

## Referee #2 comments (14.02.2025)

This manuscript provides a rather interesting fluid inclusion isotope record of two caves in the western Pyrenees, that cover the time interval from Heinrich-1 to today. The isotope record is converted to a Paleo-T record, based on the present-day relation of rainfall oxygen isotope data with temperature. The resulting temperatures show a pattern of lowest T in H-1, higher T's in GI-1, lower T's again in the Younger Dryas (GS-1), and near modern temperatures for the Holocene. This is an interesting high-resolution record, providing valuable information for the reconstruction of paleotemperatures through the deglaciation in Northern Spain

I still have some comments and suggestions, however, that may improve this ms.

Dear editor,

We are grateful for the constructive comments and helpful suggestions by the reviewer. Below is a point-by-point response to the minor comments and questions by **referee #2**:

- FI isotope analysis:

The technique used for the FI isotope analysis is well-established, and an extensive dataset has been produced for this ms. This has been a formidable achievement, and provides valuable insights. The analytical uncertainty as presented, is based on the replicates, and results in a  $\sim 2.7$  permille precision at 1SD level. Routinely analysed replicates of a standard also suggest the uncertainty is around 2.7 permille (I assume that is a 1SD statistic, but please add that to line 210).

A description will be added to line 210 implying that this is the standard deviation of the results.

Line 210: The average long-term precision ( $1\sigma$  uncertainty) of replicate measurements of an in-house calcite standard is  $\pm 2.7$  ‰ for  $\delta^2\text{H}_{\text{FI}}$  for water amounts between 0.1 and 1  $\mu\text{L}$ .

I believe it would improve the ms to further report the  $\text{d}18\text{O}_{\text{FI}}$  data that were presumably collected alongside the  $\text{d}2\text{H}_{\text{FI}}$  data. I understand and accept that the technique used does not yield top results for  $\text{d}18\text{O}_{\text{FI}}$  (particularly at lower yields), but since you probably have the data, I believe it is better to show that, than to say that. Further, if part of the  $\text{d}18\text{O}$  data are robust, then  $\text{d}2\text{H}_{\text{FI}}$  vs  $\text{d}18\text{O}_{\text{FI}}$  cross-plots can help check the robustness of the analysis as a whole, and help identify analytical artefacts, like those described by Fernandez et al. (2023). I would not ask to do that in the main text, but availability of such data (and plots?) in the supplementary materials would really bolster the quality of your dataset. My stance here would be that one should not write off the  $\text{d}18\text{O}_{\text{FI}}$  data without trying. Quite a few previous publications show that  $\text{d}18\text{O}_{\text{FI}}$  data often preserve rather well in speleothem fluid inclusions, and yield meaningful information.

In relation to that, I would strongly suggest to add a section on (isotope equilibrium) temperature calculations based on  $\text{d}18\text{O}$  calcite -  $\text{d}18\text{O}_{\text{FI}}$  pairs. This is a paleothermometer, used in several previous studies (e.g. Fernandez et al 2023) and has a different (isotope equilibrium) approach to temperature calculation than the one you use in this ms. Even if the  $\text{d}18\text{O}_{\text{FI}}$  data are not good enough, you can recalculate the  $\text{d}2\text{H}_{\text{FI}}$  data

to  $\delta^{18}\text{O}_{\text{FI}}$  data using the global or local MWL. I expect it may give valuable insights to have two different techniques to calculate paleotemperatures from the same set of data.

