Amino acid d13C values of Thalassiosira weissflogii of ten different treatments; collections from R/V Meteor M77 in the Peruvian ocean margin from November to December 2008.

Website: https://www.bco-dmo.org/dataset/715936
Data Type: Cruise Results
Version: 1
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Project
» The Use of Nitrogen Isotopes of Amino Acids To Understand Marine Sedimentary 15N Records (Amino Acid Sediment 15N)

Contributors	Affiliation	Role
McCarthy, Matthew D.	University of California-Santa Cruz (UCSC)	Principal Investigator
Ake, Hannah	Woods Hole Oceanographic Institution (WHOI BCO-DMO)	BCO-DMO Data Manager

Abstract
Amino acid d13C values of Thalassiosira weissflogii of ten different treatments; collections from R/V Meteor M77 in the Peruvian ocean margin from November to December 2008.

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Coverage

Temporal Extent: 2008-11 - 2008-12

Dataset Description

Amino acid d13C values of Thalassiosira weissflogii of ten different treatments.

These data were published in:

Larsen, T., Bach, L. T., Salvatteci, R., Wang, Y. V., Andersen, N., Ventura, M., & McCarthy, M. D. (2015). Assessing the potential of amino acid 13C patterns as a carbon source tracer in marine sediments: effects of algal growth conditions and sedimentary diagenesis. Biogeosciences, 12(16), 4979–4992. doi:10.5194/bg-12-4979-2015

Methods & Sampling

Culturing

The marine diatom Thalassiosira weissflogii Grunow (strain CCMP 1010) was cultured in sterile filtered natural

North Sea water (Schleswig-Holstein, Germany) or Baltic Sea water (Schleswig-Holstein, Germany). The medium was enriched with f/4 concentrations of macro- and micronutrients (nitrate, phosphate, silicic acid, trace metal mixture, vitamin mixture (Guillard and Ryther, 1962). All experiments were performed in sterile 2.1 L Schott Duran glass bottles. These bottles were made of borosilicate glass (filters UV radiation <310 nm) except for the quartz glass bottles (pure silica without UV radiation filter) used in the UV experiment. The cultures were either incubated in climate chambers with 400 –700 nm radiation or 10 cm below water level at low tide in Kiel Fjord in May 2011. Water temperature and light irradiance data were obtained from the weather station maintained by the GEOMAR institute in Kiel, Germany. Growth conditions for the various treatments, i.e. salinity, pH, temperature, and irradiance are given in Table 1. pH values (reported on free scale) were measured with separate glass and reference electrodes (Metrohm) and calculated with equation 3 from DOE 2007 chapter 6b (Dickson et al., 2007) as described in (Bach et al., 2012). Cultures were inoculated with densities of 20 cells ml-1. Cell densities and equivalent spherical diameters were determined with a Coulter Counter (Beckman Coulter) at the beginning and the end of the experiment, respectively. When incubations ended, cells were filtered on 47 mm diameter, 5 um mesh size Nucleopore Track-Etch Membrane filters (Whatman) and frozen at -18 deg C immediately after filtration.

Sediment sampling

Sediment samples were retrieved from a 14.97 m core, station M772-003-2, collected November 26, 2008 by the Meteor cruise M77 at 271 m water depth within the main upwelling area off Peru (15 deg 06.21´S, 75 deg 41.28´W). The Peruvian ocean margin is characterized by a high particle flux and a well-defined oxygen minimum zone. At the time of sampling, the O2 concentration at the seafloor was measured to 1.1 uM, the salinity to 34.9 psu and the temperature to 12.2 deg C.

Prior to analysis, sediment samples were pre-treated with an acid-alkali-acid cleaning with HCl and NaOH (Grootes et al., 2004).)

Analyses

Both diatom and sediment samples were freeze dried prior to isotopic analysis. To prepare aliquots for derivatization of amino acids, we used 3-4 mg of diatoms and 100-150 mg of sediments. The samples were transferred to Pyrex culture tubes (13 x 100 mm), flushed with N2 gas, sealed, and hydrolysed in 1 ml 6N HCl at 110 deg C for 20 h. After hydrolysis, lipophilic compounds were removed by vortexing with 2 ml n-hexane/DCM (6:5, v/v) for 30 sec. The aqueous phase was subsequently transferred through disposable glass pipettes lined with glass wool into 4 ml dram vials. Samples were evaporated to dryness under a stream of N2 gas for 30 min at 110 deg C before being stored at 18 deg C until required for analysis. The derivatisation procedure was modified from Corr et al. (2007) as described by Larsen et al. (2013). In short, the dried samples were methylated with acidified methanol and subsequently acetylated with a mixture of acetic anhydride, triethylamine, and acetone, forming N-acetyl methyl ester derivatives. As a precautionary measure to reduce the oxidation of amino acids, we flushed and sealed reaction vials with N2 gas prior to methylation and acetylation. Another modification from Corr et al. (2007) was that icebaths were substituted with solid aluminum blocks at room temperature. We used known d13C values of pure amino acids prepared and analyzed under the same conditions as the samples to calculate correction factors specific to each amino acid to account for carbon addition and fractionation during derivatization. The derivatised AAs were dissolved in 250 ul ethyl acetate and stored at 18 deg C until required for analysis.

