Technical Note: *n*-Alkane lipid biomarkers in loess: post-sedimentary or syn-sedimentary?

M. Zech\textsuperscript{1,2,3}, S. Kreutzer\textsuperscript{2,4}, T. Goslar\textsuperscript{5}, S. Meszner\textsuperscript{6}, T. Krause\textsuperscript{1,2,6}, D. Faust\textsuperscript{6}, and M. Fuchs\textsuperscript{4}

\textsuperscript{1}Department of Soil Physics, University of Bayreuth, Universitätsstr. 30, 95440 Bayreuth, Germany
\textsuperscript{2}Geographical Institute, Geomorphology, University of Bayreuth, Universitätsstr. 30, 95440 Bayreuth, Germany
\textsuperscript{3}Terrestrial Biogeosciences, Martin-Luther-Universität Halle-Wittenberg, Von-Seckendorff-Platz 3, 06120 Halle (Saale), Germany
\textsuperscript{4}Department of Geography, Justus-Liebig-University Giessen, Senckenbergstr. 1, 35390 Giessen, Germany
\textsuperscript{5}Poznan Radiocarbon Laboratory, ul. Rubiez 46, 61-612 Poznan, Poland
\textsuperscript{6}Department of Geography, Chair of Physical Geography, Dresden University of Technology, Helmholtzstr. 10, 01069 Dresden, Germany
Abstract

There is an ongoing discussion whether \( n \)-alkane biomarkers – and organic matter (OM) from loess in general – reflect a syn-sedimentary paleoenvironmental and paleoclimate signal or whether they are significantly a post-sedimentary feature contaminated by root-derived OM. We present first radiocarbon data for the \( n \)-alkane fraction of lipid extracts and for the first time luminescence ages for the Middle to Late Weichselian loess-paleosol sequence of Gleina in Saxony, Germany. Comparison of these biomarker ages with sedimentation ages as assessed by optically stimulated luminescence (OSL) dating shows that one \( n \)-alkane sample features a syn-sedimentary age (\(^{14}\)C: 29.2±1.4 kyrcalBP versus OSL: 27.3±3.0 kyr). By contrast, the \(^{14}\)C ages derived from the other \( n \)-alkane samples are clearly younger (20.3±0.7 kyrcalBP, 22.1±0.7 kyrcalBP and 29.8±1.4 kyrcalBP) than the corresponding OSL ages (26.6±3.1 kyr, 32.0±3.5 kyr and 45.6±5.3 kyr). This finding suggests that a post-sedimentary \( n \)-alkane contamination presumably by roots has occurred.

In order to estimate the post-sedimentary \( n \)-alkane contamination more quantitatively, we applied a \(^{14}\)C mass balance calculation based on the measured pMC (percent modern carbon) values, the calculated syn-sedimentary pMC values and pMC values suspected to reflect likely time points of post-sedimentary contamination (current, modern, 3 kyr, 6 kyr and 9 kyr). Accordingly, current and modern root-contamination would account for up to 7%, a 3 kyr old root-contamination for up to 10%, and an Early and Middle Holocene root-contamination for up to 20% of the total sedimentary \( n \)-alkane pool.

We acknowledge and encourage that these first radiocarbon results need further confirmation both from other loess-paleosol sequences and for different biomarkers, e.g. carboxylic acids or alcohols as further lipid biomarkers.
1 Introduction

Loess-paleosol sequences (LPSs) are important terrestrial archives for reconstructing Quaternary climate and landscape history (Frechen, 2011; Markovic et al., 2011; Zöller and Faust, 2009). Although biomarkers (also called molecular fossils) have been proven for decades to be of outstanding value in marine and lacustrine sediments (Eglinton and Eglinton, 2008), they are investigated in LPSs only relatively recently (M. Zech et al., 2011b). This has several reasons, amongst others the generally very low concentration of organic matter in loess. Yet, one famous application is the use of amino acid racemisation for establishing geochronologies (Novothny et al., 2009; Oches and McCoy, 2001; M. Zech et al., 2008).

