

# Particulate nitrogen concentrations, N isotopic composition, and nitrate isotopic composition from EN532

**Website:** <https://www.bco-dmo.org/dataset/652025>

**Data Type:** Cruise Results

**Version:** 2

**Version Date:** 2016-07-15

## Project

» [Functional diversity of marine eukaryotic phytoplankton and their contributions to the C and N cycling](#)  
(DimBio NABE)

## Program

» [Dimensions of Biodiversity](#) (Dimensions of Biodiversity)

Contributors	Affiliation	Role
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## Table of Contents

- [Dataset Description](#)
  - [Methods & Sampling](#)
  - [Data Processing Description](#)
- [Data Files](#)
- [Related Datasets](#)
- [Parameters](#)
- [Instruments](#)
- [Deployments](#)
- [Project Information](#)
- [Program Information](#)
- [Funding](#)

## Dataset Description

Pico- and nanoplankton cell concentrations from CTD casts made during the August-September 2013 EN532 and April-May 2014 EN538 cruises aboard R/V Endeavor. Study sites in the subarctic Atlantic Ocean along the 20 °W meridian between 50 °N and 60 °N in September 2013 and May 2014. Two transects from the US East coast to the subarctic study sites were performed as well.

### Related Dataset:

[EN532 - CTD](#)

[EN538 - CTD](#)

[Chlorophyll-a: EN532 and EN538](#)

[Nutrients: EN532 and EN538](#)

[FCM: EN532 and EN538](#)

## Methods & Sampling

Seawater samples for analysis of the N isotopic composition of nitrate+nitrite and nitrate-only were collected unfiltered at regular depth intervals from the surface to 1000 m in 60 ml (>150 m) or 125 ml (<150 m) square-bottomed, wide-mouth HDPE bottles (Nalgene). Bottles were acid-washed and rinsed with deionized water prior to sampling. At sea, pre-labelled bottles and caps were rinsed three times with sample water, filled to ~85% of the bottle volume, and frozen upright at -20°C until analysis.

Isotopic analyses were conducted using the “denitrifier method”, wherein denitrifying bacteria lacking nitrous oxide (N<sub>2</sub>O) reductase quantitatively convert nitrate and nitrite in the sample to N<sub>2</sub>O gas (Sigman et al. 2001, Casciotti et al. 2002) (see also (Weigand et al. in review) for the updated protocol used for analyzing these samples). The isotopic composition of N<sub>2</sub>O was then measured by gas chromatography-isotope ratio mass spectrometry (GC-IRMS) using a purpose-built on-line N<sub>2</sub>O extraction and purification system and a Thermo MAT 253 mass spectrometer. Seawater solutions of the international nitrate reference materials, IAEA-N3 and USGS34, as well as an in-house N<sub>2</sub>O standard, were run in parallel to the samples in order to monitor the quality of bacterial N conversion and mass spectrometric measurements. The reference materials bracketed each group of ~10 samples and were used to correct the measured  $\delta^{15}\text{N}$  to N<sub>2</sub> in air (Sigman et al. 2001, Casciotti et al. 2002, McIlvin & Casciotti 2011).

The measurement of the  $^{15}\text{N}$  of nitrate-only for samples with a detectable concentration of nitrite required a nitrite removal pre-treatment. The detection limit for nitrite in this case was 2 nmol kg<sup>-1</sup>. Samples collected between the surface and ~125 m were treated for nitrite removal via the addition of 10  $\mu\text{l}$  of sulphamic acid solution per ml of sample, which converts sample nitrite to N<sub>2</sub> gas with a reaction time of 2-8 minutes, followed by the addition of 5.5  $\mu\text{l}$  of 2M NaOH per ml of sample to restore the pH of the sample to ~7-9 (Granger & Sigman 2009). The pooled standard error for  $^{15}\text{N}$  was 0.04‰ and 0.11‰ ( $n \geq 3$ ) for nitrate+nitrite and nitrate concentrations  $\geq 0.5 \mu\text{mol l}^{-1}$  and  $< 0.5 \mu\text{mol l}^{-1}$ , respectively. Hereafter, “nitrate” in the text refers to nitrate-only, after the subtraction (for concentration) or removal (for  $^{15}\text{N}$ ) of nitrite.

