

# Effects of sample volume on N<sub>2</sub>O recovery and isotopic analysis (Biological Nitrogen Isotope Fractionation project)

**Website:** <https://www.bco-dmo.org/dataset/865053>

**Data Type:** experimental

**Version:** 1

**Version Date:** 2021-11-16

## Project

» [CAREER: The biological nitrogen isotope systematics of ammonium consumption and production](#)  
(Biological Nitrogen Isotope Fractionation)

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## Abstract

Incomplete N<sub>2</sub>O analyte recovery during sparging of high-volume samples and associated N and O isotopic ratio offsets.

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## Coverage

**Temporal Extent:** 2012-01-31 - 2020-08-24

## Acquisition Description

### Methodology:

### Sampling and analytical procedures:

□ Effects of sample volume on N<sub>2</sub>O isotope analyses

Crimp-sealed vials (20 mL) containing incremental volumes (0 to 14 mL) of DIW or NO<sub>3</sub><sup>-</sup>-deplete seawater were sparged with N<sub>2</sub> gas for 30 minutes, aliquoted with N<sub>2</sub>O gas (10 nmol N) and equilibrated for ≥ 24 hours. The N<sub>2</sub>O gas in the vials was then extracted, purified and its N and O isotopic composition analyzed on the isotope ratio mass spectrometer. In a parallel set of experiments conducted at Princeton University, referring to Trial "Bonnie 5nmol", crimp-sealed vials (20 mL) containing incremental volumes (0 to 9.3 mL) of DIW were sparged with helium gas for 30 minutes, aliquoted with N<sub>2</sub>O gas (5 nmol N) and equilibrated on a shaker for 2 hours. The N<sub>2</sub>O gas in the vials was then extracted with an autosampler

needle that only penetrated samples  $\geq 8.3$  mL. N<sub>2</sub>O thus extracted was purified and its N and O isotopic composition analyzed on an isotope ratio mass spectrometer.

## Processing Description

### Processing notes from submitting researcher:

- Data were processed using Microsoft Excel

### BCO-DMO processing notes

- Date formats were changed from mm/dd/yy to yyyy-mm-dd
- Spaces and units removed from column headers

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## Related Publications

Casciotti, K. L., Sigman, D. M., Hastings, M. G., Böhlke, J. K., & Hilkert, A. (2002). Measurement of the Oxygen Isotopic Composition of Nitrate in Seawater and Freshwater Using the Denitrifier Method. *Analytical Chemistry*, 74(19), 4905–4912. doi:[10.1021/ac020113w](https://doi.org/10.1021/ac020113w)  
*Methods*

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## Parameters

Parameter	Description	Units
Aliquot	Types of aliquot: freshwater or seawater	unitless
Date	Date of the experiments; yyyy-mm-dd	unitless
Trial	Trial name	unitless
GC_IRMS	Gas chromatography combustion isotope ratio mass spectrometry (GC_IRMS) used for isotope analysis: Thermo Delta V at Uconn or Thermo MAT253 GC-IRMS at Princeton	unitless
N2O_injection	The amount of N <sub>2</sub> O gas injected	nmol of N
Aliquot_volume	Volume of aliquot addition	mL
N2O_peak_area	Recoverd N <sub>2</sub> O peak area measured with a Thermo Delta V GC-IRMS with modified Gas Bench II and a PAL autosampler	Vs
delta_15N	N isotopic composition of N <sub>2</sub> O measured with a Thermo Delta V GC-IRMS with modified Gas Bench II and a PAL autosampler	‰ vs. N <sub>2</sub> O <sub>tank</sub>
delta_18O	O isotopic composition of N <sub>2</sub> O measured with a Thermo Delta V GC-IRMS with modified Gas Bench II and a PAL autosampler	‰ vs. N <sub>2</sub> O <sub>tank</sub>

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## Instruments

<b>Dataset-specific Instrument Name</b>	Delta V Advantage and MAT253 continuous flow gas chromatograph isotope ratio mass spectrometer (Thermo Fisher Scientific, Waltham, MA, USA)
<b>Generic Instrument Name</b>	Gas Chromatograph Mass Spectrometer
<b>Dataset-specific Description</b>	Delta V Advantage and MAT253 continuous flow gas chromatograph isotope ratio mass spectrometer (Thermo Fisher Scientific, Waltham, MA, USA) interfaced with a modified Thermo Fisher Scientific Gas Bench sample preparation device fronted by dual cold traps (Casciotti et al., 2002) and a GC Pal autosampler (CTC Analytics, Zwingen, Switzerland) - to measure N and O isotope ratio of nitrate using the denitrified method.
<b>Generic Instrument Description</b>	Instruments 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 by a mass spectrometer.

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## Project Information

### **CAREER: The biological nitrogen isotope systematics of ammonium consumption and production (Biological Nitrogen Isotope Fractionation)**

#### **NSF Award Abstract:**

The nitrogen (N) cycle in the marine environment is controlled by biological processes. Unfortunately, quantifying these processes and assessing their effect on the N cycle is difficult by direct measurements because of large spatial and temporal differences. Isotopic composition measurements of N provide a means to constrain these processes indirectly; however, there is still a great deal to be understood about isotope fractionation of recycled nitrogen through biological processes, which has made interpretation of novel nitrogen isotope data difficult. A researcher from the University of Connecticut plans to determine the influence of biological consumption and production on the isotope fractionation in ammonium. By helping to understand the processes surrounding fractionation of recycled ammonium at the organism level, this research will create a basis for which future researchers can better interpret isotope composition data to infer nitrogen cycle dynamics. A graduate student, a postdoctoral fellow, and two or more undergraduate students will be involved in the research. The researcher plans to integrate science with community-engaged learning by developing an undergraduate field and laboratory course that will require the students to present their research to stakeholders in the community. There will be a manual created for this course that will be disseminated in open-access forums for teachers hoping to develop similar courses.

Biological nitrogen isotope fractionation associated with nitrogen recycling remains poorly constrained despite the advent of a variety of new techniques to analyze nitrogen isotopes in recent years. The use of isotopic composition data can be incredibly useful to interpreting nitrogen cycle processes in the ocean that are difficult to measure directly, which makes it crucial to further understand the processes behind fractionation to catch up with the advancement of the datasets available to researchers. This research will characterize the isotope fractionation dynamics of ammonium during biological consumption and production. The researchers will investigate whether the characteristic low concentrations of ammonium in the surface ocean affect isotope fractionation when the ammonium is recycled and whether there is a trophic isotope effect associated with ammonium recycling by protozoan grazers. With this research, there will be a baseline from which researchers can interpret recycled nitrogen dynamics from ammonium isotope datasets. The methods of comparing nitrogen cycling studies will become significantly clearer with such a standard making interpretation uniform by removing significant uncertainties.

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## Funding

Funding Source	Award
<a href="#">NSF Division of Ocean Sciences (NSF OCE)</a>	<a href="#">OCE-1554474</a>

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