

Reply to Anonymous Referee #1

We thank Anonymous Reviewer 1 for his positive review and constructive comments, which will help to improve the manuscript. Detailed responses to his comments can be found below:

Referee: If you use a prepGC system to isolate the specific n-alkanes and fatty acids, respectively, you should proof that there is no change in the radiocarbon age occurring during the preparation. Can you evaluate the critical steps in sample preparation (e.g. separation, solvent removal and graphitization) and quantify their effects on the ^{14}C content?

Did you use any standard material for validation? Please, give more detailed information or cite studies, where the method is examined.

Response: We are aware of two studies which assessed potential contamination during the entire lab process in more detail (Shah & Pearson 2007; Ziolkowski & Druffel 2009) (see also p. 16909 l. 10-16.). Both studies indicate that most of the additional carbon is introduced during combustion, with values being very similar to our vacuum line blank. Ziolkowski & Druffel (2009) also found some contamination due to column bleed of the prep-GC. However, in contrary to our study, they did not clean their samples after the PrepGC step in order to remove the column bleed. The potential effect of incomplete solvent removal is discussed later in this response. Note that we used a gas ion source for the ^{14}C measurements, so there is no graphitization step involved. Minor modifications have been made in the manuscript for clarification.

Referee: 16907-Lines 27-28: What happened to the Crvenka samples? Are they homogenized and sieved, too?

Response: The Crvenka samples were only homogenized (they are loess, no particles >2 mm). The information will be added to the manuscript.

Referee: 16908-Lines 1-2: How many cycles did you use for extraction and how long did you extract the samples? Please give more information on the method.

Response: We used three cycles a 5 min per extraction. Note also that we had to extract 3x ~40 g to obtain enough compounds for radiocarbon dating. This information will be added to the manuscript.

Referee: 16908-Line 21: Could you really remove all the solvent? I know it's not easy – there may be some remains which add dead carbon and increase the age of your sample.

Response: Since our samples were very small (10-30 μg), the problem of solvent removal is less of an issue than for bigger samples. Furthermore there are no signs for significant remnants of solvent in our data:

- The $\delta^{13}\text{C}$ values are more or less stable for all samples.
- Larger samples, where the risk of remaining solvent would be arguably higher, don't show systematically older ages.

Referee: 16912-Lines 24-25: Sample Cr 10 shows the opposite trend with younger ages for the long-chain compounds. You should mention this divergent pattern and shortly explain the difference.

Response: We have currently no explanation for the younger $n\text{C}_{33-35}$ in Cr 10, but given the large uncertainties and the limited number of samples, the observed tendency to generally younger ages with decreasing chain-length should be treated with great caution. This will be shortly discussed in the revised version of the manuscript.

Referee: 26904-Lines 18-19: "... in the two investigated systems."

Response: corrected

16906-Line 13: Please homogenize the way of describing the locations in the manuscript: north-western or northwestern. Compare with lines 20 (southwestern) and 21 (northwest).

Response: Cardinal directions are going to be written in one word in the revised version of the manuscript.

Referee: 16906-Lines 21-22: Please homogenize the way of describing the locations (altitude and coordinates) in the manuscript. Compare with line 7 (site Niederbuchsiten) and line 8 (site Steinhof). First mention the altitude of the study site and then the coordinates in the same way (Degree Decimal, Minute Decimal).

Response: corrected

Referee: 16906-Line 24: Check the sentence: "A weakly developed paleosol complex formed during MIS 3 (i.e. between ~ 58 and 28 ka) is found ...".

Response: Changed to: "A weakly developed paleosol complex formed during MIS 3 (i.e. between ~ 58 and 28 ka) and is found in a depth of 4-5.5 m."

16907-Line 24: Please mention some studies where this chronostratigraphic concept is also used for terrestrial records.

Response: For sample Cr20, we had adopted a middle to upper Pleniglacial boundary age of ~28 ka from Kadereith *et al.* (2013). We realize that the most consistent approach is probably to adopt the generally accepted MIS boundaries of Lisiecki and Raymo (2005). We therefore consistently use these boundary ages in the revised manuscript (Cr20 becomes 29 ka). The age is also consistent with the interpolation of two OSL ages below and above Cr20. Note that this change does not affect the conclusions of our study.

16908-Lines 1-2: "... using dichloromethane and methanol (DCM : MeOH; 9 : 1) at ..."

Response: corrected

16910-Lines 10-13: I would shift this part to the discussion, because you're already explaining the results.

Response: Moved to the discussion in the revised version of the manuscript.

16910-Line 22: Please check the sentence. Something is missing there. "... ,respectively, in good agreement with ..."

Response: Changed to: "...respectively and in good agreement with..."

16911-Lines 2-3: Already kind of discussion again.

Response: Removed.

16912-Line 16: Please refer to Table 1.

Response: Done.

16914-Lines 1-4: What's the reference for these explanations? Matsumoto et al., 2007?

Response: The potential input of carbon dead material by human activity was described by Lichtfouse *et al.* (1995). The reference will be added to the revised version of the manuscript.

Fig. 1a: You mentioned that the C horizon in the soil profile Niederbuchsiten is developed below 3 m (16906-Lines 11-12). What does the line in the depth of ~ 2.10 m indicate? Just regarding the figure, I would expect the change from Bt to C horizon at this line.

Response: Changed to 3 m depth.

Fig. 1c: I would plot the ages on the right side of the figure just for a better overview.

Response: Done.

References:

- Kadereit, A., Kind, C.-J., and Wagner, G. A., 2013. The chronological position of the Lohne Soil in the Nussloch loess section – re-evaluation for a European loess-marker horizon, *Quaternary 10 Sci. Rev.*, 59, 67–86.
- Lichtfouse, E., Eglinton, T. I., 1995. ^{13}C and ^{14}C evidence of pollution of a soil by fossil fuel and reconstruction of the composition of the pollutant, *Org. Geochem.*, 23, 969–973.
- Lisiecki, L. E., Raymo, M.E., 2005. A Pliocene-Pleistocene stack of 57 globally distributed benthic $\delta\text{-}^{18}\text{O}$ records. *Paleoceanography* 20. PA1003, DOI: 10.1029/2004PA001071 .
- Shah, S.R., Pearson, A., 2007. Ultra-microscale (5–25 $\mu\text{g C}$) analysis of individual lipids by ^{14}C AMS: Assessment and correction for sample processing blanks. *Radiocarbon* 49, 69–82.
- Ziolkowski, L. A., Druffel, E. R. M., 2009. Quantification of Extraneous Carbon during Compound Specific Radiocarbon Analysis of Black Carbon. *Analytical Chemistry* 81, 10156–10161.