

## Author's Responses

**We thank the reviewers for their time and effort reading the manuscript and offering valuable comments and suggestions. We have modified the manuscript according to their comments, as detailed below. The detailed changes can be found in the track-changes copy of the manuscript. Our direct responses to the reviewers' comments are below. The original reviewers' comments are listed, with our response in blue font.**

## RC1

The authors of this revised manuscript have done a fairly good job responding to my comments on their previous version. They have shortened the background introduction in the abstract, which makes their topic more specific. In Fig.1 and Fig.3, the authors have used high-resolution datasets and applied a moving average, thereby making these figures more readable. Also, they have expanded discussion on the lesser cooling trend at ODP Site 1090 and given a reasonable mechanism. However, I still have some questions, especially regarding the methodology.

Comments:

1. I notice that the authors dissolved total lipid extract in DCM and directly analyzed it using GC-FID. Obviously, there would be organic compounds co-eluting with ketones. The authors then conducted the purification procedures using silica gel column and silver nitrate-impregnated silica gel column. But I really doubt that these procedures can prevent co-elution, as the silica gel column can only separate polar and nonpolar fractions while the silver nitrate has affinity for unsaturated compounds. Please give an explanation of the solvents they used in these columns. Also, using GC peaks to calculate the relative contents of ketones is rather imprecise, as there might be a few organic compounds in the same peak. Eliminating peaks with co-eluted compounds by deviations is confusing, because it is natural that different samples have different ketones and contents of co-eluted compounds, and the deviation alone cannot indicate whether there are co-eluted compounds or not. The authors mentioned that they used HPLC-MS in their experiments, and mass spectrometry is a good way to distinguish ketones, which is more intuitive than GC retention time. But there are no MS results shown in the article.

- We agree that co-elution is a potential concern when quantifying alkenones by GC-FID. However, our methodology was designed so each step— the two column chromatography procedures followed by GC-FID analysis— works iteratively to isolate the alkenones from the total lipid extract. Our purification procedure therefore included silica gel chromatography to isolate the apolar lipid fraction from more polar compounds, and AgNO<sub>3</sub>-impregnated silica gel chromatography to separate compounds according to degree of unsaturation.
- In the polar-apolar fractionation (silica gel column), nonpolar compounds elute first as we use a nonpolar solvent (hexanes), while polar compounds, including alkenones, adsorb to the silica and elute last as solvent polarity increases (here, DCM).
- In the AgNO<sub>3</sub> column, saturated compounds are eluted with DCM, whereas alkenones are retained through reversible pi-complexes formed between silver ions and double

bonds. The unsaturated compounds (including alkenones) are subsequently eluted using ethyl acetate following the protocol of D'Andrea et al. (2007). Although we cannot exclude minor co-elution, these purification steps substantially reduce sample complexity prior to GC analysis. We edited the manuscript to report solvents utilized (Lines 172–175):

- “First, we conducted polar-apolar fractionation via flash column silica gel chromatography using hexanes and DCM as solvents. This was followed by a silver nitrate-impregnated silica gel column chromatographic procedure for unsaturation-saturation separation, which utilized DCM and ethyl acetate as solvents (D'Andrea et al., 2007).”
- We would also like to note that GC-FID is a standard analytical technique for alkenone analyses, due to its cost-effective nature and its robust, reproducible mass-sensitive response. The HPLC-MS analysis was done as an extra step to validate the GC-FID method.
- The HPLC-MS results are not directly reported in the main text, but they are reported in the supplement (Table S4) and in our dataset, which has been published on PANGAEA. To assess whether residual co-elution affected  $U^{K}37$  estimates, we analyzed 16 randomly selected samples using both GC-FID and HPLC-MS. The resulting  $U^{K}37$  values showed close agreement between methods, indicating that the GC-FID peaks predominantly represent the target alkenones and that any remaining co-elution has minimal influence on reconstructed SSTs. We have added text to the manuscript highlighting this validation and directing readers to Table S4 in the results (Lines 296–300):
  - “The HPLC results show similar C37:2 and C37:3 concentrations and ratios compared to our established methodology using GC-FID. The mean difference between SSTs estimated using GC-FID and HPLC is 0.47°C, and the mean absolute difference between these analytical methods' estimated SSTs is 0.85°C. This less than 1°C difference between methods indicates that the GC-FID peaks predominantly represent the target alkenones and that any remaining co-elution has minimal influence on reconstructed SSTs.”

