatic for the sediments under study. But OSL dating is not precise enough to set up an annual or decadal resolution for fluvial sediment records owing to the error between 5 and 10 % and the possibility

of insufficient bleaching. IRSL dating yielded very likely age overestimation for the fluvial sands from the Bremgarten section, if compared with radiocarbon data and OSL dating results. Radiocarbon ages and IRSL age estimates are in agreement within 1-sigma standard deviation. From a stratigraphical point of view, the IRSL dating results are also reasonable and could give the true deposition age. However, fading correction was not applied for these feldspar extracts. A similar behaviour was described for samples from historically deposited sediments from the Rhine-Meuse system in the Netherlands (WALLINGA et al. 2001; ERKENS et al. 2009). The likely reason for this age overestimation is insufficient bleaching prior to deposition. However, the results are in stratigraphic order and small offsets for the youngest samples indicate that insufficient bleaching does not seem to be dramatic for the samples from Bremgarten. It is still under discussion whether changes in sensitivity during the measurement of the natural signal or contamination of the quartz fast component OSL signal by a less stable slow component are responsible for the IRSL age overestimation alternatively OSL age underestimation. It remains risky to extrapolate the results of Holocene fluvial sediments to those fluvially transported and deposited during the Weichselian glaciation. Heterogenous radiation fields including an unequal distribution of radioisotopes in the sedi-

Table 3: Equivalent dose (D_e) values in Gray (Gy) and luminescence age estimates in 1000 years (ka) for quartz and feldspar extracts using SAR protocols.

Tab. 3: Äquivalentdosis (D_e) in Gray (Gy) und Lumineszenz-Alter in 1000 Jahren (ka) für Quarz- und Feldspatextrakte mit einem SAR-Protokoll gemessen.

Sample	LUM	De value in [Gy]		OSL Age in [ka]	
		SAR-Fsp	SAR-Qz	SAR-Fsp	SAR-Qz
BRE1	62	2.1 ± 0.1	3.0 ± 0.3	0.88 ± 0.06	1.9 ± 0.2
BRE2	63	2.4 ± 0.1	2.4 ± 0.1	0.95 ± 0.06	1.4 ± 0.1
BRE3	1219	1.6 ± 0.6	0.75 ± 0.27	0.74 ± 0.27	0.46 ± 0.17
Hot bleach	1219		0.59 ± 0.21		0.37 ± 0.13
BRE4	1220	2.0 ± 0.5	1.10 ± 0.26	0.89 ± 0.06	0.68 ± 0.16
Hot bleach	1220		0.97 ± 0.26		0.60 ± 0.19

Table 4: Radiocarbon data.