**Suspended particulate N:** Suspended PN was collected at various depths throughout the euphotic zone, including within the surface mixed layer and at the depth of maximum chlorophyll concentration, by gentle vacuum filtration (<135 mbar), of 8 l of seawater through a GF-75 filter. Filters were transferred to pre-combusted (500°C for 5 h) aluminium foil envelopes, and immediately frozen at -80°C until analysis. In the laboratory, the PN filters were dried in a desiccating oven at 40°C. Three subsamples were cored from each filter and transferred to combusted 4 mL glass Wheaton vials. PN was oxidised to nitrate using the persulphate oxidation method of Knapp et al. (2005), and as modified by Fawcett et al. (2011; 2014); this was conducted in a laminar flow hood equipped with an ammonia/amine filter. Briefly, 2 ml of persulphate oxidizing reagent (POR) were added to each sample vial, as well as to triplicate vials containing a filter blank plus varying quantities of two L-glutamic acid isotope standards, USGS-40 and USGS-41 (Qi et al. 2003); this allows determination of the N content and  $^{15}\text{N}$  of the POR+filter blank. The POR was made by dissolving 2.5 g of 4× recrystallised, methanol-rinsed potassium persulphate and 2.5 g of sodium hydroxide in 100 ml of ultra high-purity deionised water. Following POR addition, vials were autoclaved at 121°C for 55 minutes on a slow-vent setting, after which sample pH was lowered to 5-8 using 12N HCl. The concentration and  $\delta^{15}\text{N}$  of the resultant nitrate was measured via chemiluminescent analysis (Braman & Hendrix 1989) and the denitrifier method (see above) (Sigman et al. 2001, Casciotti et al. 2002). The final N content and  $\delta^{15}\text{N}$  of the oxidised samples was corrected for the POR+filter blank. N content was converted to PN concentration by normalising to whole-filter area and volume of seawater filtered.

#### References:

Braman RS, Hendrix SA (1989) Nanogram nitrite and nitrate determination in environmental and biological materials by vanadium(iii) reduction with chemi-luminescence detection. *Anal Chem* 61:2715-2718

Casciotti K, Sigman D, Hastings MG, Böhlke J, Hilkert A (2002) Measurement of the oxygen isotopic composition of nitrate in seawater and freshwater using the denitrifier method. *Anal Chem* 74:4905-4912

Fawcett SE, Lomas M, Casey JR, Ward BB, Sigman DM (2011) Assimilation of upwelled nitrate by small eukaryotes in the Sargasso Sea. *Nature Geoscience* 4:717-722

Fawcett SE, Lomas MW, Ward BB, Sigman DM (2014) The counterintuitive effect of summer-to-fall mixed layer deepening on eukaryotic new production in the Sargasso Sea. *Glob Biogeochem Cycle* 28:86-102

Granger J, Sigman DM (2009) Removal of nitrite with sulfamic acid for nitrate N and O isotope analysis with the denitrifier method. *Rapid Commun Mass Spectrom* 23:3753-3762

Knapp AN, Sigman DM, Lipschultz F (2005) N isotopic composition of dissolved organic nitrogen and nitrate at the Bermuda Atlantic Time-series Study site. *Glob Biogeochem Cycle* 19

McIlvin MR, Casciotti KL (2011) Technical updates to the bacterial method for nitrate isotopic analyses. Anal Chem 83:1850-1856

Qi H, Coplen TB, Geilmann H, Brand WA, Böhlke J (2003) Two new organic reference materials for  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  measurements and a new value for the  $\delta^{13}\text{C}$  of NBS 22 oil. Rapid Commun Mass Spectrom 17:2483-2487

Sigman D, Casciotti K, Andreani M, Barford C, Galanter M, Böhlke J (2001) A bacterial method for the nitrogen isotopic analysis of nitrate in seawater and freshwater. Anal Chem 73:4145-4153

Weigand MA, Foriel J, Barnett B, Oleynik S, Sigman DM (in review) Updates to instrumentation and protocols for isotopic analysis of nitrate by the denitrifier method. Rapid Commun Mass Spectrom

## Data Processing Description

Standard deviations are derived from at least two analytical measurements

### BCO-DMO Processing:

- added conventional header with dataset name, PI name, version date
- renamed parameters to BCO-DMO standard
- replaced blank cells with nd
- formatted lat and long to 4 decimal places
- replaced original 2016-07-13 EN532 data with new version submitted 2017-07-14. Cast numbers were rearranged.

