

Science Education Collection

Extraction of Biomarkers from Sediments - Accelerated Solvent Extraction

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Overview

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The distribution of a group of organic biomarkers called glycerol-dialkyl glycerol-tetraethers (GDGTs), produced by a suite of archaea and bacteria, were found in modern sediments to change in a predictable manner in response to air or water temperature^{1,2}. Therefore, the distribution of these biomarkers in a sequence of sediments of known age can be used to reconstruct the evolution of air and/or water temperature on decadal to millennial timescales (**Figure 1**). The production of long high-resolution records of past climates, called paleoclimatology, depends on the rapid analysis of hundreds, possibly thousands of samples. Older extraction techniques, such as sonication or Soxhlet, are too slow. However, the newer Accelerated Solvent Extraction technique was designed with efficiency in mind.

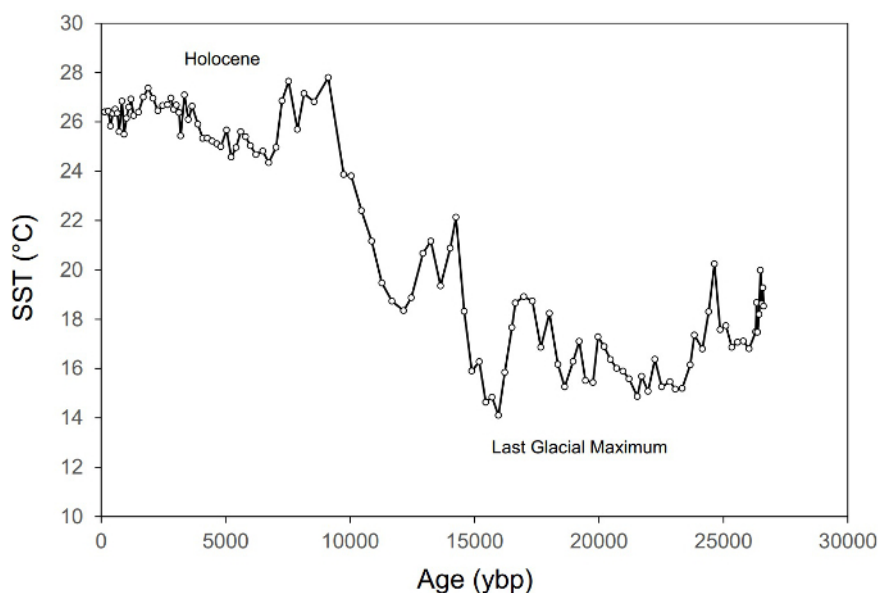


Figure 1. An example of a paleoclimate record showing changes in sea surface temperature (SST) in the eastern Mediterranean Sea during the past ~27,000 years³. This record comprises ~115 samples and is based on the isoprenoidal GDGT-based TEX₈₆ SST proxy.

Principles

Accelerated solvent extraction is a trademarked (Thermo Scientific Dionex) method of extraction that utilizes high temperatures (~100 °C) and pressures (~1,200 psi) to increase the kinetics of the extraction process. The extractor, called an Accelerated Solvent Extractor, or ASE (**Figure 2**), can hold up to 24 individual samples. Once the ASE is loaded and set to run, it is completely automated. The ASE allows electronic control of the entire extraction process: extraction temperature, pressure, solvent volume, solvent mixture, duration, rinse, and repetition are all adjustable from sample to sample. Most organic geochemistry laboratories now use the ASE as the standard method of solvent extraction.



Figure 2. An Accelerated Solvent Extractor (ASE).

Procedure

1. Collection of the Necessary Materials

1. Extract samples. Samples (in this case, sediment) are frozen, freeze-dried, crushed, and homogenized prior to extraction, and extracted in groups to maximize efficiency.
2. Depending on the size of the sample, use collection vials with volumes of 40 or 60 mL. For this experiment, borosilicate glass vials (40 mL) and solvent safe caps are used. Combust vials, borosilicate glass pipettes, and weighing tins at 550 °C for 6 h prior to ensure removal of possible organic contaminants.
3. Dichloromethane (DCM) and methanol are common in most organic geochemistry laboratories. Use them individually, (methanol first, followed by DCM) to rinse lab tools and glassware before use. A mixture of dichloromethane to methanol (MeOH; 9:1) is used in many labs to efficiently extract biomarkers with a wide range of polarities. Solvents should be free of organic contaminants.
4. Obtain an Accelerated Solvent Extractor to use for this experiment.

2. Preparation of Sample Cells

1. Assemble a sample cell for each sample to be extracted, plus one blank.
 1. For each cell, screw an end cap onto one end of the cell body.
 2. Place a combusted glass fiber filter on top of each cell using solvent-rinsed tweezers. Then, gently and slowly press the filter down into the cell using the filter plunger.
 3. Label the cell bodies by number (e.g. 1 - 22) for each sample and write "blank" on the blank cell.
 4. Fill the blank with diatomaceous earth (or sand) and cap with a second end cap. Tighten by hand.

3. Preparation of Samples

1. Place a combusted weighing tin on the lab scale and then tare.
2. Rinse the lab spatula with solvent, then use it to transfer an appropriate mass of sample into the weighing tin, and record the mass.