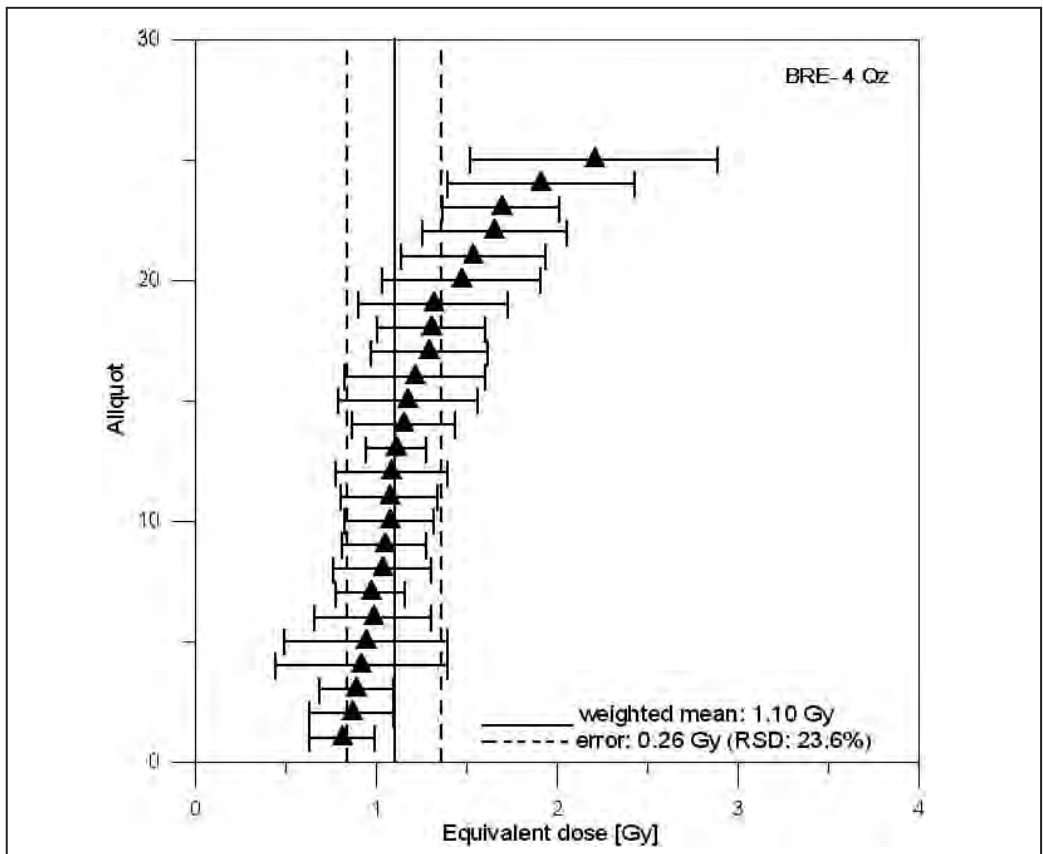
Tab. 4: Radiokohlenstoff-Daten.

Hv	Sample	Material	Depth	$\delta^{13}\text{C}$	^{14}C Age	calibrated time interval
			m	‰	Years BP.	cal ...
25350	BRE 1	Holz	-	-26.5	510 ± 45	AD 1330 – 1440
25637	BRE 2	Holz	-	-24.9	440 ± 45	AD 1420 – 1480
25638	BRE 3	Holz	-	-25.3	485 ± 45	AD 1405 – 1445

ment and the occurrence of insufficient bleaching prior to deposition can cause problems for the determination of D_e values, if small aliquots or single-grain techniques are applied (MAYYA et al. 2006). Titanite, monazite and zircon are uranium- and thorium-rich minerals resulting in radioactive hot spots in the sedi-

ments and thus giving reason for heterogenous microdosimetry, as previously described by using spatially resolved detection of luminescence (GREILICH & WAGNER 2006).

At about 2800 cal BP during the transition from Subboreal to Subatlantic, climate abruptly changed from a relatively warm and continental

Fig. 12: Distribution of D_e values in increased order for quartz extracts of sample BRE4.Abb. 12: Verteilung der D_e -Werte von Probe BRE4 (Quarz) in ansteigender Richtung angeordnet.