Amino acid d13C values were obtained from Leibniz-Laboratory for Radiometric Dating and Stable Isotope Research in Kiel. We injected the AA derivatives into a PTV injector held at 250 deg C for 4 min. before GC separation on an Agilent 6890N GC. Diatom samples were separated on an Rtx-200 column (60m x 0.32mm x 0.25um, Fig. S1) and sediment samples on a Thermo Trace GOLD TG-200MS GC column (60m x 0.32mm x 0.25um). For both GC columns, the oven temperature of the GC was started at 50 deg C and heated at 15 deg C min-1 to 140 deg C, followed by 3 deg C min-1 to 152 deg C and held for 4 min, then 10 deg C min-1 to 245 deg C and held for 10 min, and finally 5 deg C min-1 to 290 deg C and held for 5 min. The GC was interfaced with a MAT 253 isotope ratio mass spectrometer (IRMS) via a GC-III combustion (C) interface (Thermo-Finnigan Corporation). We obtained consistently good chromatography for alanine (Ala), valine (Val), leucine (Leu), isoleucine (Ile), asparagine/aspartate (Asx), threonine (Thr), methionine (Met), glutamine/glutamate (Glx), phenylalanine (Phe), tyrosine (Tyr), lysine (Lys), and arginine (Arg) with the exception that Asx and Thr partially coeluted with the Rtx-200 column. Serine (Ser) and proline (Pro) coeluted on both columns. The average reproducibility for the norleucine internal standard was ± 0.4‰ (n = 3 for each sample), and the reproducibility of amino acid standards ranged from ± 0.1‰ for Phe to ± 0.6‰ for Thr (n = 3).

Amino acid composition of the diatom samples was determined with the derivative samples used for d13CAA analysis. The amino acids were separated on an Rxi-35SIL MS column (30m x 0.32mm x 0.25um) with an Agilent 6890 N GC with a flame ionization detector. With this column we obtained good chromatography for

Ala, Asx, Glx, Gly, Ser, Tyr, Arg, Ile, Leu, Lys, Met, Phe, Thr, and Val. For quantification, we used internal references consisting of pure amino acids (Alfa Aesar, Karlsruhe, Germany). The composition of the amino acids are shown in Table 3 according to the following biosynthetic families: Pyruvate (Ala, Leu, Val), Oxaloacetate (Asx, Ile, Lys, Met, Thr), α-ketoglutarate (Arg, Glx), 3-phosphoglycerate (Gly, Ser), and Shikimate (Phe, Tyr).

Bulk 13C, %C, 15N and %N values of the diatom samples were determined at the UC Davis Stable Isotope Facility using a PDZ Europa ANCA-GSL elemental analyzer interfaced to a PDZ Europa 20-20 isotope ratio mass spectrometer (Sercon Ltd., Cheshire, UK). The dry weight of the samples ranged between 1.5 and 2.5 mg. During analysis, samples were interspersed with several replicates of at least three different laboratory standards. These laboratory standards, which were selected to be compositionally similar to the samples being analyzed, had previously calibrated against NIST Standard Reference Materials (IAEA-N1, IAEA-N2, IAEA-N3, USGS-40, and USGS-41). A sample's preliminary isotope ratio was measured relative to reference gases analyzed with each sample. These preliminary values were finalized by correcting the values for the entire batch based on the known values of the included laboratory standards. The long term standard deviation is 0.2‰ for 13C and 0.3‰ for 15N.

Data Processing Description

Thermo-Finnigan Isodat software and Microsoft Excel 2013.

