Methods:

Twenty-eight samples were examined from piston core M98-1P (10°15.99S, 34°19.19), covering the time interval from 0.2 to 23 cal ka, while three samples (at 0.46, 0.53 and 0.60 cal ka) were examined from gravity core M98-2PG (9°58.6'S, 34°13.8'E).

Compound extraction:

Freeze dried sediment samples were soxhlet-extracted with 2:1 methylene chloride/methanol for 24 hours to obtain a total lipid extract (TLE). The TLE was further separated into neutral lipid, fatty acid, and phospholipid fatty acid fractions using aminopropylsilyl bond elute columns (Russell and Werne, 2007). Prior to loading the sample, bond elute columns were pre-cleaned by running 10mL of methanol and 10mL of 1:1 methylene chloride: 2-propanol through the column. Eight mL each of 1:1 methylene chloride: 2-propanol, 4% glacial acetic acid in ethyl ether, and methanol were used to elute the neutral lipid, fatty acid, and phospholipid fatty acid fractions, respectively. Silica-gel column chromatography was used to further separate compounds in the neutral fraction following the procedures outlined by Wakeham and Pease (1992). The \textit{n}-alkanes were present in the first apolar fraction, which was eluted with hexane. This fraction was next passed through an Ag$^+$ impregnated silica pipette column to separate the saturated and unsaturated hydrocarbons.

Sediment samples were soxhlet extracted in groups of five and an additional blank sample was run with every batch. This extraction blank was then worked up in the same manner as the sediment samples to ensure that no contamination was introduced to the samples during any of the steps.

Compound identification:

Molecular identification of compounds (\textit{n}-alkanes) was performed on a Hewlett-Packard 6890 gas chromatograph (GC) coupled to an HP 5973 mass spectrometer (MS). An HP-1 capillary column (25m x 32mm x 0.5μm) was used with He flow rates set at 2mL/min. The GC/MS oven temperature program initiated at 50°C and increased at a rate of 10°C /min to 130°C, and next at a rate of 4°C to 320°C. The final temperature of 320°C was held for 10 minutes. Mass scans were made over the interval from 50 to 650. Compounds were identified by interpretation of characteristic mass spectra fragmentation patterns, gas chromatographic relative retention times, and by comparison with literature.

Compound-specific carbon isotope analysis:

The carbon isotopic composition of \textit{n}-alkanes was determined by gas-chromatography-isotope ratio-mass spectrometry (GC-IRMS). An HP 6890 GC (DB-1 column: 60m, 0.32mm diameter, 0.1μm film thickness) was connected to a Finnigan
MAT Delta+ XL mass spectrometer via a combustion interface. The GC temperature program initiated at 40ºC and increased at a rate of 20ºC/min to 220ºC and next at a rate of 6ºC/min to 315ºC. The final temperature of 315ºC was held for 10 minutes. Compounds separated by the GC column were oxidized at 940ºC and converted to CO2. A standard mixture consisting of four fatty acids with known δ\(^{13}\)C values was measured multiple times daily to ensure accuracy. The standard deviation of all compounds in this standard mixture was less than ± 0.28 ‰. Each n-alkane sample was run in duplicate and the standard deviation of the C\(_{29}\), C\(_{31}\) and C\(_{33}\) n-alkanes is better than ± 0.38‰, ± 0.28 ‰, and ± 0.5‰, respectively. All δ\(^{13}\)C values are reported relative to the Vienna Pee Dee Belemnite (vPDB) standard using standard delta (per mil) notation:

\[
\delta^{13}C = \left(\frac{^{13}C/^{12}C_{\text{sample}}}{^{13}C/^{12}C_{\text{standard}}} - 1\right) \times 1000
\]

REFERENCES:
