Concentration and δ15N of amino acids in size-fractionated particles from the eastern tropical North Pacific ODZ in December 2020

Website: https://www.bco-dmo.org/dataset/955207
Data Type: Cruise Results
Version: 1
Version Date: 2025-03-12

Project
» Collaborative research: Using individual amino acids N isotopes in sinking particles and surficial sediments to reconstruct euphotic zone N sources and trophic structure (Amino Acids N Isotopes)

Contributors	Affiliation	Role
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Abstract
This dataset includes the concentration and δ15N of bulk N and amino acids in size-fractionated particles. Particle samples were collected aboard the R/V Sally Ride (cruise SR2011) from December 23 – 30, 2020 with a McLane large volume pumping system (WTS-LV). These data assess the nitrogen sources utilized by different phytoplankton communities inhabiting the two chlorophyll maxima and the transformation of particulate organic matter within the oxygen-deficient zone.

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Coverage

Location: Eastern tropical North Pacific oxygen deficient zone
Spatial Extent: **N**:17.29 **E**:-104.27 **S**:14.02 **W**:-107.78
Temporal Extent: 2020-12-22 - 2020-12-30

Methods & Sampling

Two particle fractions were collected at five stations in the eastern tropical North Pacific region. At each station, seawater was filtered sequentially through a 53-micrometer (µm) Nitex mesh and then a pre-combusted 142-millimeter (mm) diameter glass fiber filter (GF-75: 0.3 µm retention size; or GF/F: 0.7 µm nominal retention size) with the McLane pump in situ. These filters were promptly frozen upon retrieval.

Each 53 µm Nitex mesh was immersed in approximately 100 milliliters (ml) of MilliQ water and subjected to a 5-minute sonication. The solution was filtered onto pre-combusted 47-mm glass fiber filters (GF/F, 0.7 µm pore

size), which retain the > 53 µm particle fraction.

The GF-75 and GF/F filters (0.3 or 0.7 – 53 µm fractions) were subsampled, packed in tin capsules, and analyzed for concentration and δ15N of bulk materials by an elemental analyzer isotope-ratio mass spectrometer at the UC Davis Stable Isotope Facility. Bulk δ15N analysis was not performed for the > 53 µm fraction due to the limited amount of collected material.

The sample pre-treatment procedures for δ15N-amino acids were based on the method detailed in Zhang et al. (2021). The glass fiber filters underwent hydrolysis with 6N HCl for 22 hours at 110 degrees Celsius (°C). Hydrophobic impurities were eliminated from the hydrolysates through liquid-liquid extraction using n-hexane/dichloromethane (6:5, v/v), followed by evaporation to dryness in a vacuum evaporator (RapidVap, Labconco). The samples were subsequently redissolved in 0.05N HCl and further purified via cation-exchange resin, following procedures adapted from Takano et al. (2010) to remove metal ions and salts. The purified amino acids in the samples were completely dried under vacuum.

Amino acids in the samples were separated and collected as individual fractions using an ICS-5000+ Ion-exchange chromatography system with the instrumental method adapted from Zhang et al. (2021). For each sample, Phe, Glu, and their corresponding IC procedural blanks were collected from 1 to 3 replicate injections. The IC-collected fractions were sequentially converted to NO2- and N2O through a two-step process involving hypochlorite oxidation and azide reduction, as described in McIlvin and Altabet (2005); Zhang et al. (2007); Zhang and Altabet (2008); and Zhang et al. (2021). δ15N-N2O was determined with a GV IsoPrime IRMS.

Data Processing Description

Concentrations of Phe and Glu in the particle samples were determined by calibrating them against the chromatographic peak areas of a set of amino acid standard mixtures at varying concentration levels between 1 and 5 millimolar (mM).

The δ15N of Phe and Glu were calibrated by three replicate injections of multiple amino acid isotopic standard mixtures with distinct δ15N values were carried out as outlined in Zhang et al. (2021). Trophic position (TP) was calculated from δ15N of Phe and Glu with an empirical formula proposed by Chikaraishi et al. (2009).

Standard deviations were reported for samples with δ15N measurements obtained from replicate IC injections. The error propagated from uncertainties of the instrument, calibration, and correction was calculated for samples collected from a single injection.

