# Purification of sterols and alkenones for compound specific hydrogen isotopic analysis using HPLC

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Here we present two variations of a method for purifying sterols and alkenones out of total lipid extracts using an HPLC-MS coupled to a fraction collector. The presented methods reduce the amount of work needed and simplify the procedure to obtain fractions pure enough for compound specific irm-GC/MS analysis, compared to traditional wet chemical techniques. This allows a higher throughput of samples so that high-resolution paleoclimatic or paleo-environmental proxy records based on compound-specific isotope measurements can be obtained more efficiently. The presented method was developed for hydrogen isotope analysis, and introduces no isotopic fractionation. The method could also be used in other cases where purification of lipid biomarkers out of total lipid extracts is required.

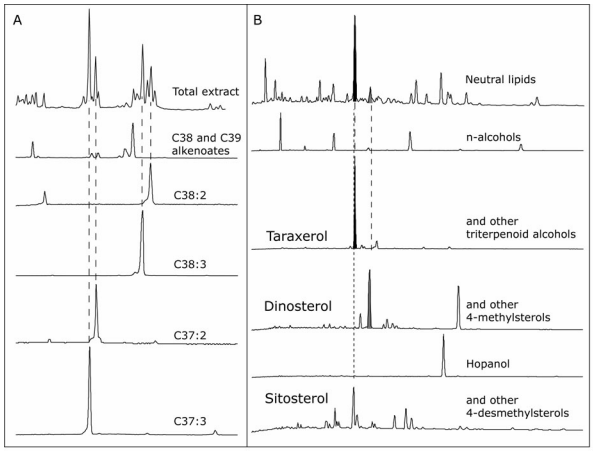


**Figure 1:** Gas Chromatograms of:
A) Total lipid extract of a Chesapeake Bay sediment and fractions as purified by semi-preparative HPLC. Individual alkenones are separated from notoriously co-eluting alkenoates.
B) Neutral fraction of a sediment from Palau (West Pacific) and collected fractions containing various alcohol classes.

# Continental temperatures from the Paleocene-Eocene boundary in the Big Horn Basin, WY from carbonate clumped isotope thermometry

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We used carbonate clumped isotope thermometry to constrain growth temperatures of paleosol carbonates and fossil unionid bivalves collected from the Big Horn Basin (Wyoming) from sections that span the Paleocene-Eocene boundary. Long-term global warming of ~10°C occurred from the Late Paleocene to the Early Eocene. The Paleocene-Eocene Thermal Maximum (PETM) is an extreme thermal event of short duration (< 200 ky) superimposed on the long-term warming trend, and has been identified globally in the ocean sediment record and on the continents in sedimentary basins. The Big Horn Basin is one such basin that has been extensively studied with multiple climatic and biotic proxies in an attempt to characterize the PETM. Therefore, it is an ideal case study for the new paleothermometry technique we use here.

Temperature estimates for the paleosol carbonates capture the pattern of temperature change through time suggested by other paleotemperature proxies, but are consistently higher than previous estimates. Temperature estimates from the fossil mollusk shells, however, are too high to reflect original climatic conditions and do not mimic the stratigraphic change in temperature seen in other proxies. These samples were buried to > 1 km and subsequently exhumed. Our results suggest the paleosol carbonate samples were not dramatically reset by burial metamorphism, whereas the mollusk fossil carbonate was reset by re-crystallization or other processes. We speculate that carbonate that originally forms as calcite is more resistant to resetting during burial metamorphism than carbonate intially formed as metastable aragonite. Although X-ray diffraction analyses detected primary aragonite and no calcite in these fossil mollusks, trace metal analysis and more detailed SEM and/or XRD studies may be required to identify sufficiently unaltered fossil mollusks, if they exist (Came *et al*, in revision, 2007). We conclude that the soil carbonate data constrain continental climate across the Paleocene to Eocene transition. In addition, the contrast between soil carbonates and fossil molllusks provides an important first case study of the relative ability of different forms of carbonate to retain primary temperatures as measured by clumped isotope thermometry.

## Reference

Came, R.E., Eiler, J.M., Veizer, J., Azmy, K., Brand, U., and Weidman, C.R. (2007) *In Revision*.