BCO-DMO Processing:

- changed column names to comply with BCO-DMO standards
- changed the name of the standard deviation columns to include the amino acid they were referencing. Example Thr_st_dev.
- filled all blank cells with nd

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Data Files

File
Thalassiosira.csv (Comma Separated Values (.csv), 3.53 KB) MD5:8387d8f81e39a751dbdbac405a942fe0 Primary data file for dataset ID 715936

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Related Publications

Bach, L. T., Bauke, C., Meier, K. J. S., Riebesell, U., & Schulz, K. G. (2012). Influence of changing carbonate chemistry on morphology and weight of coccoliths formed by Emiliania huxleyi Biogeosciences, 9(8), 3449–3463. doi:10.5194/bg-9-3449-2012
Methods

Corr, L. T., Berstan, R., & Evershed, R. P. (2007). Development ofN-Acetyl Methyl Ester Derivatives for the Determination of δ13C Values of Amino Acids Using Gas Chromatography-Combustion- Isotope Ratio Mass Spectrometry. Analytical Chemistry, 79(23), 9082–9090. doi:10.1021/ac071223b
Methods

Dickson, A.G., Sabine, C.L. and Christian, J.R. (Eds.) 2007. Guide to best practices for ocean CO2 measurements. PICES Special Publication 3, 191 pp. ISBN: 1-897176-07-4. URL: https://www.nodc.noaa.gov/ocads/oceans/Handbook_2007.html https://hdl.handle.net/11329/249
Methods

Grootes, P. M., Nadeau, M.-J., & Rieck, A. (2004). 14C-AMS at the Leibniz-Labor: radiometric dating and isotope research. Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, 223-224, 55–61. doi:10.1016/j.nimb.2004.04.015

Methods

Guillard, R. R. L., & Ryther, J. H. (1962). STUDIES OF MARINE PLANKTONIC DIATOMS: I. CYCLOTELLA NANA HUSTEDT, AND DETONULA CONFERVACEA (CLEVE) GRAN. Canadian Journal of Microbiology, 8(2), 229–239. doi:10.1139/m62-029
Methods

Larsen, T., Bach, L. T., Salvatteci, R., Wang, Y. V., Andersen, N., Ventura, M., & McCarthy, M. D. (2015). Assessing the potential of amino acid 13C patterns as a carbon source tracer in marine sediments: effects of algal growth conditions and sedimentary diagenesis. Biogeosciences, 12(16), 4979–4992. doi:10.5194/bg-12-4979-2015
Results

Larsen, T., Ventura, M., Andersen, N., O'Brien, D. M., Piatkowski, U., & McCarthy, M. D. (2013). Tracing Carbon Sources through Aquatic and Terrestrial Food Webs Using Amino Acid Stable Isotope Fingerprinting. PLoS ONE, 8(9), e73441. doi:10.1371/journal.pone.0073441
Methods

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Parameters

Parameter	Description	Units
Treatment	Treatment type	unitless
Thr	Threonine d13C value	per mil
Thr_st_dev	Standard deviation	per mil
Asx	Asn+Asp d13C value	per mil
Asx_st_dev	Standard deviation	per mil
Lys	Lysine d13C value	per mil
Lys_st_dev	Standard deviation	per mil
Ile	Isoleucine d13C value	per mil
Ile_st_dev	Standard deviation	per mil
Met	Methionine d13C value	per mil
Met_st_dev	Standard deviation	per mil
Ala	Alanine d13C value	per mil
Ala_st_dev	Standard deviation	per mil
Val	Valine d13C value	per mil
Val_st_dev	Standard deviation	per mil
Leu	Leucine d13C value	per mil
Leu_st_dev	Standard deviation	per mil
Arg	Arginine d13C value	per mil
Arg_st_dev	Standard deviation	per mil
Glx	Gln+Glu d13C value	per mil
Glx_st_dev	Standard deviation	per mil
Tyr	Tyrosine d13C value	per mil
Tyr_st_dev	Standard deviation	per mil
Phe	Phenylalanine d13C value	per mil
Phe_st_dev	Standard deviation	per mil

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Instruments

Dataset-specific Instrument Name	PDZ Europa ANCA-GSL elemental analyzer
Generic Instrument Name	Elemental Analyzer
Dataset-specific Description	Interfaced to a PDZ Europa 20-20 isotope ratio mass spectrometer (Sercon Ltd., Cheshire, UK)
Generic Instrument Description	Instruments that quantify carbon, nitrogen and sometimes other elements by combusting the sample at very high temperature and assaying the resulting gaseous oxides. Usually used for samples including organic material.