During the last decade, additionally plant leaf wax derived lipids, namely n-alkanes came into the focus of organic geochemists focussing on loess research (Bai et al., 2009; Xie et al., 2003; Zhang et al., 2006). This can be partly attributed to the relatively easy lipid extraction procedure, which allows obtaining results even from very organic-poor loess samples. On the one hand, n-alkanes are used to infer vegetations changes in terms of grasses (dominated by the alkane homologues n-C_{31} and n-C_{33}) versus trees (dominated by the alkane homologues n-C_{27} and n-C_{29}). Only very recently, it was highlighted that a correction procedure for degradation is indispensable to avoid misinterpretations (Buggle et al., 2010; M. Zech et al., 2009, 2012a). On the other hand, the compound-specific deuterium/hydrogen (D/H) ratio of n-alkanes is used to infer paleoclimatic information (Liu and Huang, 2005; R. Zech et al., 2011).

A major and crucial assumption of all studies focussing on n-alkanes, as well as on any other biomarker in loess, is that the n-alkanes are deposited syn-sedimentary and are not contaminated significantly by post-sedimentary processes. While this seems not to hold true for certain biomarkers, e.g. because of subsurface production of archaea- and bacteria-derived membrane lipids (GDGTs = glycerol dialkyl glycerol tetraethers) called “growth depth effect” (R. Zech et al., 2012), post-sedimentary n-alkane contamination in loess is still highly disputed. Given that n-alkanes are
hydrophobic, they are not dissolved in and leached by soil water. Furthermore, the admitted still very scarce data about \( n \)-alkane concentrations in roots point to much lower concentrations compared to leaves (Huang et al., 2011; M. Zech et al., 2012b). However, it was highlighted recently, that a significant soil microbial contribution during early litter degradation does not only occur for short- and middle-, but also for the long-chain \( n \)-alkanes (Nguyen Tu et al., 2011; M. Zech et al., 2011). Last but not least, Andersson et al. (2011) showed that the contribution of \( n \)-alkanes from rootlets has to be considered in peat sequences, and studying rhizolith organic matter (OM) in loess, Gocke et al. (2010, 2011) conclude that overprinting of loess OM even in loess distant to former roots cannot be excluded. While \(^{14}C\)-dating of the \( n \)-alkane fraction has already been proven to be a powerful tool in soil science and sea sediments (Huang et al., 1996; Kusch et al., 2010; Lichtfouse and Eglinton, 1995), according to our knowledge, \(^{14}C\)-dating of alkanes is not yet applied in loess research.

The aim of this study is to evaluate the potential of \( n \)-alkane-specific \(^{14}C\)-dating for answering the question whether \( n \)-alkane biomarkers in loess are of syn-sedimentary or post-sedimentary age. Here we present for the first time a comparison of \( n \)-alkane \(^{14}C\)-ages with sedimentation ages obtained by optically stimulated luminescence (OSL) dating. This approach allows quantitatively estimating the amount of post-sedimentary contamination and therefore helping to shed light on the crucial question of post-sedimentary \( n \)-alkane contamination in loess.

2 Material and methods

2.1 The Gleina loess-paleosol sequence

Loess and paleosol samples were collected from the Gleina loess-paleosol sequence (51° 13′ 58″ N, 13° 14′ 39″ E), which is located in the Saxonian Loess Region, Eastern Germany. A detailed description of the stratigraphy was provided previously by Meszner et al. (2011). In brief, the uppermost meter is built up of decalcified material.
representing the Holocene pedogenetic impact. Major parts of the Holocene soil (presumably a Luvisol) are truncated and a plough horizon (Ap-horizon) nowadays forms the topsoil. Below, down to approximately 8 m depth, six paleosols, which can be described as gelic Gleysols and brown tundra Gleysols, alternate with loess layers (Fig. 1a). From approximately 8 m on, there is strong evidence for accumulation of partly reworked material. In situ pedogenesis contributed to the formation of a gelic Gleysol in approximately 8.5 m depth and to the formation of the decalcified so-called “Gleina complex” in approximately 10 m depth. The underlying so-called “Lommatzsch Complex” was sampled from a nearby profile (approximately 20 m) and is separated in the sketch by an unconformity.

Modern root contamination partly occurred in the upper 4.5 m of the sequence, whereas hardly any modern roots were found in the lower part of the sequence. The risk of a modern lateral root contamination was minimised by setting back the exposure wall by at least half a meter over the whole height of the exposure. Some few rhizoliths, possibly documenting a Holocene root contamination were found in the gelic gleysol in 2 m depth.

### 2.2 n-Alkane preparation, radiocarbon dating and percentage estimation of post-sedimentary n-alkane contamination by roots

For radiocarbon dating of the n-alkane fraction, four samples were selected from the Gleina loess-paleosol sequence based on the n-alkane concentrations published by M. Zech et al. (2012a) (Fig. 1). Additionally, four presumably ~160 kyr old samples from a NE-Siberian permafrost paleosols sequence (M. Zech et al., 2010) were analysed as blanks (Table 1).

