

# Flow through sediment core incubations for nitrogen concentration and isotopic fluxes collected in 2013 on the Island of Sylt, Germany in the North Sea.

**Website:** <https://www.bco-dmo.org/dataset/827378>

**Data Type:** experimental

**Version:** 1

**Version Date:** 2022-04-08

## Project

» [Collaborative Research: Nitrous Oxide Production and Fluxes in Coastal Sediments: Response to Environmental Change](#) (Coastal\_Nitrous\_Oxide)

Contributors	Affiliation	Role
<a href="#">Wankel, Scott</a>	Woods Hole Oceanographic Institution (WHOI)	Principal Investigator
<a href="#">Ziebis, Wiebke</a>	University of Southern California (USC)	Co-Principal Investigator
<a href="#">Haskins, Christina</a>	Woods Hole Oceanographic Institution (WHOI BCO-DMO)	BCO-DMO Data Manager

## Abstract

Flow through sediment core incubations for nitrogen concentration and isotopic fluxes collected in 2013 on the Island of Sylt, Germany in the North Sea.

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## Coverage

**Temporal Extent:** 2013-08-14 - 2013-08-22

## Acquisition Description

Sediments were collected from three intertidal sites near Königshafen on the island of Sylt in the North Sea, Germany. The 'Schlickwatt (CL)' and 'Mischwatt (SLT)' sites were located inside a small lagoon, while the 'Sandwatt (SD)' site was more openly exposed to wind and waves. Thirty intact push cores (30cm length, 10cm OD) were taken using polycarbonate core liners having vertical lines of silicone sealed holes ( $\varnothing$  3mm) at 1-cm intervals to allow porewater collection. Cores were retrieved leaving ~10 cm of overlying water and sealed with double o-ring caps to minimize gas exchange during transport, and brought immediately back to the laboratory. The gas-tight sealed sediment cores were incubated in the dark at *in situ* temperatures (19°C) while being continuously supplied with filtered seawater at a flow rate of 1.8 0.06 ml/min for ~8 days. For experimental manipulations, four different inflow seawater compositions were used: "Low nitrate" (air sparged; ~20uM; LN), "Low oxygen, low nitrate" (sparged with N<sub>2</sub> to 30-35% O<sub>2</sub> saturation; ~20uM; LOLN), "High nitrate" (amended with NaNO<sub>3</sub> to ~120uM (above background nitrate); HN) and "low oxygen, high nitrate" (combined treatments; LOHN). Samples of each sediment core effluent were taken twice per day.

Concentrations of NO<sub>3</sub><sup>-</sup> + NO<sub>2</sub><sup>-</sup> were measured by chemiluminescence after reduction in a hot acidic vanadyl sulfate solution on a NO<sub>x</sub> analyzer (Braman and Hendrix, 1989). Concentrations of NO<sub>2</sub><sup>-</sup> were quantified by using the Griess-Ilosvay method followed by measuring absorption 540nm, and NO<sub>3</sub><sup>-</sup> was quantified by difference (Grasshoff et al., 1999). Concentrations of NH<sub>4</sub><sup>+</sup> were measured by fluorescence using the OPA

method (Holmes et al., 1999). Concentrations of N<sub>2</sub>O were made using the integrated peak area of the m/z 44 beam on the IRMS, standardizing to analyses of known amounts of N<sub>2</sub>O (injected into N<sub>2</sub> sparged seawater in 160ml serum bottles) and normalizing to sample volume (158ml).

