Microwave-Assisted Extraction facilitates seabed sediment research

Source of information on melting Antarctic ice cap

Peter Bijl, PhD and his OceaNice research project are working to improve our ability to predict the effect of Southern Ocean warming on the Antarctic ice cap.

Utrecht University's Marine Palynology and Paleoceanography research group uses the newest extraction equipment to study primeval seabed sediment. The findings could help predict how, and how quickly, climate change can melt the ice caps.

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The Southern Ocean is getting warmer, and therefore quickly melting the Antarctic ice cap. It's unclear whether this process is reversible, and if not, how long it will take for it to melt entirely. Lab research on some of the oldest known seabed sediment may in the next few decades shed light on these ice and ocean dynamics. At Utrecht University, the research is being done by OceaNice, a project that got underway in February 2019 and is led by Peter Bijl, who holds a PhD in the environmental and climatological evolution of the early Paleogene Southern Ocean.

OceaNice

The project is exploring the influence of ocean conditions on the melting process of Antarctica's ice cap during climatically warm periods in the geological past, when carbon dioxide conditions were similar to today's. OceaNice mainly uses sedimentary archives from drilling done by the International Ocean Discovery Program (IODP). Bijl is particularly interested in the organic material in the cores containing molecular fossils. "The Arechaea, a group of single-cell marine organisms, adjust their cell walls to the ambient water temperature. These cell wall molecules are present in the sediment on the ocean floor. Researchers at the Royal Netherlands Institute of Sea Research have taken samples of today's seabeds and determined their molecular composition and the temperature of the ambient seawater. This has created a baseline for extrapolating the temperature of the water from the molecular make-up of any seabed sample. In OceaNice we're applying this method to reconstruct the oceanic conditions close to the Antarctic ice cap during warm periods in the geological past. We opted to study the South Pole, because that's where you'll find about 85 percent of the ice that would cause sea level rise if melted. The reconstructions show how greenhouse effects and the changing ocean circulation over the past 80 million years have affected the global climate and sea levels."

> *'What can past ocean conditions around the Antarctic ice cap tell us about today and tomorrow?'*

Atmospheric boiling point

To analyze bores, Bijl's team uses Microwave-Assisted Extraction (MAE), based on US EPA method 3546. This is a procedure for extracting water-insoluble or slightly water-soluble organic compounds from soils, clays, sediments, sludges, and solid wastes. The solvability of the analytes increases sharply when

The sediment sample is ground into a fine powder, which is put in a glass tube with a mixture of dichloromethane and methanol. The tube is put in a Teflon-coated vessel and sealed before extraction is begun.

24 samples in 40 minutes

The Ethos X microwave extraction system with a Milestone fastEX-24 rotor has a 70+ liter chamber and an advanced, non-contact temperature sensor. Its 1900 W power makes it the most powerful solvent extraction system currently on the market. The fastEX-24 has a 24 capacity carousel. At 24 samples in 40 minutes, its productivity is high. The instrument has a space-saving control panel with preprogrammed extraction methods and a full-color touchscreen display with pictograms. It complies with applicable DIN, NEN and ISO standards.

SAMPLE EXTRACTION

Right:
Analyst Mariska Hoorweg is processing sediment samples from bores drilled in the South Pole seabed.

Below:
The Ethos X's full-color touchscreen display with pictograms.

> *'What ocean temperature did these organic molecular fossils live in?'*

the temperature of the solvent increases. The solvent, contained in closed vessels, is heated by microwave to temperatures exceeding atmospheric boiling point. The pressure in the closed system rises, causing the solvents to remain liquid even above their boiling point. The use of glass tubes prevents cross-contamination, which is crucial considering the tiny amounts of organic material in the samples.

Microwave

MAE is faster and produces more accurate and reproducible results than other methods. It is relatively inexpensive, too, because only small amounts of solvents and energy are needed and little cleaning and waste removal is required. That was more than enough reason for OceaNice to approach lab equipment supplier Salm en Kipp, from whom they purchased an Ethos X microwave extraction system with a Milestone fastEX-24 rotor.

Mariska Hoorweg, analyst at Salm en Kipp, explains the procedure: "Sample preparation consists of grinding the sample into a fine powder with an old-fashioned pestle and mortar. Next, the powder is put in a glass tube and we add the solvent, a mixture of dichloromethane and methanol. The tube is then sealed in a Teflon-coated vessel. The extraction container is heated for 10 to 20 minutes, until it reaches extraction temperature. Heating the solvent creates a balance between the molecules in the gas and liquid phases and pressurizes the closed vessel." Once the solvent has extracted the sediment, it is cooled down to room temperature and pipetted off. This is followed by two rinsing steps. The resulting mixture is concentrated by evaporation and then filtered "through a sodium sulphate column." The result is "a mixture of 'total lipid extracts'."

Back to the future

Based on its chemical properties, this mixture is separated further, Bijl explains. "The resulting polar fractions are separated using HPLC and mass spectrometry consecutively, to determine the peak masses. We're interested in the ratio of those fractions. Measuring those for the various molecules allows us to estimate the ocean temperature that the organic molecular fossils used to live in. This enables us to answer the question: what were the ocean conditions around the Antarctic ice cap like in the geological past and what does this tell us about today and tomorrow? In other words, Back to the Future, but based on solid science."