The n-alkane preparation followed mainly the procedure described by Zech and Glaser (2008), however being adopted by using Soxhlet extraction (methanol/toluene, 7/3) and glass fibre extraction thimbles, which were pre-heated to 300°C for several hours. After saponification of the total lipid extract by heating with sodium hydroxide (NaOH, 0.5 M in methanol) the n-alkane fraction was solvent-extracted with hexane.
(2 x 3 ml) and purified over silica/aluminium oxide (each 3 g and 5 % deactivated) columns, from which they were eluted with hexane/toluene (85/15). The \( n \)-alkane patterns and relative abundance of the \( n \)-alkanes were determined by gas chromatography (HP 6890) using a flame ionisation detector (chromatograms are shown in Fig. 1).

For radiocarbon analysis, the \( n \)-alkane fractions (500 to 2000 µg) in the GC-vials were dried up, redissolved in dichloromethane (DCM) and transferred to quartz combustion tubes in the Poznan Radiocarbon Laboratory, Poland. After drying out DCM, the samples were washed with ethanol; the ethanol was sucked out with a pipette and dried out. Finally, the samples were washed with water; again, water was sucked out with a pipette and the samples were dried out. Tests with fossil coal showed that only this procedure ensured the quantitative removal of solvent-derived carbon (Table 1).

The two blank samples from Siberia, which were treated with the washing procedure described above, yielded pMC (percent modern carbon) values of 2.53 and 2.27, respectively (Table 1). Given that this is clearly above the normally measured background values of 0.3 ± 0.1 pMC, we suppose that this has to be attributed to a slight but seemingly reproducible \( ^{14} \)C contamination during \( n \)-alkane extraction and preparation. By contrast, we exclude a modern in-situ contamination for the Siberian soil samples because of permafrost conditions. Hence, we performed a background correction for sample pMC values assuming a 2.4 ± 0.8 % contamination with modern carbon (\(~ 100 \) pMC). Calibration of the \( ^{14} \)C ages was done with quickcal2007 vers.1.5 (Danzeglocke et al., 2012).

Given that furthermore three of the \( n \)-alkane samples yielded calibrated \( ^{14} \)C ages being clearly younger than the respective sedimentation ages as assessed by OSL dating (Fig. 1), we estimated for these samples the syn-sedimentary pMC values based on the (partly interpolated) OSL ages according to the equation

\[
pMC_{\text{syn-sedimentary}} = 100 \times \left( \frac{1}{2} \right)^{\frac{t}{T_{1/2}}},
\]  

(1)
where 100 represents the modern $^{14}$C value, i.e. 100 pMC, $t$ is the estimated uncalibrated $^{14}$C age (assuming that OSL age = calibrated $^{14}$C age and using quickcal2007 vers.1.4, Danzeglocke et al., 2012) and $T_{1/2}$ is the so-called Libby’s half-life time of radiocarbon (5.568 kyr). Using the same equation we also estimated the pMC$_{\text{post-sedimentary}}$ values for suspected Holocene times of post-sedimentary $n$-alkane contamination by roots (3, 6 and 9 kyr, respectively; Table 2).

The percentage of post-sedimentary $n$-alkane contamination “$x$” was then estimated using a $^{14}$C mass balance calculation according to the equation

$$x\% = \frac{\text{pMC}_{\text{measured}} - \text{pMC}_{\text{syn-sedimentary}}}{\text{pMC}_{\text{post-sedimentary}} - \text{pMC}_{\text{syn-sedimentary}}}.$$

(2)

2.3 Optically stimulated luminescence (OSL) dating

A numerical chronostratigraphy of the section Gleina was established by OSL dating using fine grain (4–11 µm) quartz separates. Therefore eight luminescence samples were taken during nighttime and prepared in the laboratory for equivalent dose ($D_e$) determination using standard methods for fine grain quartz dating (Fuchs et al., 2005).