All N and O isotopic composition measurements (d<sub>15</sub>N and d<sub>18</sub>O (or d<sub>17</sub>O); where d<sub>15</sub>N = [(15R<sub>sample</sub>/15R<sub>Air</sub>)-1]\*1000 in units of ‰, and 15R = 15N/14N and where d<sub>18</sub>O = [(18R<sub>sample</sub>/18R<sub>VSMOW</sub>)-1]\*1000 in units of ‰, and 18R = 18O/16O (or 17O/16O) were made after conversion of analytes to nitrous oxide, followed by purification with a customized purge and trap system similar to that previously described (McIlvin and Casciotti, 2010) and analysis on a continuous flow IsoPrime 100 isotope ratio mass spectrometer (IRMS). D<sub>17</sub>O refers to the excess 17O beyond that defined by the terrestrial fractionation line for the oxygen isotope system and is defined as D<sub>17</sub>O = d<sub>17</sub>O\*0.52 - d<sub>18</sub>O. Nitrate was converted to N<sub>2</sub>O using the denitrifier method (Casciotti et al., 2002; Sigman et al., 2001) after removal of nitrite by addition of sulfamic acid (Granger and Sigman, 2009). Corrections for drift, size and fractionation of O isotopes during bacterial conversion were carried out as previously described using NO<sub>3</sub>- standards USGS 32, USGS 34 and USGS 35 (Casciotti et al., 2002; McIlvin and Casciotti, 2011), with a typical reproducibility of 0.2‰ and 0.4‰ for d<sub>15</sub>N and d<sub>18</sub>O, respectively. Nitrate D<sub>17</sub>O measurements were made on separate aliquots by routing denitrifier-produced N<sub>2</sub>O through a gold tube (1/16" OD) held at 780°C, thermally decomposing the N<sub>2</sub>O into N<sub>2</sub> and O<sub>2</sub>, which were chromatographically separated using a 2m column (1/16" OD) packed with molecular sieve (5Å) before being sent into the IRMS (Kaiser et al., 2007; Komatsu et al., 2008). Nitrate standards USGS 35 and USGS 34 were used to normalize any scale contraction during conversion, with typical reproducibility of D<sub>17</sub>O measurements of 0.8‰. All samples for nitrite N and O isotope measurements were converted to N<sub>2</sub>O within 2 hours of collection using the azide method (McIlvin and Altabet, 2005). Internal nitrite isotope standards (WILIS 10, 11 and 20) were run in parallel at 3 different sizes to correct for any variations in sample size and instrumental drift, with a typical reproducibility for both d<sub>15</sub>N and d<sub>18</sub>O is 0.2‰. Based on calibrations against isotope standards USGS 32, 34 and 35 for d<sub>15</sub>N (Böhlke et al., 2003) and N<sub>23</sub>, N7373, and N10129 for d<sub>18</sub>O (Casciotti et al., 2007), the values of internal standards WILIS 10, 11, and 20 are reported here as -1.7, +57.1, and -7.8‰ for d<sub>15</sub>N and +13.2, +8.6 and +47.6‰ for d<sub>18</sub>O, respectively. Nitrite D<sub>17</sub>O measurements were made after conversion to N<sub>2</sub>O using the azide method and normalized using a combination of NO<sub>2</sub>- and NO<sub>3</sub>- isotopic standards. D<sub>17</sub>O values of NO<sub>2</sub>- isotope standards WILIS 10 and WILIS 11 were calibrated previously against USGS 34 and USGS 35 using the denitrifier method followed by thermal decomposition of N<sub>2</sub>O to N<sub>2</sub> and O<sub>2</sub> as described above - yielding D<sub>17</sub>O values of 0‰ for both. For sample NO<sub>2</sub>-, raw d<sub>17</sub>O and d<sub>18</sub>O values were first normalized for oxygen isotopic exchange with water during the azide reaction (McIlvin and Altabet, 2005) using the