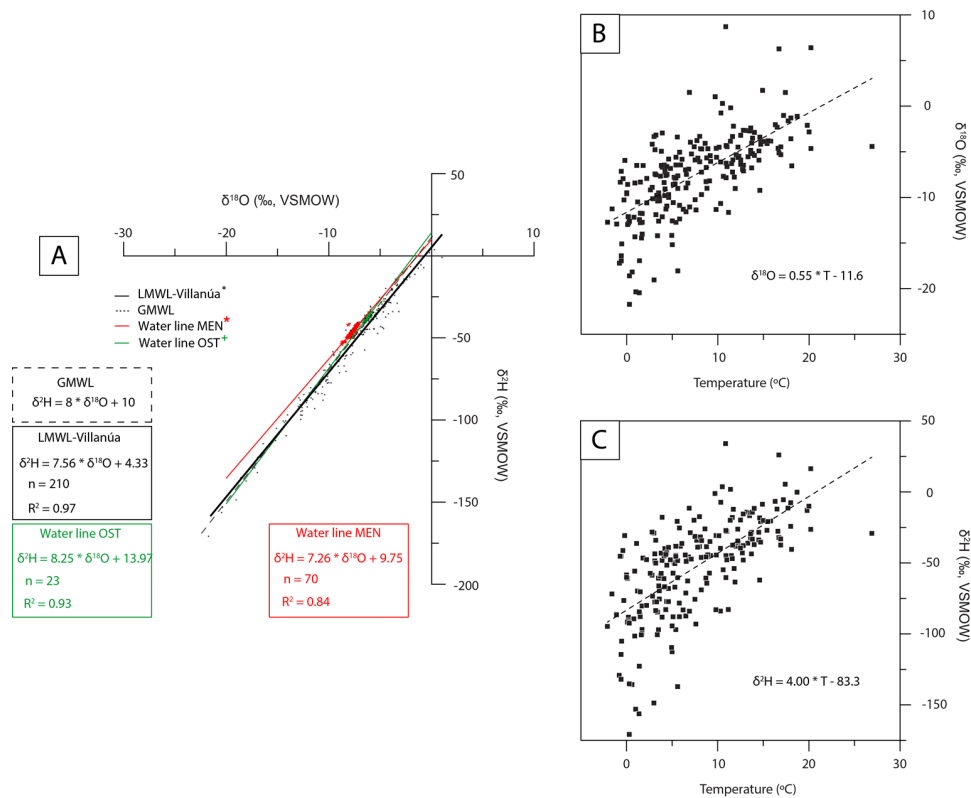
We agree with the reviewer that oxygen values from fluid inclusions may be relevant in many cases for interpretation in terms of paleotemperatures and that if they are available, they should also be included in a manuscript such as the one presented here. Even so, in this manuscript only  $\delta^2\text{H}_{\text{FI}}$  values were used for calculating paleotemperatures for the following reasons: The post-depositional processes can alter the original  $\delta^{18}\text{O}_{\text{FI}}$  in fluid inclusion water and thus limit the use of them for paleotemperature calculations. On the other hand,  $\delta^2\text{H}_{\text{FI}}$  is not affected by isotopic fractionation during calcite precipitation and remains unaltered as there is no hydrogen source once the water is entrapped in the calcite matrix. With this setback, the decision was not to carry out the analysis of the  $\delta^{18}\text{O}_{\text{FI}}$  and follow the usually methodology of other scientific articles about paleotemperatures using speleothem fluid inclusions analyzing only the  $\delta^2\text{H}_{\text{FI}}$  values (Dublyansky and Spötl, 2009; Wilcox et al., 2020 Honiat et al., 2023).

- Paleothermometer calibration:

The description on how you calibrated the paleothermometer (with a modern rainfall record further E and higher up in the Pyrenees) is not so clear to me. You use a dataset of individual rain shower datapoints to make a  $\delta^{18}\text{O}$  vs T relationship, and then calculate that back to  $\delta^2\text{H}$  vs T relationship using “the factor eight” which I presume is the slope of the global MWL (or are you using a local MWL?). Several other corrections are applied (ice volume, elevation), which is correct I believe, but makes the entire calculation process a bit difficult to follow for the non-expert reader. Even when there is more info in Giménez et al (2021), I would ask to clarify this in more detail in the text of this paper, because the calculation is essential to your interpretation of the data.

Another specific question is: You calculate via  $\delta^{18}\text{O}$  data now (Fig 2B), but why did you not use the  $\delta^2\text{H}$  data of the rainwater samples, from Giménez et al 2021, straight away (because that saves you an unnecessary conversion from  $\delta^{18}\text{O}$  to  $\delta^2\text{H}$  values)?

We agree with the reviewer that the derivation of a transfer function to calculate past temperatures in this work is difficult to understand and involves more steps than necessary. To correct this, we will add the  $\delta^2\text{H}$  rainfall data compared to the temperature values for each event in Fig. 2 (Fig. 2C) and also we will modify the explanation of how the paleotemperature calculations were carried out. This will make possible to calculate paleotemperatures directly and it will make it easier for the reader to understand.