The OSL measurements were carried out on a Risø TL/OSL DA-15 readers in the UV band (340 ∆ 80 nm) using blue stimulation (470 ∆ 30 nm). For $D_e$ determination on 12 aliquots per sample a standard single aliquot regenerative (SAR) protocol according to Murray and Wintle (2000) was applied using the first 0.2 s after subtracting the background from the last 8 s of the shine-down curves. The protocol parameters were previously deduced from test measurements. The purity of the quartz extracts was tested by infrared light (IR) stimulation. The mean $D_e$ and the standard error for each sample were used for age calculation. For dose rate calculation the U, Th and K concentration were measured using ICP-MS (K) and thick source alpha-counting (e.g. Zöller and Pernicka, 1989). The $\alpha$-effectiveness (a-value) was measured following the procedure described in Mauz et al. (2006) and Lai et al. (2008). The cosmic dose rate was calculated according to Prescott and Hutton (1994). A water content of 20 ± 5 %
was used. Further methodological details on the luminescence dating are given in the Supplement.

3 Results and discussion

3.1 n-Alkane patterns and $^{14}$C ages of the n-alkane fractions

The $n$-alkane patterns of the four $^{14}$C-dated $n$-alkane fractions reveal three noteworthy features (Fig. 1). First, the long-chain $n$-alkanes ($> n$-$C_{25}$) are characterised by high odd-over-even predominances (OEP), i.e. particularly the $n$-alkanes $n$-$C_{27}$, $n$-$C_{29}$, $n$-$C_{31}$ and $n$-$C_{33}$ are by far more abundant than the $n$-alkanes $n$-$C_{28}$, $n$-$C_{30}$ and $n$-$C_{32}$. Such patterns are typical for plant leaf wax derived $n$-alkanes (Eglington and Hamilton, 1967; Kolattukudy, 1976) and the dominance of $n$-$C_{31}$ furthermore indicates an prevailing origin from grasses (M. Zech et al., 2012a). Second, also $n$-$C_{18}$ and $n$-$C_{20}$ occur in relatively high amounts; in sample 15 additionally $n$-$C_{21}$ and $n$-$C_{22}$ (Fig. 1). While these $n$-alkanes typically do not occur in higher plant leaf waxes or at least in very low concentrations, Wiesenberg et al. (2009) showed that charring of grass biomass at 400 to 500 °C produces exactly such $n$-alkane patterns. We hence conclude that apart from plant leaf waxes ($> n$-$C_{25}$), also charred biomass contributed significantly to the investigated $n$-alkane fractions with $n$-$C_{18}$ and $n$-$C_{20}$ serving as respective molecular markers. Third, although to a different degree, the baselines from $\sim 15$ to 23 min in the chromatograms are elevated. Such humps are called unresolved complex mixture (UCM) and are often observed in GC-chromatograms of petroleum (Frysinger et al., 2003). In soil and paleosols where a petroleum contamination can be excluded, increased UCM humps may indicate soil OM degradation (M. Zech et al., 2012a) and/or biomass burning. Indeed, Eckmeier and Wiesenberg (2009) reported that UCM humps are present in Mesolithic and Neolithic pit fillings containing charred organic matter or black carbon and UCM humps are also formed in the charring experiments of Wiesen-berg et al. (2009).
The radiocarbon dating of the $n$-alkane fractions of the four investigated samples yielded background and $\delta^{13}C$-corrected $^{14}C$ ages ranging from $20.3 \pm 0.7$ kyr calBP for the uppermost sample 15 to $29.8 \pm 1.4$ kyr calBP for the lowermost sample 23 (Table 1 and Fig. 1). A striking chronostratigraphic inconsistency characterises the second uppermost sample 19 being dated to $29.1 \pm 1.4$ kyr calBP and the subjacent sample 21 being dated to $22.1 \pm 0.8$ kyr calBP (Fig. 1). In this context, it is noteworthy, that below 7.8 m depth, the loess-paleosol sequence of Gleina is partly affected by deposition of reworked, older soil material. On the one hand, this could result in “too old” radiocarbon ages for the dated $n$-alkane fractions provided that the reworked soil material contained significant amounts of $n$-alkanes. Yet, M. Zech et al. (2012b) found that particularly the Eemian soil horizons of both the Gleina and the Nussloch (W-Germany) loess-paleosols sequences are characterised by extremely low $n$-alkane concentrations. On the other hand, deposition of reworked sediments may have implications for the interpretation of luminescence data when reconstructing the sedimentation history of loess-paleosol sequences due to insufficient signal resetting, which may cause discrepancies between the $^{14}C$- and OSL-derived ages.