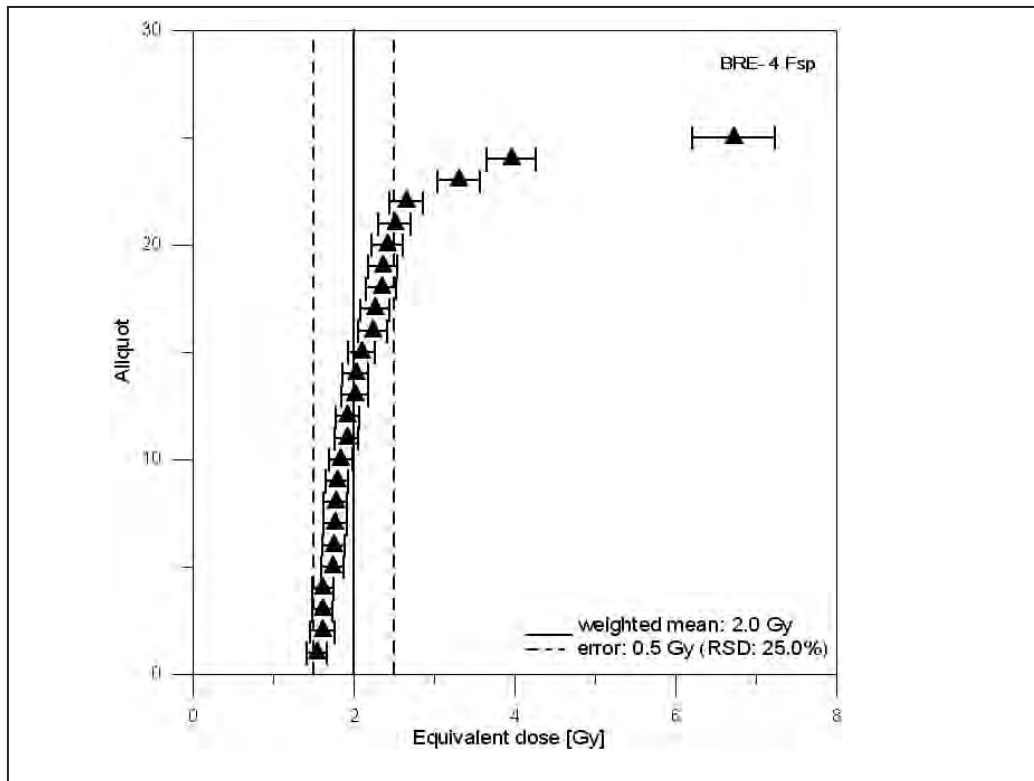


Fig. 13: Distribution of De values in increased order for feldspar extracts of sample BRE4.

Abb. 13: Verteilung der De-Werte von Probe BRE4 (Feldspat) in ansteigender Richtung angeordnet.

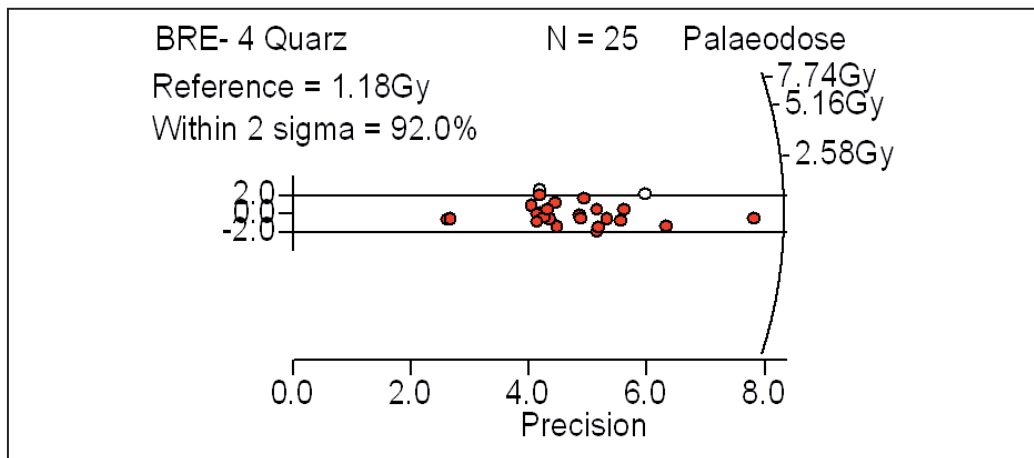


Fig. 14: Radial plot of De values for quartz extracts of sample BRE4. 92 % of the data points are within the 2-sigma standard deviation.

Abb. 14: Radialplot der De-Werte von Probe BRE4 (Quarz). 92 % der Datenpunkte liegen innerhalb der 2-sigma Standardabweichung.

climate to cooler and wetter conditions. The wetter climate in combination with intense human activity caused enhanced deforestation resulting in an increased erosion and run-off. Large amounts of waterlogged sediment were transported into the River Rhine causing a change in the meandering pattern of the river (Bos et al. 2008). Luminescence and radiocarbon dating results give evidence for a short period of major erosion and re-sedimentation of fluvial sediments from the "Tiefgestade" at the Bremgarten section between 500 and 600 years before present. This time period correlates with the beginning of the Little Ice Age lasting from about AD 1450 to 1850 (BORK 1989). Several severe floods occurred between 1500 and 1750, all correlated to the Little Ice Age; e.g. destruction of the village of Neuenburg AD 1525 (GLASER 2001). Furthermore, an extreme weather event resulting in a millenium flood is described for the year AD 1342. This flood caused a significant alteration of landscape such as up to 15 m deep incised canyons and reworking of 6-8 m thick fluvial sediments as debris flows in southern Germany (BORK 1989). The weather anomaly of AD 1342 could be very likely the reason for the fluvial dynamics including the aggradation of fluvial sediments more than 3 m thick in the southern