1. The mass of the sample varies depending on its organic matter content. Relatively organic matter lean material (marine mud) may require several grams, while organic matter rich material (leaf tissue) may require much less.
3. Transfer all of the material in the weighing tin into a prepared ASE cell.
4. Place another glass fiber filter onto the top of the cell, then slowly and gently press down until it reaches the top of the sample using the filter plunger.
5. Add diatomaceous earth (or sand) to the cell until it is almost full. Be careful to remove any debris from the cell body threads.
6. Cap the top of the cell with another end cap.
7. Repeat steps 3.1 - 3.6 for each sample.

4. Preparation of Collection Vials

1. Label each vial with the number of a corresponding cell (e.g. 1 - 22 or blank) and cap with ASE collection vial cap.

5. Extraction

1. Place each sample cell into a numbered slot on the upper ASE tray.
2. Place the corresponding collection vial in the same number slot on the lower ASE tray.
3. Create the extraction method using the keypad on the ASE. Extract at 100 °C and 1,200 psi. Extract each sample 3x with a static hold of 10 min and flush the cell body with 50% of its total volume between static holds.
4. Make sure the solvent bottle contains enough solvent to extract all of the samples.
5. Rinse the ASE 3x before starting the run by pushing the "rinse" button on the ASE control pad.
6. Press start.

Results

At the end of the extraction, there is a total lipid extract (TLE) for each sample. Each vial now contains the extractable organic matter from a sediment, soil, or plant tissue. These TLEs can be analyzed, and their chemical constituents identified and quantified.

Applications and Summary

The TLEs of the extracted samples contain a wide spectrum of different organic compounds, including the GDGTs to be used to reconstruct ancient temperatures. Glycerol-dialkyl glycerol-tetraethers are a large suite of biomarkers that show sensitivity to growth temperatures. There are two groups of GDGTs, branched and isoprenoid, which differ in the character of the branching patterns on the core alkyl groups (**Figure 3**). In the ocean, a cosmopolitan group of archaea, called Thaumarchaeota, produce isoprenoidal GDGTs⁴. Branched GDGTs are produced on land in soils⁵, lakes, and in lake sediments⁶ by as yet unidentified bacteria, likely Acidobacteria⁷. Both archaea and bacteria adjust the number of methyl branches and ring structures in the core alkyl group according to growth temperature, and because GDGTs are stable in sediments for millions of years, long high resolution records of climate change are generated using them.

The TEX₈₆ paleo water temperature proxy is based on the ratio of certain isoprenoidal GDGTs, each containing 86 carbon atoms in its core alkyl group (**Figure 3**):

$$\text{TEX}_{86} = (\text{GDGT-2} + \text{GDGT-3} + \text{GDGT-4}') / (\text{GDGT-1} + \text{GDGT-2} + \text{GDGT-3} + \text{GDGT-4}')$$

Paleo water temperature is then inferred using a calibration, such as the original equation:

$$\text{TEX}_{86} = 0.015T + 0.28 \quad (R^2 = 0.92)$$

Proposed by Schouten *et al.*¹, where T is paleotemperature. Several other regional and local calibrations have been developed to further refine the proxy for use in large lakes or in the tropics, for example.

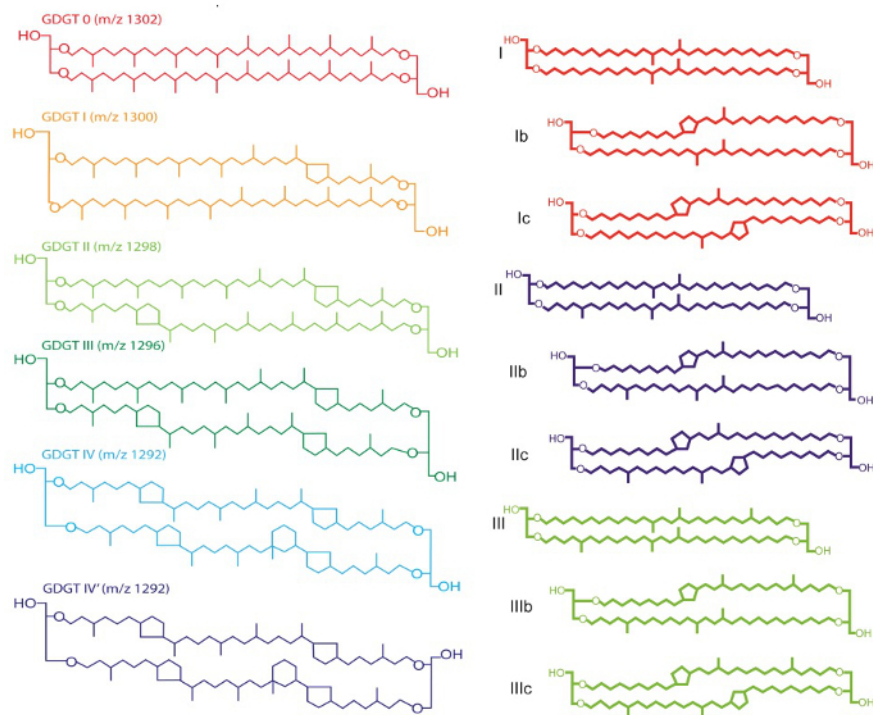


Figure 3. Chemical structures of isoprenoidal and branched GDGTs. [Please click here to view a larger version of this figure.](#)

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