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Climate of the Past Discussions
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Interactive comment on "Reconstruction of multi-millennial summer climate variations in central Japan by integrating tree-ring cellulose oxygen and hydrogen isotope ratios" *by* Takeshi Nakatsuka et al.

Anonymous Referee #2

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SYNOPTIC COMMENT The authors use of d2H and d18O tree-ring series for reconstruction of central Japan hydroclimate. Combining composite data set constitutes a serious technical challenge. The authors selected stem segments mostly of Japanese cypress from living trees, excavated archeological wood, architectural wood and naturally buried logs.

They propose an iterative calculation method to merge 67 series from the various types of wood samples, including the buried archeological and construction wood pieces, and a tentatively quantitative method (factors A and B) to calculate past climate based on

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Discussion paper

d2H and d18O values, using a suite of equations derived from Roden's et al (2000). However, they did not nitrate their samples prior to analysing the d2H values of tree-ring cellulose, so that the exchangeable H is included in their analyses. The simple determination of d2H values on cellulose can generate artefacts. Additionally, the number of trees studied for d2H results are significantly lower than for the d18O determination, and the expressed population signals obtained for the composite d2H series are too low (Fig. 3, b, d). The fact that the d2H and d18O series do not derive from the same populations of trees may generate artefacts. Another point is that the authors did not evaluate the reliability of the isotopic signals for the buried pieces of wood, but alteration of cellulose can occur due to microbial activities during long periods of burial.

Overall, the article is lengthy for what it brings, but generally clearly written. The discussion of the low-frequency trends (long-periodicity variations) is confusing. The authors interpret them unguardedly as age trends, without presenting supporting arguments, and then they bring up the option of these trends possibly relating to changes in growth rates (lines 149-150; 160-165). This potential interpretation implies that environmental conditions may have generated these trends, at least partly. Moreover, the use of ring width for specifically deducing the cause of inverse d2H and d18O trends is risky because in many cases, the isotopic and ring width series do not respond to the same environmental factors.

Another important point is that some of the sampled populations of trees belong to forests exposed to human perturbations; such sites are not suitable for producing isotopic series to be used for climatic reconstruction.

Concluding that (1) d2H analyses would be more reliable if performed on nitrated cellulose, and (2) memory effects occur when performing online pyrolysis, are not new findings and do not bring constructive information in this field of research. Furthermore, as mentioned above, it underlines the fact that 50% of the data used for evaluating paleoclimate is faulty, and weakens the basis for the final reconstruction.

Overall, given the purpose of the article and the unfortunate non-rigorous sample selection and treatment, CP should not accept this article.

SPECIFIC POINTS Line 30 – replace However by In addition.

Line 75 – Please provide a minimum of details about the direct cellulose extraction of 1-mm thick wood samples so the reader does not have to read two other articles to find out. Do you mean that all stem segments were dissected into suite 1 mm-thick samples regardless of ring width and age?

Line 76 – Please explain what are ‘level offsets’.

Line 80 – Simultaneous (?) measurements of d2H and d18O values? How possible with good precision? Using more than one standards is required for a good calibration (two end members with distant isotopic values defining a range broader thant the measured isotopic ranges, and a third standard as an intermediate checkpoint), but the described analytical procedure does not mention this required approach.

Lines 86-87 – Please modify text. . . for reconstructing climate over the past 2,600 yr. . .

Lines 91-96 what are the average, minimum and maximum ring widths of the studied samples; this information will help follow the wood slicing procedure of next section.

Lines 96-97 – Please briefly explain how the new tree-ring d18O time series were used for dating rings. Usage of a statistically strong constructed and multiply verified d18O suite as dating method? How widely is this applicable? For which geographical area was the dating series constructed? What is the operating time resolution on which the comparison is used?

Lines 102-107 – Scientists have recognized for a long time that the production of tree-ring d2H series to be coherent requires nitrating cellulose, so that only the C-bond hydrogen is analysed (Epstein et al., 1976; a reference they use and list). Otherwise, exchangeable H may blur true environmental effects. It seems here that the authors have chosen to save time by analysing simultaneously d2H and d18O in non-nitrated cellulose. No surprise that they conclude they need to improve their analytical procedure (lines 485-487). However, this information has been available to scientists since 1976, with a methods proposed for improving the throughput and reducing the amount of material required back in 2006 and 2009 (Filot et al., 2006; Sauer, 2009; rapid comm. mass spectrom.).

Line 105 – Strict rigor would require indicating the true significant numbers for precisions (reproducibility), i.e., 0.1 or 0.2‰ and 1 or 2‰ for d18O and d2H values, respectively. In the light of the moderate correspondence between nitrated cellulose and cellulose, and of the analytical protocol (only one standard, memory effects not dealt with, peak jumping), it seems hardly conceivable that the d2H precision and accuracy would be of 1‰ it is likely no better than 3‰Ėven with limited effects from OH-exchangeable fraction, the analytical precisions are rarely better than 2‰ (Filot et al., 2006; Sauer, 2009).

Equations 1 and 2 – The ‰ sign should be on the left of the equations, near the delta notation. Otherwise, ‰ x 1000 implies no change in the reported values.

Lines 112-113 - Why all the cellulose samples could not be nitrated? Not enough material extracted from wood? The authors decided to follow an alternative approach, not clearly defined (temperature, time of equilibration), but apparently different than the Filot approach, so that their cellulose and nitrated cellulose only show correlations (r) between 0.74 and 0.77, which is significantly lower than the correspondence obtained using the rigorous protocol of Filot et al (0.94). This compromise is not ideal when producing d2H series destined to climatic reconstruction.

Lines 118-119 – The authors should revisit this statement and write with more nuance, because they sacrifice on the reliability of the d2H series by analysing cellulose instead of nitrated cellulose, or by apparently using an alternative protocol that unfortunately does not perform as well as previous equilibration protocols documented in the literature (Filot et al., 2006; Sauer, 2009).

Lines 199-121 – The memory effects are well known when dealing with online pyrolysis systems, and there are several ways to avoid analytical artefacts due to them. Possibilities include placing a blank (empty capsule) between each samples in the carousels, or analyzing samples in triplicates, or a combination of the two approaches, etc. The appropriate analytical protocol with the instrument should be decided upfront, prior to producing the results. Unfortunately, again, the authors underline the issue after conducting all analyses, but truly this issue could have been easily dealt with prior to producing the isotopic series.

Lines 125-129 – This text and Figure 3 do not inform the reader about the distribution of the woo types. Which isotopic series derive from buried pieces of wood? Departures from real values are reported to occur for altered cellulose/wood (Yapp, 2001; Mancini et al., 2003; Savard et al., 2012).

Mancini, S.A., Ulrich, A.C., Lacrampe-Couloume, G., Sleep, B., Edwards, E.A., Lollar, B.S., 2003. Carbon and hydrogen isotopic fractionation during anaerobic biodegradation of benzene. Applied and Environmental Microbiology 69, 191–198.

Yapp, C., 2001. Rusty relics of earth history: iron(III) oxides, isotopes, and surficial environments. Annual Review of Earth and Planetary Sciences 29, 165–199.

Savard, M. M., Bégin, C., Marion, J., Arseneault, D., and Bégin, Y., 2012. Evaluating the integrity of C and O isotopes in sub-fossil wood from boreal lakes, Palaeogeogr. Palaeoclim. Palaeoecol., 348–349, 21– 31.

Lines 129 – What are the indications that these are age trends? Are trends visible on all individual tree segments prior to combining them? Or are they visible after combining them? In the later case, the authors should consider discussing the possibility of an artefact to the treatment of the data.

Lines 136-139 – The authors clearly state here why their d2H series are not reliable or suitable for climatic reconstruction. Ideally, they should not be used in the following parts of the article (or in the article).

Section 3.2 – The entire discussion about the supposed 'age trends' is misleading. What are the arguments supporting this interpretation? If growth rates correlate with d2H and d18O, inversely and directly, what are the most logical environmental reasons for that? Why do d2H and d18O trends inversely correlate (assuming that the d2H trends reflect something real)? Any possible mechanisms in teleconnection that could explain coeval long-term changes in the three proxies (growth, d2H and d18O)?

Lines 214-215 – It seems that the sites selected for this research are not suited for climatic reconstruction.

Lines 251-252 & Eq 18 – The physiological effects does not always generate a negative relationship between d2H and d18O series. Is it not right?

Lines 258 & 267 – Using constant A and B values implies multiple big assumptions.

Line 296-298 – Another big assumption that this simple combination cancels out the inter-tree average offsets.

Section 3.7 – How can the authors attest that this approach does not generate artefacts at the point of junction between series (e.g., Gagen et al., 2012).

Gagen, M., McCarroll, D., Jalkanen, R., Loader, N. J., Robertson, I., and Young, G. H. F., 2012. A rapid method for the production of robust millennial length stable isotope tree ring series for climate reconstruction, Global Planet. Change, 82–83, 96-103.

Section 3.8 – It seems that there are several short cuts slid in the procedure for attempting to correct for limitations introduced by the analytical approach (lines 367-371). Since there is no true comparison with a fully rigorous approach, the assessment of the procedure is impossible. The comparisons made with reconstructions from other proxies show significant departures and do not allow assessing the proposed procedure (section 3.11).

Line 359 – Note clear... as. . . Please rewrite.

Lines 394-396 – The idea is with paying attention, but unfortunately, the basic sampling and analytical procedures selected for this research are not rigorous enough to allow evaluating the approach in this article.

Table 1 and Figure 2 – It seems that the term 'sample' here refers to stem segments.

Figure 2 – 70% line? Not clear what it is and what it means?

The number of figures is high; perhaps some of them would find a better place in a supplement of information, for examples figures 9, 13, 14.

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