Climate of the Past Discussions

Clim. Past Discuss., referee comment RC3
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Comment on cp-2022-19

Anonymous Referee #3

Referee comment on "Evaluation of the distributions of hydroxylated glycerol dibiphytanyl glycerol tetraethers (GDGTs) in Holocene Baltic Sea sediments for reconstruction of sea surface temperature: the effect of changing salinity" by Jaap S. Sinninghe Damsté et al., Clim. Past Discuss., https://doi.org/10.5194/cp-2022-19-RC3, 2022

Sinninghe Damsté et al. describe the distribution of OH-GDGT lipids in Baltic Sea surface sediments, sediment cores and a thaumarchaeal culture grown at different temperatures. The manuscript is thorough and well-written and many significant, open questions on OH-GDGT proxies are addressed by the authors. However, there are some issues with the methodology that could complicate interpretation of the results. First, the authors used base hydrolysis to degrade intact polar OH-GDGTs into core lipids, presumably to facilitate analysis. While this method preserves the hydroxyl groups compared to acid hydrolysis, it does not quantitatively remove the headgroups of thaumarchaeal IPLs (Schouten et al., 2008, AEM) and may selectively degrade IPLs according to their headgroup composition. This will lead to biases in the resulting OH-GDGT quantifications and indices because it is known that OH-GDGTs mostly occur in the form of glycosidic IPLs rather than phosphatidic IPLs in Thaumarchaeota (Schouten et al., 2008, AEM; Pitcher et al. 2011, AEM; Elling et al., 2014, GCA), which are not significantly degraded to core lipids by base hydrolysis. Further, the proportion of core structures is different for different IPL types and markedly different between core OH-GDGTs and OH-GDGTs (predominance of OH-GDGT-0) and IPL GDGTs (predominance of OH-GDGT-2) in thaumarchaeal cultures including the strain most closely related to the organism used in this study, N. limnia (Elling et al., 2014 GCA; Elling et al. 2017 GCA; Pitcher et al., 2011 AEM). It is unclear why the lipids were not analyzed as IPLs, which would have circumvented these issues. If aliquots of the lipid extracts are still available, I would recommend re-analyzing the samples using a method appropriate for detection of IPLs. Second, the authors quantified the fragmentation products of OH-GDGTs at m/z 1300, 1298, 1296 and did not include the 1318,1316, 1314 ions, which could explain some of the variation compared to previous studies and during replicate analysis due to variations in ionization efficiencies due to. This approach was used by some previous studies (e.g., Lü et al., 2015, OG), i.e., in contradiction to what is stated in lines 159-161 and 321-322. Third, the authors do not provide sufficient information on the growth of the cultures such as detailed growth conditions (e.g., volume of medium, volume and type of container and closure, substrate concentration, medium composition) or growth data (e.g. growth curves) to allow assessment of the influence culture

conditions on lipid profiles. For instance, it is probable that growth at 4 °C was much slower than at 22 °C. It is known that growth limitation and growth rate influence GDGT cyclization (Qin et al., 2015, PNAS; Hurley et al., 2016, PNAS) and thus it is probable that these factors influenced the presented OH-GDGT data in addition to the influence of temperature. These issues need to be discussed in the manuscript and their potential impact on the results from the culture sample needs to be quantified or acknowledged.

Line comments:

Line 18: Missing word? OH-GDGT [indices] for SST reconstruction?

Line 145: Please add the original citation of the extraction method, not just a derivative.

Line 225-227: This is confusing. If the concentrations of OH-GDGTs could not be determined, then how was %OH-GDGT calculated?

Figure 1: There are several issues with the legend and caption. The caption states that surface sediments are green squares, but the map and legend show blue circles. The caption further states that sediment cores are blue circles but they are red squares in the map and legend. The red text on brown background in the map are hard to read. Consider coloring land areas grey.

Figures 2-5 are very low resolution and hard to read. Adjust?

Figure 4: The colors for the "Baltic Sea (Kaiser and Arz, 2016)" and "global set" should be changed. They are indistinguishable for red/green colorblind people.

Figure 5b: This panel needs a legend displayed on the figure. The colors of the triangles are indistinguishable for red/green colorblind people.