"bdl" in data file = "below detection limit"

BCO-DMO Curation Notes

- Imported original file "BCODMO data_final.xlsx" into the BCO-DMO system.
- Marked "/" as a missing data identifier (missing data are empty/blank in the final CSV file).
- Replaced "#" with "bdl" ("below detection limit").
- Created date-time field in ISO 8601 format, and removed the separate "Date" and "Time" columns.
- Renamed fields to comply with BCO-DMO naming conventions (replaced spaces with underscores).
- Saved the final file as "955207_v1_sr2011_conc_d15n_amino_acids_particles.csv".

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Data Files

File	
955207_v1_sr2011_conc_d15n_amino_acids_particles.csv	(Comma Separated Values (.csv), 3.48 KB) MD5:355061ca24ffa3cf3b3c61f913194c91
Primary data file for dataset ID 955207, version 1	

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Related Publications

Chikaraishi, Y., Ogawa, N. O., Kashiyama, Y., Takano, Y., Suga, H., Tomitani, A., Miyashita, H., Kitazato, H., & Ohkouchi, N. (2009). Determination of aquatic food-web structure based on compound-specific nitrogen isotopic composition of amino acids. Limnology and Oceanography: Methods, 7(11), 740–750. Portico. https://doi.org/10.4319/lom.2009.7.740
Methods

Lee, C. W. M., Altabet, M., Mnich, A., & Zhang, L. (2025). Using δ15N of Amino Acids and Nitrate to Investigate Particle Production and Transformation in the Ocean: A Case Study From the Eastern Tropical North Pacific Oxygen Deficient Zone. Global Biogeochemical Cycles, 39(1). Portico. https://doi.org/10.1029/2024gb008280 https://doi.org/10.1029/2024GB008280
Results

McIlvin, M. R., & Altabet, M. A. (2005). Chemical Conversion of Nitrate and Nitrite to Nitrous Oxide for Nitrogen and Oxygen Isotopic Analysis in Freshwater and Seawater. Analytical Chemistry, 77(17), 5589–5595. doi:10.1021/ac050528s
Methods

Takano, Y., Kashiyama, Y., Ogawa, N. O., Chikaraishi, Y., & Ohkouchi, N. (2010). Isolation and desalting with cation-exchange chromatography for compound-specific nitrogen isotope analysis of amino acids: application to biogeochemical samples. Rapid Communications in Mass Spectrometry, 24(16), 2317–2323. Portico. https://doi.org/10.1002/rcm.4651
Methods

Zhang, L., & Altabet, M. A. (2008). Amino-group-specific natural abundance nitrogen isotope ratio analysis in amino acids. Rapid Communications in Mass Spectrometry, 22(4), 559–566. https://doi.org/10.1002/rcm.3393
Methods

Zhang, L., Altabet, M. A., Wu, T., & Hadas, O. (2007). Sensitive Measurement of NH4+15N/14N (δ15NH4+) at Natural Abundance Levels in Fresh and Saltwaters. Analytical Chemistry, 79(14), 5297–5303. doi:10.1021/ac070106d
Methods

Zhang, L., Lee, W. (Charlotte), Kreider-Mueller, A., Kuhnel, E., Baca, J., Ji, C., & Altabet, M. (2021). High-precision measurement of phenylalanine and glutamic acid δ15N by coupling ion-exchange chromatography and purge-and-trap continuous-flow isotope ratio mass spectrometry. Rapid Communications in Mass Spectrometry, 35(13). Portico. https://doi.org/10.1002/rcm.9085
Methods

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Parameters

Parameter	Description	Units
ISO_DateTime_UTC	Sampling date and time (UTC) in ISO 8601 format	unitless
Lat	Latitude in decimal degrees North	decimal degrees
Lon	Longitude in decimal degrees West	decimal degrees
Station	Sampling station number	unitless
Size_fraction	Size fraction of particles in micrometers	micrometers
Depth	Depth of sample collection	meters (m)
Bulk_N	Concentration of bulk N	nanomoles per liter (nmol/L)
Phe	Concentration of phenylalanine	nanomoles per liter (nmol/L)
Glu	Concentration of glutamic acid	nanomoles per liter (nmol/L)
bulk_d15N	d15N of bulk N	per mil relative to air
d15N_Phe	d15N of phenylalanine	per mil relative to air
d15N_Phe_sd	Standard deviation of d15N of phenylalanine	per mil relative to air
d15N_Glu	d15N of glutamic acid	per mil relative to air
d15N_Glu_sd	Standard deviation of d15N of glutamic acid	per mil relative to air
TP	Trophic position	unitless
TP_sd	Standard deviation of trophic position	unitless