[ [table of contents](#) | [back to top](#) ]

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## Data Files

File
<b>EN532_PN_NO3_isotopes.csv</b> (Comma Separated Values (.csv), 22.96 KB) MD5:e2d7b2af5ee17f5382b2891483a63299
Primary data file for dataset ID 652025

[ [table of contents](#) | [back to top](#) ]

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## Related Datasets

### IsRelatedTo

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Ward, B. B., Allen, A. E., Sigman, D. M. (2022) **Chlorophyll-a concentrations from CTD cast deployments and underway seawater inflow from Endeavor 532 and Endeavor 538 cruises in 2013 and 2014**. Biological and Chemical Oceanography Data Management Office (BCO-DMO). (Version 2) Version Date 2017-07-17 doi:10.26008/1912/bco-dmo.651784.2 [[view at BCO-DMO](#)]

[ [table of contents](#) | [back to top](#) ]

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## Parameters

Parameter	Description	Units
cruise_id	cruise identification	unitless
cast	cast number	unitless
ISO_DateTime_UTC	UTC day and decimal time; as 326.5 for the 326th day of the year or November 22 at 1200 hours (noon).	yrday_utc
lat	latitude; north is positive	decimal degrees
lon	longitude; east is positive	decimal degrees
depth	depth	meters
PN_muM	particulate nitrogen concentration	umol/liter
SD_PN_muM	standard deviation of particulate nitrogen concentration	umol/liter
d15NPN_permil	delta 15N vs. atmospheric N2 of particulate nitrogen	per mille
SDd15NPN_permil	standard deviation of delta 15N vs. atmospheric N2 of particulate nitrogen	per mille
d15NNO3NO2_permil	delta 15N vs. atmospheric N2 of nitrate+nitrite	per mille
SDd15NNO3NO2_permil	standard deviation of delta 15N vs. atmospheric N2 of nitrate+nitrite	per mille
d18ONO3NO2_permil	delta 18O vs. VSMOW of nitrate+nitrite	per mille
SDd18ONO3NO2_permil	standard deviation of delta 18O vs. VSMOW of nitrate+nitrite	per mille
d15NNO3_permil	delta 15N vs. atmospheric N2 of nitrate	per mille
SDd15NNO3_permil	standard deviation of delta 15N vs. atmospheric N2 of nitrate	per mille
d18ONO3_permil	delta 18O vs. VSMOW of nitrate	per mille
SDd18ONO3_permil	standard deviation of delta 18O vs. VSMOW of nitrate	per mille

## Instruments

<b>Dataset-specific Instrument Name</b>	
<b>Generic Instrument Name</b>	CTD Sea-Bird SBE 911plus
<b>Generic Instrument Description</b>	The Sea-Bird SBE 911 plus is a type of CTD instrument package for continuous measurement of conductivity, temperature and pressure. The SBE 911 plus includes the SBE 9plus Underwater Unit and the SBE 11plus Deck Unit (for real-time readout using conductive wire) for deployment from a vessel. The combination of the SBE 9 plus and SBE 11 plus is called a SBE 911 plus. The SBE 9 plus uses Sea-Bird's standard modular temperature and conductivity sensors (SBE 3 plus and SBE 4). The SBE 9 plus CTD can be configured with up to eight auxiliary sensors to measure other parameters including dissolved oxygen, pH, turbidity, fluorescence, light (PAR), light transmission, etc.). more information from Sea-Bird Electronics

<b>Dataset-specific Instrument Name</b>	GC-IRMS
<b>Generic Instrument Name</b>	Isotope-ratio Mass Spectrometer
<b>Dataset-specific Description</b>	Thermo MAT 253 mass spectrometer
<b>Generic Instrument Description</b>	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).

<b>Dataset-specific Instrument Name</b>	
<b>Generic Instrument Name</b>	Niskin bottle
<b>Generic Instrument Description</b>	A Niskin bottle (a next generation water sampler based on the Nansen bottle) is a cylindrical, non-metallic water collection device with stoppers at both ends. The bottles can be attached individually on a hydrowire or deployed in 12, 24, or 36 bottle Rosette systems mounted on a frame and combined with a CTD. Niskin bottles are used to collect discrete water samples for a range of measurements including pigments, nutrients, plankton, etc.