2. The timelines in the two plots of Fig.S1 need to be aligned, or they should share an x-axis. In the upper image, some compound peaks extend beyond the coordinate axes, resulting in a less than ideal overall presentation. However, I understand that scaling down the image would make smaller peaks harder to identify. I hope you can adjust the scale so that as many complete peaks as possible are visible, while still keeping the target peaks clearly distinguishable.

- We changed the x-axis of the bottom plot to match that of the top plot.
- To fully capture the peaks, we would need to more than triple the extent of the y-axis in the top plot, making it nearly impossible to identify the smaller peaks. As these peaks represent compounds not essential to our work, we opted to not adjust the y-axis scale.

3. The color bar of Fig.1 still needs some adjustments. It's too dark now. And why are there orange areas in the landmass?

- To brighten up the figure, we adjusted the colorbar limits to better fit the range of SST data in the figure, so it now utilizes the full range of colors on the scale and is easier to distinguish. The colored areas on the landmass are not an error; this dataset includes

surface temperatures of non-ocean waterbodies like lakes, rivers, etc., which are the orange areas the reviewer noted.

4. Line 68: Change 'ky' to 'ka', as Ma has been used. Check full text.
  - Change was made as suggested. Full text was checked and additional corrections were made in the first paragraph of the results section.
5. Line 128: Change 'alkenones' tendency for incredibly high preservation' to 'the exceptionally high preservation potential of alkenones'.
  - Change was made as suggested.
6. Line 205: Change 'were picked by hand' to 'were selected manually'.
  - Change was made as suggested.
7. Line 327: Delete ', '. Check the same mistakes thoroughly. Such as Line 339.
  - Change made as suggested. Also checked the whole manuscript and corrected any errors (e.g. Line 420).

## RC 2

Review of Hoegler et al. (2026)

I was asked to review the manuscript from Hoegler et al. for the second time. First of all, I would like to apologize for the late review. In general, I think my concerns have been mostly addressed by the authors. This time I only have a few minor, technical comments left.

Line 21:  $p\text{CO}_2$ , p should be italic.

- This has been corrected.

Line 60: Write out ODP here, not in line 79, and refer to your Fig. 1.

- This has been switched and we added the figure reference as suggested.

Line 71: Which reconstruction?

- This is referring to the Martínez-García et al. (2010) reconstruction, which we clarified in the main text.

Line 80: AAC?

- This was a typo and should be ACC. ACC is referring to the Antarctic Circumpolar Current, which we define in line 89.

Line 82: Expansion of zones, migration of fronts

- We rephrased "northward expansion" to "northward migration."

Line 83: The amplitude of SST in Bard and Rickaby (2009) is about 4-6°C. Regarding the latitudinal migration, maybe it is better to include some more recent publications.

- We have updated the manuscript to say "up to 6°C" in alignment with your comment. The previously utilized 8 degree number was referring to data in the 2002 work cited, but we agree that the more recent work by Bard and Rickaby is perhaps the more appropriate data to utilize.
- We added a newer citation and updated the manuscript in regard to latitudinal migration of these fronts: "Biomarker, isotope, and microfossil data suggests these fronts may have migrated anywhere from ~5° equatorward during the Mid-Pleistocene Transition (de Castro et al., 2025) to upwards of 7° latitude during cold stadials in the Pleistocene,

resulting in SST changes of up to 6°C between glacial/interglacial periods (Bard & Rickaby, 2009; Becquey & Gersonde, 2002).”

- Reference: de Castro, M. J., Venancio, I. M., Santos, T. P., Lessa, D. V. O., Ballalai, J. M., Albuquerque, A. L. S., and IODP Expedition 383 Shipboard Scientists: Migration of oceanic fronts in the Pacific Southern Ocean during the Mid-Pleistocene transition, *Marine Micropaleontology*, 200, 102501, <https://doi.org/10.1016/j.marmicro.2025.102501>, 2025.