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Instruments

Dataset-specific Instrument Name	GV IsoPrime IRMS
Generic Instrument Name	GV Instruments IsoPrime Isotope Ratio Mass Spectrometer
Dataset-specific Description	GV IsoPrime IRMS: Isotopic analysis was conducted using a GV IsoPrime IRMS equipped with a custom purge-trap sample preparation system, and a CTC PAL autosampler.
Generic Instrument Description	The GV Instruments IsoPrime Isotope Ratio Mass Spectrometer is a laboratory benchtop isotope ratio mass spectrometer (IRMS), that operates in dual inlet or continuous flow modes. It forms part of the IsoPrime system which has a range of sample preparation and purification modules that can be coupled with the IsoPrime IRMS. The modules can analyse a range of samples (solids, liquids or gases) and each prepares the sample to be introduced into the IRMS as purified gases. The resultant prepared gases such as H2, CO2, CO, N2, SO2 or N2O are then ionised and analysed for their isotopic content by the IRMS. The instrument was originally manufactured GV instruments as part of the IsoPrime series, but this part of the business was sold to Isoprime Ltd (a group member of Elementar Analysensysteme GmbH) and this instrument has been superseded by more recent models.

Dataset-specific Instrument Name	McLane Large Volume Pumping System WTS-LV
Generic Instrument Name	McLane Large Volume Pumping System WTS-LV
Dataset-specific Description	McLane Large Volume Pumping System WTS-LV: Size-fractionated particle samples were collected by filtering seawater in-situ with the McLane pump.
Generic Instrument Description	The WTS-LV is a Water Transfer System (WTS) Large Volume (LV) pumping instrument designed and manufactured by McLane Research Labs (Falmouth, MA, USA). It is a large-volume, single-event sampler that collects suspended and dissolved particulate samples in situ. Ambient water is drawn through a modular filter holder onto a 142-millimeter (mm) membrane without passing through the pump. The standard two-tier filter holder provides prefiltering and size fractioning. Collection targets include chlorophyll maximum, particulate trace metals, and phytoplankton. It features different flow rates and filter porosity to support a range of specimen collection. Sampling can be programmed to start at a scheduled time or begin with a countdown delay. It also features a dynamic pump speed algorithm that adjusts flow to protect the sample as material accumulates on the filter. Several pump options range from 0.5 to 30 liters per minute, with a max volume of 2,500 to 36,000 liters depending on the pump and battery pack used. The standard model is depth rated to 5,500 meters, with a deeper 7,000-meter option available. The operating temperature is -4 to 35 degrees Celsius. The WTS-LV is available in four different configurations: Standard, Upright, Bore Hole, and Dual Filter Sampler. The high-capacity upright WTS-LV model provides three times the battery life of the standard model. The Bore-Hole WTS-LV is designed to fit through a narrow opening such as a 30-centimeter borehole. The dual filter WTS-LV features two vertical intake 142 mm filter holders to allow simultaneous filtering using two different porosities.

Dataset-specific Instrument Name	ICS-5000+ Ion-change chromatography system
Generic Instrument Name	Thermo Fisher Scientific Dionex ICS-5000 ion chromatography (IC) system
Dataset-specific Description	ICS-5000+ Ion-change chromatography system: Amino acids in particle samples were separated and collected as individual fractions by the ICS-5000 system.
Generic Instrument Description	The Thermo Fisher Scientific Dionex ICS-5000 ion chromatography (IC) system is an ion chromatography system that offers a full range of reagent-free components. This instrument can be configured to use single or dual pumps. The single-channel Dionex ICS-5000 can be configured to run capillary, microbore or standard bore IC applications. A dual Dionex ICS-5000 system can be configured with any combination of these applications. This system uses an eluent generator (EG) to generate high purity acid or base eluents from deionized water, in the amount and concentration needed for sample analysis, configurable for single or dual channel operation. Eluent regeneration may also be used without an EG - eluent regeneration uses the suppressor to reconstitute the starting eluent, allowing use of a single 4-liter bottle of eluent for up to four weeks. An eluent organizer (EO) module is used to contain eluent spills and leaks. The ICS-5000 detector/chromatography module (DC) can accommodate components for two channels, plumbed either serially or in parallel, in a temperature-controlled environment. Available DC components include conductivity detectors, electrochemical detectors, injection valves, switching valves, guard and separator columns, suppressors, and Dionex IC cubes or ICS-5000 Automation Manager. Detectors outside of the DC include a Dionex ICS Series Photodiode Array Detector (PDA); Dionex ICS Series Variable Wavelength Detector (VWD); MSQ Plus Mass Spectrometer.