### 3.2 OSL ages and establishment of a chronostratigraphy for the Gleina LPS

OSL fine grain quartz ages and their $D_e$ values are shown in Table 3 and Fig. 1. Age results are given as mean and 2-$\sigma$ error. All age values consistently increase with profile depth and within errors no age inversion occurred. The quartz luminescence signals were almost bright, fast decaying and highly reproducible during determined preheat and dose recovery tests. For all samples no significant feldspar contamination (IRSL/OSL < 1 %) was detected. Consequently, the derived $D_e$ distributions show a narrow scatter ($c_\nu$ ca. 5 %). The dose rate (Supplement) varies between $3.1 \pm 0.2$ Gy kyr$^{-1}$ (BT836) and $3.5 \pm 0.2$ Gy kyr$^{-1}$ (BT842) showing values typical for loess (e.g. Kreutzer et al., 2012).
For the uppermost part of the profile (ca. 3 m up to ca. 8 m) ages between 24.5 ± 2.7 kyr (BT835) and 26.6 ± 2.9 kyr (BT839) indicating a rapid aeolian deposition during the presumed Last Glacial Maximum (MIS2, Lisiecki and Raymo, 2005). These results are in accordance with previous findings from the Saxonian Loess Region ( Kreutzer et al., 2012; Meszner et al., 2011, 2012). The onset of loess sedimentation at ca. 10 m was attributed to the MIS3 (BT842: 45.6 ± 5.3 kyr, BT840: 39.0 ± 4.4 kyr). Below a hiatus at ca. 10.2 m the sediment was dated as early Weichselian (72.8 ± 8.1 kyr BT844, MIS5a to MIS4). This hiatus has been observed for all investigated loess profiles in the Saxonian Loess Region so far. In contrast to other investigated loess profiles in Saxony (Ostrau, Seilitz: Kreutzer et al., 2012; Meszner et al., 2012), where the onset of loess sedimentation has been dated at ca. 30 kyr (transition MIS3 to MIS2), for the Gleina loess the OSL ages reveal an onset of loess deposition in the early MIS3. These findings may indicate locally limited loess preservation due to a special morphographic position. However, an age overestimation due to an incomplete signal resetting during secondary translocation processes cannot be fully excluded, since it is not possible to detect any incomplete signal resetting using fine grain quartz dating. Therefore, finally the reasons for the observed discrepancy remain unknown. Further details on the luminescence dating results are provided in the Supplement.

3.3 Comparison – towards a quantification of post-sedimentary \( n \)-alkane contamination

The comparison of the \(^{14} \text{C} \) ages with the OSL ages (Fig. 1) shows that within the errors the \( n \)-alkane fraction of sample 19 reveals the same age (29.2 ± 1.4 kyr calBP) as the two surrounding OSL samples BT838 and BT839 (mean age 27.3 ± 3.0 kyr). By contrast, the three other \( n \)-alkane samples are clearly younger (20.3 ± 0.7 kyr calBP, 22.1 ± 0.7 kyr calBP and 29.8 ± 1.4 kyr calBP) than the corresponding OSL ages (BT837: 26.5 ± 3.1 kyr, interpolated: 32.0 ± 3.5 kyr and BT842: 45.6 ± 5.3 kyr, respectively). Assuming that all OSL ages reflect sedimentation ages and are not affected significantly by insufficient bleaching (see discussion above), this finding suggests
that one \( n \)-alkane sample (sample 19) has a syn-sedimentary age, whereas a post-sedimentary \( n \)-alkane contamination presumably by roots made the three other \( n \)-alkane samples younger.

In order to estimate the post-sedimentary \( n \)-alkane contamination more quantitatively, we calculated the theoretical syn-sedimentary pMC values according to Eq. (1) using the OSL ages as sedimentation ages (Table 2). Furthermore, we suspected on the one hand a current (\( \sim 100 \) pMC) or modern (last decades) \( n \)-alkane contamination by roots. Given that Levin et al. (1985) observed atmospheric pMC values of more than 120 from 1962 to 1985 (up to almost 200 pMC in the year 1963 due to nuclear weapon tests) in the Northern Hemisphere, we used a pMC value of 120 as representing modern contamination. On the other hand, we suspected a Holocene root-contamination and calculated according to Eq. (1) the theoretical pMC values for three Holocene time points (3, 6 and 9 kyr, see Table 2). This is based on the finding of Gocke et al. (2010) and Pustovoytov and Terhorst (2004) that rhizoliths in last-glacial loess are of Holocene age. Using the measured and the calculated syn-sedimentary and post-sedimentary pMC values the percentages of post-sedimentary \( n \)-alkane contamination for the different contamination scenarios were then estimated by applying a \( ^{14} \)C mass balance calculation (Eq. 2). Accordingly, a current and modern root-contamination would account for up to 7% in the samples 15 and 21 and for up to 4% in the lowermost sample 23. When assuming a 3 kyr old contamination the amount of post-sedimentary \( n \)-alkanes in the samples 15, 21 and 23 increases to 9%, 10% and 5%, respectively. In the most progressive assumption, Early and Middle Holocene roots would have accounted for up to 19%, 20% and 11% (sample 15, 21 and 23, respectively) of the post-sedimentary \( n \)-alkane contamination.