## Deployments

EN532

<b>Website</b>	<a href="https://www.bco-dmo.org/deployment/563687">https://www.bco-dmo.org/deployment/563687</a>
<b>Platform</b>	R/V Endeavor
<b>Report</b>	<a href="http://dmoserv3.bco-dmo.org/data_docs/DimBio_NABE/EN532_CruiseReport.pdf">http://dmoserv3.bco-dmo.org/data_docs/DimBio_NABE/EN532_CruiseReport.pdf</a>
<b>Start Date</b>	2013-08-22
<b>End Date</b>	2013-09-15
<b>Description</b>	Study sites in the subtropical North-Atlantic Ocean near the Bermuda Atlantic Time Series in February 2012 and August 2012, and in the subarctic Atlantic Ocean along the 20W meridian between 50N and 60N in September 2013 and May 2014. Two transects from the US East coast to the subarctic study sites were performed as well.

[ [table of contents](#) | [back to top](#) ]

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## Project Information

### Functional diversity of marine eukaryotic phytoplankton and their contributions to the C and N cycling (DimBio NABE)

**Coverage:** North Atlantic Ocean, transects from southwest to northeast

This project will investigate the taxonomic, genetic and functional diversity of eukaryotic phytoplankton at two North Atlantic sites (subarctic and subtropical) in two seasons. The PIs will use diagnostic microarrays for community analysis based on functional genes (both DNA and RNA) and next generation sequencing (i.e., transcriptomics using 454 technology) to identify the players, both in terms of community composition and activity, and to explore the functional diversity of the natural assemblage. In order to identify which groups are active in C and N assimilation and which N source is being utilized by the different size and functional groups, both filter-separated and flow cytometry-sorted samples will be used to 1) measure <sup>13</sup>C primary production and <sup>15</sup>N assimilation by incubations with isotope tracers, 2) measure the natural stable N isotope signatures of different taxonomic groups and 3) link the molecular diversity to the functional diversity in C and N transformations. Using flow cytometry linked to mass spectrometry, these investigators have found an unexpectedly strong differentiation in the form of N assimilated by prokaryotes and eukaryotes, with eukaryotes being more dynamic.

This project will investigate the taxonomic, genetic and functional diversity of eukaryotic phytoplankton and to link this diversity and assemblage composition to the carbon and nitrogen biogeochemistry of the surface ocean. Taxonomic diversity will be investigated by identifying the components of the phytoplankton assemblages using molecular, chemical and microscope methods. Genetic diversity will be explored at several levels, including direct sequencing of clone libraries of key functional genes and metatranscriptomic sequencing and microarray analysis of size fractionated/sorted phytoplankton assemblages. Using natural abundance and tracer stable isotope methods, genetic and taxonomic diversity will be linked to functional diversity in C and N assimilation in size- fractionated and taxon-sorted populations.

[ [table of contents](#) | [back to top](#) ]

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## Program Information

### Dimensions of Biodiversity (Dimensions of Biodiversity)

**Website:** [http://www.nsf.gov/funding/pgm\\_summ.jsp?pims\\_id=503446](http://www.nsf.gov/funding/pgm_summ.jsp?pims_id=503446)

**Coverage:** global

(adapted from the NSF Synopsis of Program)

Dimensions of Biodiversity is a program solicitation from the NSF Directorate for Biological Sciences. FY 2010 was year one of the program. [\[MORE from NSF\]](#)

The NSF Dimensions of Biodiversity program seeks to characterize biodiversity on Earth by using integrative, innovative approaches to fill rapidly the most substantial gaps in our understanding. The program will take a broad view of biodiversity, and in its initial phase will focus on the integration of genetic, taxonomic, and functional dimensions of biodiversity. Project investigators are encouraged to integrate these three dimensions to understand the interactions and feedbacks among them. While this focus complements several core NSF programs, it differs by requiring that multiple dimensions of biodiversity be addressed simultaneously, to understand the roles of biodiversity in critical ecological and evolutionary processes.

[ [table of contents](#) | [back to top](#) ]

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## Funding

Funding Source	Award
<a href="#">NSF Division of Ocean Sciences (NSF OCE)</a>	<a href="#">OCE-1136345</a>

[ [table of contents](#) | [back to top](#) ]