- Propagation of uncertainties:

The authors state the uncertainties of the analyses and calibration are fully propagated but I could find no details on how that is done. If I look at the data underlying the d18O vs T relation (Fig 2B), then I see a significant, but seemingly not so strong correlation. The plot suggests a lot of d18O variability is not controlled by T, and I guess that should be very similar if you use the d2H data. You probably need to report the  $r^2$  for the Villanúa d2H vs T plot to quantify the eventual effect on the uncertainties of the calculated T's. What I don't quite understand from the text, in that context, is how you calculated the rather good (0.03 permille) uncertainty of mean annual d18Or and MAAT.

It would be good to provide more error propagation details to underpin the uncertainties on the eventual T-data that you produce, and the choices you made to get there.

We thank reviewer 2 for identifying the lack of clarity and transparency in the calculation of errors associated with the calculation of paleotemperatures in this work. The issue of error propagation in the calculation of paleotemperatures is a difficult one to follow when considering several variables. Not only in the particular case of this manuscript, but most articles on fluid inclusions in stalagmites do not explain in depth what this concept of "propagation" of error refers to. A brief explanation of how error propagation is calculated for different types of operations that are applied in Equation (1) of the manuscript will be incorporated in the text of the Table 3A of the Appendix.

On the other hand, to obtain the value of the transfer function  $\delta^{18}\text{O}/T$  it is necessary to develop a linear regression between both variables (Fig. 2). These parameters, having been obtained from experimental results, must also have an associated uncertainty. This standard error of the regression can be evaluated from the deviations between the experimental points and the predictions of the straight line. In the case of  $\delta^{18}\text{O}/T$  the

standard error of the regression is 0.03. Thanks to the comments of referee 2 we have realized that it is not more direct to use the values of  $\delta^2\text{H}_r$  of above Las Güixas cave directly. In the case of the  $\delta^2\text{H}/T$  relationship the standard error of the regression is 0.31 and the  $r^2 = 4.0$ .

Fernandez et al., 2023: Characterization and Correction of Evaporative Artifacts in Speleothem Fluid Inclusion Isotope Analyses as Applied to a Stalagmite From Borneo (G<sup>3</sup>)

More specific comments and suggestions

286: Is that really significant? I do agree it is at the “right” point in time, but as a result this does not really stand out for me.

We believe that this result does stand out in the OM-FIT record with values very similar to those seen in the GS-1 for both caves. It should be noted that the temperature variations are not as clear as those seen in the Ostolo part of the record, but the change during the 8.2 kyr BP event is still notable, with a decrease in  $\delta^2\text{H}$  values of up to 10 ‰ compared to the average of these values during the Holocene. Furthermore, this change is visible not only in one of the stalagmites in this work, but in two of them (MEN-5 and MEN-2) with high chronological precision.

324-329: I don't quite understand what you have done here. This may need further explanation.

This paragraph refers to a statistical calculation made in the article by Gimenez et al. (2021). We agree that this is not clear in the sentence in question and seems to be a calculation made during the preparation of this manuscript, and that it is also confusing when we explain it. The sentence will be corrected by giving as an example the multiple regression model of the different climatic parameters with the  $\delta^2\text{H}$  results of the rainfall, which is the isotopic indicator used to prepare the temperature transfer function to the detriment of the  $\delta^{18}\text{O}$  values.

Line 324: The observed correlation between  $\delta^{18}\text{O}_r$ ,  $\delta^2\text{H}_r$  and air temperature is verified at biannual scale above Las Güixas cave, with significant correlation between MAAT and the weighted average of  $\delta^{18}\text{O}_r$  and  $\delta^2\text{H}_r$ , based on a multiple regression model using a univariate Spearman's correlation of different climatic parameters (Giménez et al., 2021). For example, the correlation between  $\delta^2\text{H}_r$  and air temperature at the time of precipitation (same data series) results in a  $r_s = 0.69$  ( $p < 0.01$ ) by applying this multiple regression model (Giménez et al., 2021).

351: you mention Younger Dryas here, but that is not used in your figures (you use GS-1 there)

We will use only the term GS-1 along the text and the figures.

453-454 I don't immediately understand what a more smoothed T signal is? Less amplitude?

It is true that this sentence is not entirely clear. It would be clarified that it refers to the amplitude of change in isotopic values and specified which records are being compared.