Given that especially the luminescence data have relatively large errors (\( \geq 3 \) kyr, see Table 2), we performed the \( ^{14} \)C mass balance calculation also for the extreme values, which were calculated by subtracting and adding the 2-sigma uncertainty of the mean OSL ages. According to the thus determined error ranges (Table 2), the calculated percentages of root-contamination are rather robust for the current, modern and 3 kyr
contamination scenarios. By contrast, the error ranges are considerably higher for the 6 kyr and 9 kyr contamination scenarios (up to 16–23 %, sample 21, 9 kyr scenario, Table 2). An extension of the above presented approach using a $^{14}$C mass calculation based on more data is therefore inevitably needed to confirm the extent of contamination as derived from our first results.

4 Conclusions

In this study, the $n$-alkane fraction of lipid extracts from a loess-paleosol sequence (LPS Gleina) was dated for the first time. The calibrated $^{14}$C ages of the lipid biomarkers and the comparison with sedimentation ages as assessed by OSL dating suggest that three of four samples experienced a post-sedimentary $n$-alkane contamination presumably by roots. Using a $^{14}$C mass balance calculation the post-sedimentary contamination can be estimated. Accordingly, the extent of contamination strongly depends on the contamination scenario, i.e. it ranges from 0 % to 7 % for current and modern contamination, whereas it ranges from 0 % to 20 % for 9 kyr old contamination. There is no evidence for an overall overprinting of sedimentary $n$-alkane patterns and we hence confirm the value of $n$-alkanes as plant-derived biomarkers in loess. Nevertheless, our finding also highlights that more radiocarbon dating is needed for rhizoliths in loess (time of root-contamination) in order to assess the question “syn-sedimentary or post-sedimentary?” more quantitatively.

Furthermore, we acknowledge and encourage that our first radiocarbon results need further confirmation both from other localities and for different biomarkers, e.g. carboxylic acids or alcohols as further lipid biomarkers. While lipid biomarkers have the great advantage that they can be easily extracted even from very organic-poor loess, we explicitly suggest that $n$-alkanes may not be representative biomarkers for assessing post-sedimentary contamination of bulk OM of loess, because they are not dissolvable in water and occur in comparatively low amounts in roots.
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References


n-Alkane lipid biomarkers in loess

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Table 1. Results of radiocarbon analyses of blanks and \( n \)-alkane samples.

<table>
<thead>
<tr>
<th>Sample code</th>
<th>Sample name and treatment</th>
<th>Material</th>
<th>pMC</th>
<th>( \delta^{13} \text{C} ) (‰)</th>
<th>( ^{14} \text{C} ) age (kyr)</th>
<th>Error (kyr)</th>
<th>Calibrated Age ( ^{1} ) (kyr cal BP)</th>
<th>68 % range (kyr cal BP)</th>
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<tr>
<td>Poz-43739</td>
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<td>21.29–22.84</td>
</tr>
<tr>
<td>Poz-46751</td>
<td>sample 23 + DCM + alk + H(_2)O</td>
<td>alkanes from sediments</td>
<td>4.36(^{2})</td>
<td>−36.0</td>
<td>25.16</td>
<td>1.42</td>
<td>29.83</td>
<td>28.39–31.26</td>
</tr>
</tbody>
</table>

\(^{1}\) Calibration was done with quickcal2007 vers.1.5 (Danzeglocke et al., 2012). \(^{2}\) With correction for background 2.4 ± 0.8 pMC, assuming a 2.4 % contamination of the samples with modern carbon (≈ 100 pMC).
**Table 2.** Measured pMC values of the $n$-alkane fractions, sedimentation OSL ages, estimated uncalibrated syn-sedimentary $^{14}$C ages, post-sedimentary pMC values and calculated percentages of post-sedimentary $n$-alkane contamination by roots.