Line 90, Figure 1: Should Agulhas Leakage eddies be clockwise or counter-clockwise? I think they are counter-clockwise.

- The commenter is correct that they are counterclockwise; we have amended this in the figure.

Figure 2: You can move it upward into the introduction part.

- To accommodate this suggestion, we moved up Figure 1 to just above where it is first mentioned (Line 67) and moved up Figure 2 to the introduction (around Line 80).

Table 1: You are using UK'37 in the table while UK37 in the main text. Please make it consistent, preferably K.

- We went through the main text and the supplement, and we changed all instances of UK'37 and Uk37 to UK'37 and UK37, respectively.

Line 170: In my opinion, it better to describe the method as unsaturation-saturation separation.

- We agree with the commenter that unsaturation-saturation separation is a better way to describe the silver nitrate column procedure, so we have specified this in the manuscript. As the silica gel column procedure does separate polar and apolar compounds, we left the descriptor “polar-apolar fractionation” in the manuscript to describe this specifically.

Line 172: GC-FID instead of \_, check throughout.

- Amended and checked manuscript for similar errors.

Line 175: 2 µL insert?

- Corrected, should be 300 µL.

Line 276, Figure S6: I would plot two age models together in one panel.

- We made this change, thank you!

Line 318-324: Is there a figure showing all these regressions?

- We included a figure with these regressions in our original submission (Nov. 2025 manuscript draft, Fig. 2), but ended up swapping that figure for what is now Fig. 3, using a 400-kyr and 100-kyr moving average to better illustrate the evolving long-term trends in the two records. To avoid over-crowding the figure and because the difference in slopes is minute, we opted to not show the linear regressions and instead only report them in-text.

Line 344, Figure 4: With “analytically meaningful” you mean the absence of C37:4? Please be clear here. Also, please consider adding some denotations in Figure 4 to support “little to no indication of anomalously cold events at ODP Site 1090 uniquely preceding iNHG”. What are the proxies used for U1313 and 846 and what are the references (also for Lines 367-368)?

- We clarified in Line 349 that it is specifically the observed differences in SST that are analytically meaningful.
- To better illustrate the lack of anomalously cold events at Site 1090 prior to iNHG, we made the following changes to the figure: The interval preceding the iNHG following the

MIS M2 cooling event is shaded in all panels, and the mean SST of the plotted interval for all records is denoted by a dotted black line.

- Both the 846 and U1313 SST records are also based on the alkenone paleothermometer; more information about these studies are recorded in Table 1. We added the references to the figure caption and Lines 375-376.

Line 374: Reference.

- Added citations to papers postulating Southern Hemisphere cooling driving Northern Hemisphere glaciation (McKay et al., 2012; McClymont et al., 2023; Woodard et al., 2014)

Lines 393-394: The phrasing of “sensitivity” is a bit problematic. I think here you only mean there is higher power in the precession band, instead of the sensitivity of the proxy (SST) to precession. Please refer to Levy et al. (2019) for the method of sensitivity test, otherwise you can simply rephrase. In addition, I would suggest a wavelet/evolutionary spectral analysis to investigate if there was a change in the periods through time, in which time interval obliquity signal is stronger and if they are synchronous globally. But I will leave this to the authors to decide.

- Reworded: “Both ODP 846 and 1090 showed significant power in the precession bands (23 kyr cycles), but no site showed power in the eccentricity band (100 kyr cycles) that reached the 95% significance threshold.”
- For the purpose of this paper, we do not discuss evolutionary spectral analysis results. However, we did conduct these analyses for utilizing these datasets and are excited to expand on the implications in future publications.

Reference:

Levy, R. H., Meyers, S. R., Naish, T. R., Golledge, N. R., McKay, R. M., Crampton, J. S., DeConto, R. M., De Santis, L., Florindo, F., Gasson, E. G. W., Harwood, D. M., Luyendyk, B. P., Powell, R. D., Clowes, C., and Kulhanek, D. K.: Antarctic ice-sheet sensitivity to obliquity forcing enhanced through ocean connections, *Nature Geosci*, 12, 132–137, <https://doi.org/10.1038/s41561-018-0284-4>, 2019.