calibrated d<sub>17</sub>O and d<sub>18</sub>O values of WILIS 10 and WILIS 11. During the same IRMS run, N<sub>2</sub>O produced from USGS 34 and USGS 35 via the denitrifier method was also thermally converted and analyzed as N<sub>2</sub> and O<sub>2</sub>. Because any isotope fractionation occurring during these reactions is mass dependent (e.g., D<sub>17</sub>O is unaffected), the D<sub>17</sub>O of NO<sub>2</sub>- can be calculated by normalizing to D<sub>17</sub>O values of these NO<sub>3</sub>- standards. We disregard the small amount of oxygen isotope exchange occurring during the denitrifier method, as this would have only a small impact on the calculated D<sub>17</sub>O values. Total reduced nitrogen (TRN, e.g., DON + NH<sub>4</sub><sup>+</sup>) was measured in a subset of incubation cores by oxidation of the total dissolved nitrogen (TDN) pool via persulfate digest - followed by d<sub>15</sub>N analysis using the denitrifier method, similar to that previously described (Knapp et al., 2005). The d<sub>15</sub>N of the TRN pool was then calculated by mass balance by subtracting the molar contribution of the measured d<sub>15</sub>N of NO<sub>3</sub>- and NO<sub>2</sub>- pools to the TDN pool. Based on the measurement of NH<sub>4</sub><sup>+</sup> concentrations, the DON flux was generally of the same magnitude as the NH<sub>4</sub><sup>+</sup> flux (not shown). For dissolved N<sub>2</sub>O, samples were extracted from the 160ml serum bottles using a purge and trap approach similar to that previously described (McIlvin and Casciotti, 2010). Liquid samples were quantitatively transferred from the sample bottle into a purging flask using a 20psi He stream, followed by He-sparging (~45 min) and cryogenic trapping using the same system described above for nitrate and nitrite derived N<sub>2</sub>O. Isotopic composition of the dissolved N<sub>2</sub>O was measured by direct comparison against the N<sub>2</sub>O reference tank. The composition of this tank (d<sub>15</sub>N bulk = -0.7‰; d<sub>18</sub>O = +39.1‰; site preference (SP) = -5.3‰, where SP = d<sub>15</sub>N alpha - d<sub>15</sub>N beta, and alpha and beta refer to the central and outer N atoms in the linear N<sub>2</sub>O molecule, respectively) was calibrated directly against aliquots of two previously calibrated N<sub>2</sub>O tanks from the Ostrom Lab at Michigan State University, having been calibrated by Tokyo Tech. Several sample analyses of tropospheric N<sub>2</sub>O from the study site using this system yielded isotope values of +6.8 - 0.7‰ for d<sub>15</sub>N bulk, +44.1 - 1.7‰ for d<sub>18</sub>O and +17.4 - 2.2‰ for SP. Reported values have been corrected for any size linearity of isotopic ratios (31/30, 45/44 and 46/44) by using a series of reference tank subsamples injected into 20ml headspace vials using a gastight syringe. Precision for replicate analyses of our reference gas analyzed as samples for d<sub>15</sub>N is 0.3‰, for d<sub>18</sub>O is 0.4‰ and for SP is 0.8‰. The D<sub>17</sub>O of N<sub>2</sub>O was calculated similar to that described above for NO<sub>2</sub>-. After extraction and cryotrapping, the N<sub>2</sub>O sample is thermally decomposed to N<sub>2</sub> and O<sub>2</sub> and chromatographically separated before measurement on the IRMS. Regular analyses of N<sub>2</sub>O converted from NO<sub>3</sub>- isotope standards (USGS 35 and USGS 34) via the denitrifier method were made to normalize D<sub>17</sub>O values.