<table>
<thead>
<tr>
<th>Sample name</th>
<th>pMC$_{measured}$</th>
<th>Sedimentation OSL age (kyr)</th>
<th>Estimated uncalibrated syn-sedimentary $^{14}$C ages$^*$ (kyr)</th>
<th>pMC$_{syn-sedimentary}$</th>
<th>Suspected time of contamination (kyr/krcalBP$^{-1}$)</th>
<th>pMC$_{post-sedimentary}$</th>
<th>Percentage root-contamination</th>
<th>Error range based on OSL errors</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample 15</td>
<td>12.10</td>
<td>26.5 ± 3.1</td>
<td>22.1</td>
<td>6.31</td>
<td>current last decades 100 120 3.0/2.85 6.0/5.2 9.0/8.1</td>
<td>70.1 52.3 36.5</td>
<td>6%</td>
<td>3–8%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>9% 13% 19%</td>
<td>4–12% 6–16% 10–24%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample 19</td>
<td>4.75</td>
<td>27.3 ± 3.0</td>
<td>22.6</td>
<td>6.00</td>
<td>current last decades 100 120 3.0/2.85 6.0/5.2 9.0/8.1</td>
<td>none</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample 21</td>
<td>10.07</td>
<td>32.0 ± 3.5</td>
<td>27.4</td>
<td>3.30</td>
<td>current last decades 100 120 3.0/2.85 6.0/5.2 9.0/8.1</td>
<td>70.1 52.3 36.5</td>
<td>7%</td>
<td>5–8%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>10% 14% 20%</td>
<td>8–12% 11–16% 16–23%</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample 23</td>
<td>4.36</td>
<td>45.6 ± 5.3</td>
<td>42.0</td>
<td>0.54</td>
<td>current last decades 100 120 3.0/2.85 6.0/5.2 9.0/8.1</td>
<td>70.1 52.3 36.5</td>
<td>4%</td>
<td>3–4%</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>5% 7% 11%</td>
<td>5–6% 6–8% 9–11%</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

$^*$ Assuming that OSL age = calibrated $^{14}$C age and using quickcal2007 vers.1.5 (Danzeglocke et al., 2012).
Table 3. OSL quartz fine grain age estimates.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Depth (m)</th>
<th>Fit</th>
<th>$D_e$ (Gy)</th>
<th>Age (kyr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BT835</td>
<td>2.9</td>
<td>EXP + LIN</td>
<td>80.4 ± 0.69</td>
<td>24.5 ± 2.7</td>
</tr>
<tr>
<td>BT836</td>
<td>3.4</td>
<td>EXP + LIN</td>
<td>71.04 ± 1.14</td>
<td>22.9 ± 2.6</td>
</tr>
<tr>
<td>BT837</td>
<td>5.3</td>
<td>EXP + LIN</td>
<td>89.73 ± 1.72</td>
<td>26.5 ± 3.1</td>
</tr>
<tr>
<td>BT838</td>
<td>6.2</td>
<td>EXP + LIN</td>
<td>88.17 ± 0.92</td>
<td>28.0 ± 3.0</td>
</tr>
<tr>
<td>BT839</td>
<td>7.9</td>
<td>EXP + LIN</td>
<td>87.71 ± 0.99</td>
<td>26.6 ± 2.9</td>
</tr>
<tr>
<td>BT840</td>
<td>9.8</td>
<td>EXP + LIN</td>
<td>132.91 ± 1.33</td>
<td>39.0 ± 4.4</td>
</tr>
<tr>
<td>BT842</td>
<td>10</td>
<td>EXP + LIN</td>
<td>161.38 ± 1.25</td>
<td>45.6 ± 5.3</td>
</tr>
<tr>
<td>BT844</td>
<td>10.8</td>
<td>EXP + LIN</td>
<td>235.46 ± 2.41</td>
<td>72.8 ± 8.1</td>
</tr>
</tbody>
</table>

Note: Ages given as mean with 2-sigma uncertainty.
Fig. 1. (A) Stratigraphy of the Gleina loess-paleosol sequence. (B) Age-depth profile of calibrated radiocarbon ages and OSL ages including error bars. (C) n-Alkane concentrations (from M. Zech et al., 2012b) and (D) GC-FID n-alkane chromatograms for the four radiocarbon-dated n-alkane samples.