Dataset-specific Instrument Name	Thermo Trace GOLD GC
Generic Instrument Name	Gas Chromatograph
Dataset-specific Description	Used with MAT 253 isotope ratio mass spectrometer (IRMS) via a GC-III combustion (C) interface (Thermo-Finnigan Corporation)
Generic Instrument Description	Instrument separating gases, volatile substances, or substances dissolved in a volatile solvent by transporting an inert gas through a column packed with a sorbent to a detector for assay. (from SeaDataNet, BODC)

Dataset-specific Instrument Name	PDZ Europa 20-20 isotope ratio mass spectrometer
Generic Instrument Name	Isotope-ratio Mass Spectrometer
Dataset-specific Description	Used with PDZ Europa ANCA-GSL elemental analyzer
Generic Instrument Description	The Isotope-ratio Mass Spectrometer is a particular type of mass spectrometer used to measure the relative abundance of isotopes in a given sample (e.g. VG Prism II Isotope Ratio Mass-Spectrometer).

Dataset-specific Instrument Name	MAT 253 isotope ratio mass spectrometer (IRMS)
Generic Instrument Name	Isotope-ratio Mass Spectrometer
Dataset-specific Description	Used with Thermo Trace GOLD GC
Generic Instrument Description	The Isotope-ratio Mass Spectrometer is a particular type of mass spectrometer used to measure the relative abundance of isotopes in a given sample (e.g. VG Prism II Isotope Ratio Mass-Spectrometer).

Dataset-specific Instrument Name	Light meter
Generic Instrument Name	Light Meter
Dataset-specific Description	Used to measure irradiance
Generic Instrument Description	Light meters are instruments that measure light intensity. Common units of measure for light intensity are umol/m2/s or uE/m2/s (micromoles per meter squared per second or microEinsteins per meter squared per second). (example: LI-COR 250A)

Dataset-specific Instrument Name	pH sensor
Generic Instrument Name	pH Sensor
Dataset-specific Description	Used to measure pH
Generic Instrument Description	An instrument that measures the hydrogen ion activity in solutions. The overall concentration of hydrogen ions is inversely related to its pH. The pH scale ranges from 0 to 14 and indicates whether acidic (more H+) or basic (less H+).

Dataset-specific Instrument Name	Salinity sensor
Generic Instrument Name	Salinity Sensor
Dataset-specific Description	Used to measure salinity
Generic Instrument Description	Category of instrument that simultaneously measures electrical conductivity and temperature in the water column to provide temperature and salinity data.

Dataset-specific Instrument Name	Water Temperature Sensor
Generic Instrument Name	Water Temperature Sensor
Dataset-specific Description	Used to measure temperature
Generic Instrument Description	General term for an instrument that measures the temperature of the water with which it is in contact (thermometer).

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Deployments

M77

Website	https://www.bco-dmo.org/deployment/715334
Platform	R/V Meteor
Report	https://www.ldf.uni-hamburg.de/meteor/wochenberichte/wochenberichte-meteor/m77/m77-2-scr.pdf
Start Date	2008-11-24
End Date	2008-12-21
Description	Main research topic of cruise M77-2 was the investigation of the oxygen minimum zone (OMZ) in the coastal upwelling areas off Peru and off Ecuador.

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Project Information

The Use of Nitrogen Isotopes of Amino Acids To Understand Marine Sedimentary 15N Records (Amino Acid Sediment 15N)

Coverage: California Margin , Santa Barbara Basin , CA current system, Eastern Tropical Pacific

The bioavailability of nutrients plays a crucial role in oceanic biological productivity, the carbon cycle, and climate change. The global ocean inventory of nitrogen (N) is determined by the balance of N-fixation (sources) and denitrification (sinks). In this three-year project, a researcher from the University of California, Santa Cruz, will focus on developing compound-specific N isotope (d15N) analysis of amino acids as a new tool for understanding N source and transformation of organic matter in paleo-reservoirs. The offsets in the isotopic ratios of individual amino acid groups may yield information about trophic transfer, heterotrophic microbial reworking, and autotrophic versus heterotrophic sources. By measuring and comparing the bulk and amino acid d15N in size-fractioned samples from plankton tows, sediments traps, and multi-cores in oxic and suboxic depositional environments, the researcher will: (1) Provide a proxy of the d15N of average exported photoautotrophic organic matter; and (2) Provide a new level of detail into sedimentary organic N degradation and preservation.

Broader impacts:
This project will improve understanding of the fundamental underpinnings and behaviors of d15N amino acid patterns and how they behave in contrasting sedimentary environments, while also developing a potential paleoceanographic proxy. Funding will support a graduate student and undergraduate research at the institution. The researcher will also conduct community outreach in the form of a workshop/tutorial on the proxy development.

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Funding

Funding Source	Award
NSF Division of Ocean Sciences (NSF OCE)	OCE-1131816

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