Line 453: In contrast, the  $\delta^{18}\text{O}_c$  of speleothems from Pyrenean caves is predominantly controlled by temperature (Bartolomé et al., 2015; Cheng et al., 2020; Bernal-Wormull et al., 2021), resulting in a **subtler** temperature signal (**less amplitude of isotopic change**) in the case of MEN  $\delta^{18}\text{O}_c$  compared to the OST  $\delta^{18}\text{O}_c$  record during GS-1, a cold and dry period (Fletcher et al., 2010).

477 the word “record” is once too many in this sentence.

We agree. The word “record” will be erased twice in this same sentence.

482-483: masking? How does that work? Is that centennial-scale d2H variability that is not T-related?

We believe that the use of the word "masking" in this case is inappropriate and we will remove it. In this paragraph our intention is to make it clear that the slight tendency towards lower temperatures during the middle Holocene cannot be discussed with the high variability of the centennial-scale  $\delta^2\text{H}$  record and the associated errors in determining paleotemperatures.

Line 515: That 2.7 degrees C is awfully close to your T uncertainty. I would certainly not dare to say that that is more pronounced than elsewhere (like you do in line 522) based on the uncertainties you are dealing with.

We fully agree with the reviewer on this occasion. The high uncertainty in the OM-FIT does not allow for a comparison of temperature changes during the “8.2 kyr event”. The sentence will be modified to make this point clearer.

Line 513: The 8.2-kyr event overlapped a multi-centennial cool period from  $8.29 \pm 0.04$  kyr BP recorded by MEN  $\delta^{18}\text{O}_c$ , characterized by an abrupt drop in temperature of about  $\sim 3.0^\circ\text{C}$  between  $8.31 \pm 0.06$  and  $8.29 \pm 0.07$  kyr BP in the OM-FIT record (Fig. 6). This cooling within an interglacial coincided with significant vegetation changes in the Iberian Peninsula (Allen et al., 1996; Carrión and Van Geel, 1999; González-Sampériz et al., 2006). This could be important for assessing future climate conditions in this region if changes in large parts of the climate system (climate tipping elements; Armstrong McKay et al., 2022) intensify beyond a warming threshold.

The cooling amplitude during the 8.2 kyr event recorded by OM-FIT appears at first glance more pronounced than in other Northern Hemisphere temperature and precipitation records, with proxy evidence across Europe indicating a cooling by  $\sim 1\text{--}1.7^\circ\text{C}$  during this event (Davis et al., 2003; Morrill et al., 2013; Baldini et al., 2019), **but unfortunately the uncertainties involved in the OM-FIT do not allow a valid comparison to be made on this occasion.**

Table A1: reporting reproducibility with a 1SD metric on only two replicates is statistically a bit on the edge, perhaps.

We agree with the reviewer that reporting the standard deviation of a point with only two measurements is not ideal. Still, it is a common practice in this type of records (see Wilcox et al., 2020; Honiat et al., 2023) where the material is scarce and where the amounts of water to be analyzed are mostly too small to be considered reliable for determining

paleotemperatures. In this case, the values are later compared with carbonate isotopes of both caves, which further supports the variability seen in the  $\delta^2\text{H}$  values of the fluid inclusions throughout the last deglaciation.

Fig 4 panel B: Y axis labels are in the wrong place (swapped).

We will fix the titles of the axis in Fig. 4B.

Dublyansky, Y.V., and Spötl, C., 2009, Hydrogen and oxygen isotopes of water from inclusions in minerals: design of a new crushing system and on-line continuous-flow isotope ratio mass spectrometric analysis: *Rapid Communications in Mass Spectrometry*, v. 23, p. 2605–2613, doi:10.1002/rcm.4155.

Honiat, C., Koltai, G., Dublyansky, Y., Edwards, R.L., Zhang, H., Cheng, H., and Spötl, C., 2023, A paleoprecipitation and paleotemperature reconstruction of the Last Interglacial in the southeastern Alps: *Climate of the Past*, v. 19, p. 1177–1199, doi:10.5194/cp-19-1177-2023.

Wilcox, P.S., Honiat, C., Trüssel, M., Edwards, R.L., and Spötl, C., 2020, Exceptional warmth and climate instability occurred in the European Alps during the Last Interglacial period: *Communications Earth & Environment*, v. 1, p. 57, doi:10.1038/s43247-020-00063-w.