URG. The latest radiocarbon data make a correlation of this major sediment mobilisation with the weather anomaly of AD 1342 unlikely.

Based on the radiocarbon data, the serious historical earthquake around the city of Basel in the year AD 1356 seems to be also unlikely to have triggered such a flood causing erosion and accumulation of about 3.50 m of gravel at the Bremgarten section. However, the OSL age estimates do not clearly exclude a deposition age around the weather anomaly of AD 1342 or the Basel earthquake in the year AD 1356 owing to the large error.

In the Netherlands, the youngest drift sands from the Maas River yielded a radiocarbon age of <1000 BP and an OSL age estimate of 0.6 ± 0.1 ka (BATEMAN & VAN HUISSTEDEN 1999) and so might be correlated with the remobilised fluvial sediments from the southern URG.

The present data set leaves us uncomfortably with questions about the linkage of the aggradation periods with climate forcing of the Rhine system in southern Germany. The results contradict the cold climate origin of terrace sediments in the southern Upper Rhine Graben and open up new challenging questions. Methodological problems, such as the degree of bleaching during fluvial transport are still

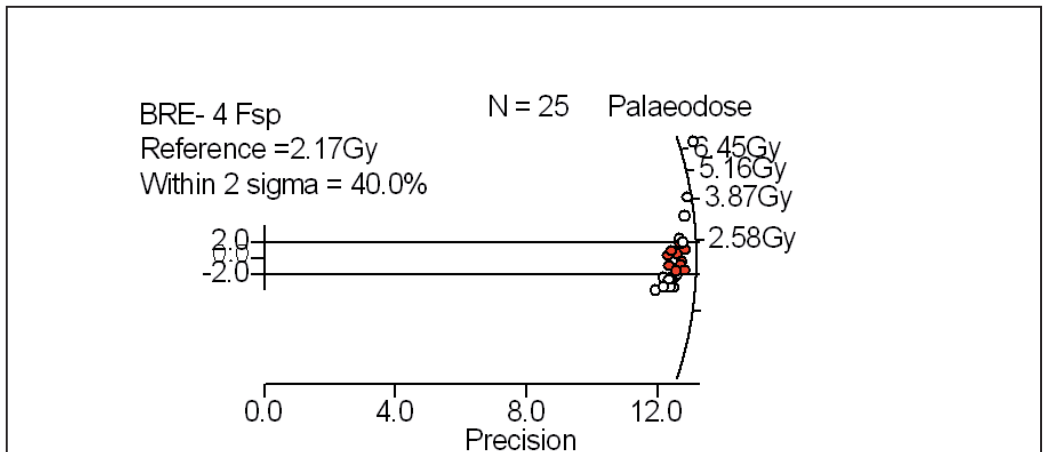


Fig. 15: Radial plot of De values for feldspar extracts of sample BRE4. 40 % of the data points are within the 2-sigma standard deviation.

Abb. 15: Radialplot der De-Werte von Probe BRE4 (Feldspat). 40 % der Datenpunkte liegen innerhalb der 2-sigma Standardabweichung.

difficult to determine, although age overestimation in large river systems seems to be not as dramatical as previously described. The quartz SAR protocol is very likely the best method to apply OSL dating to fluvial deposits. However, the IRSL age estimates, although not fading corrected, are also in agreement within the 1-sigma standard deviation, if compared to radiocarbon dates and so give also likely true deposition ages. This is part of an ongoing study investigating the timing of periods of increased aggradation in the Rhine system highlighting the response of a Central European large river system situated in a tectonically active region to environmental and climate change.

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