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Deployments

SR2011

Website	https://www.bco-dmo.org/deployment/955215
Platform	R/V Sally Ride
Start Date	2020-12-16
End Date	2021-01-06
Description	More information is available from R2R: https://www.rvdata.us/search/cruise/SR2011

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Project Information

Collaborative research: Using individual amino acids N isotopes in sinking particles and surficial sediments to reconstruct euphotic zone N sources and trophic structure (Amino Acids N Isotopes)

Coverage: Gulf of California, Equatorial Pacific, Sargasso Sea

NSF Award Abstract:
Nitrogen is a limiting nutrient over most of the surface ocean. Fixed nitrogen (N) such as nitrate controls absorption of atmospheric carbon dioxide and production of organic matter by marine plants and algae.

Nitrogen availability, use patterns, and biological community structure in the surface ocean help determine the amount of organic matter passed onto higher organisms. Nitrogen availability also controls how much organic matter sinks into deep waters. This project will reconstruct past sources of nitrogen, use patterns, and trophic structures in surface waters of the Gulf of California, equatorial Pacific, and Sargasso Sea. The tool employed by the principal investigators from Texas A&M University in Corpus Christi and University of Massachusetts Dartmouth is nitrogen isotope ratios of individual amino acids. The investigators will measure isotope ratios in sinking particle samples collected by sediment traps such as those used by the Ocean Flux Program in the Sargasso Sea. This study will train graduate students in stable isotope biogeochemistry and oceanography. This project will also provide research funds for students in the McNair program. McNair students come from underrepresented and economically challenged backgrounds to pursue degrees in STEM fields at Texas A&M University Corpus Christi, a Hispanic and Minority Serving Institution. Data from this project will be made available to the public through the Biological and Chemical Oceanography-Data Management Office (www.bco-dmo.org).

There is great interest in reconstructing past climate-forced variations in nitrogen sources, their patterns of utilization, and euphotic zone community structure using compound specific N isotope ratios in amino acids liberated from preserved proteinaceous materials in sediments and coral skeletons. However, it has not yet been verified whether 1) the nitrogen isotope ratios of individual amino acids produced in the euphotic zone are transported with fidelity by sinking particles to deep-sea corals and sediments and 2) the nitrogen isotope ratios of individual amino acids liberated from sedimentary organic matter have been altered by diagenesis. Through analysis of sediment trap material collected over time, this project seeks to verify that nitrogen isotope ratios in individual amino acids reflect the 1) overall spatial contrast in N sources, utilization patterns, and trophic structures among the Gulf of California, equatorial Pacific, and Sargasso Sea and 2) temporal variations in nitrogen sources, utilization patterns, and trophic structures within both the Gulf of California and equatorial Pacific due to seasonal upwelling and/or El Nino-Southern Oscillation. This study will also test if the nitrogen isotope ratios of total hydrolysable amino acids in sedimentary organic matter from the three locations retain the unaltered nitrogen isotope patterns carried by sinking particles. This project will, for the first time, compare nitrogen stable isotope ratios in amino acids collected from sediment trap samples with surficial sediments from deep-sea oxic sites to verify whether total hydrolysable amino acids in deep-sea sediments preserve unaltered nitrogen isotope signals produced in overlying euphotic zone, which can provide insights on addressing diagenetic alteration of bulk N isotope ratios that have hindered paleo-nitrogen cycle reconstruction.

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Funding

Funding Source	Award
NSF Division of Ocean Sciences (NSF OCE)	OCE-1829947
NSF Division of Ocean Sciences (NSF OCE)	OCE-1829834

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