## Processing Description

Mass fluxes were calculated as a function of the steady-state difference between influent and effluent concentrations ([C]), flow rate (r) and sediment surface area (A) using:  $\text{Flux} = ([C]\text{change} * r)/A$ . Error estimates of fluxes incorporate variations in both measured flow rates as well as steady state concentrations.

For dissolved ions, effluent was directed into HDPE bottles and allowed to fill for ~60 minutes before subsampling, filtering (0.2µm) and freezing (-20°C). Separate 20ml aliquots were taken for measurement of dissolved inorganic nitrogen concentrations (nitrate, nitrite and ammonium) and stable isotopic composition. Concentrations of nitrite and ammonium were made immediately, while nitrate concentrations were measured back in the Wankel lab at WHOI. Samples for dissolved N<sub>2</sub>O were directed through gas impermeable PEEK tubing directly into pre-evacuated Tedlar gas sampling bags followed by gentle transfer into 160ml serum bottles using a ¼" OD silicone tubing, filling from the bottom to minimize turbulence and gas exchange. Sample water was allowed to overflow the bottle volume for at least two volumes before crimp-sealing and preserving with 100µl of a saturated HgCl<sub>2</sub> solution.

BCO-DMO Data Manager Processing Notes:

- \* added a conventional header with dataset name, PI name, version date
- \* modified parameter names to conform with BCO-DMO naming conventions
- \* blank values in this dataset are displayed as "nd" for "no data." nd is the default missing data identifier in the BCO-DMO system.
- \* removed all spaces in headers and replaced with underscores
- \* removed all units from headers
- \* converted dates to ISO Format yyyy-mm-dd
- \* created Date\_Local column to replace Date column
- \* created ISO\_DateTime\_Local from Time\_Stamp column
- \* set Types for each data column

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## Related Publications

Braman, R. S., & Hendrix, S. A. (1989). Nanogram nitrite and nitrate determination in environmental and biological materials by vanadium(III) reduction with chemiluminescence detection. *Analytical Chemistry*, 61(24), 2715–2718. doi:[10.1021/ac00199a007](https://doi.org/10.1021/ac00199a007)

*Methods*

Böhlke, J. K., Mroczkowski, S. J., & Coplen, T. B. (2003). Oxygen isotopes in nitrate: new reference materials for 18O:17O:16O measurements and observations on nitrate-water equilibration. *Rapid Communications in Mass Spectrometry*, 17(16), 1835–1846. doi:[10.1002/rcm.1123](https://doi.org/10.1002/rcm.1123)

*Methods*

Casciotti, K. L., Böhlke, J. K., McIlvin, M. R., Mroczkowski, S. J., & Hannon, J. E. (2007). Oxygen Isotopes in Nitrite: Analysis, Calibration, and Equilibration. *Analytical Chemistry*, 79(6), 2427–2436. doi:[10.1021/ac061598h](https://doi.org/10.1021/ac061598h)

*Methods*

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*

Granger, J., & Sigman, D. M. (2009). Removal of nitrite with sulfamic acid for nitrate N and O isotope analysis with the denitrifier method. *Rapid Communications in Mass Spectrometry*, 23(23), 3753–3762.

doi:[10.1002/rcm.4307](https://doi.org/10.1002/rcm.4307)

*Methods*

Grasshoff, K., Kremling, K., & Ehrhardt, M. (Eds.). (2009). *Methods of seawater analysis*. John Wiley & Sons. <https://isbnsearch.org/isbn/978-3-527-61399-1>

*Methods*

Holmes, R. M., Aminot, A., Kérouel, R., Hooker, B. A., & Peterson, B. J. (1999). A simple and precise method for measuring ammonium in marine and freshwater ecosystems. *Canadian Journal of Fisheries and Aquatic*

Sciences, 56(10), 1801–1808. doi:[10.1139/f99-128](https://doi.org/10.1139/f99-128)  
*Methods*

Kaiser, J., Hastings, M. G., Houlton, B. Z., Röckmann, T., & Sigman, D. M. (2007). Triple Oxygen Isotope Analysis of Nitrate Using the Denitrifier Method and Thermal Decomposition of N<sub>2</sub>O. *Analytical Chemistry*, 79(2), 599–607. doi:[10.1021/ac061022s](https://doi.org/10.1021/ac061022s)  
*Methods*

Knapp, A. N., Sigman, D. M., & Lipschultz, F. (2005). N isotopic composition of dissolved organic nitrogen and nitrate at the Bermuda Atlantic Time-series Study site. *Global Biogeochemical Cycles*, 19(1). doi:10.1029/2004gb002320 <https://doi.org/10.1029/2004GB002320>  
*Methods*

Komatsu, D. D., Ishimura, T., Nakagawa, F., & Tsunogai, U. (2008). Determination of the <sup>15</sup>N/<sup>14</sup>N, <sup>17</sup>O/<sup>16</sup>O, and <sup>18</sup>O/<sup>16</sup>O ratios of nitrous oxide by using continuous-flow isotope-ratio mass spectrometry. *Rapid Communications in Mass Spectrometry*, 22(10), 1587–1596. doi:[10.1002/rcm.3493](https://doi.org/10.1002/rcm.3493)  
*Methods*

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](https://doi.org/10.1021/ac050528s)  
*Methods*

McIlvin, M. R., & Casciotti, K. L. (2010). Fully automated system for stable isotopic analyses of dissolved nitrous oxide at natural abundance levels. *Limnology and Oceanography: Methods*, 8(2), 54–66. doi:[10.4319/lom.2010.8.54](https://doi.org/10.4319/lom.2010.8.54)  
*Methods*

McIlvin, M. R., & Casciotti, K. L. (2011). Technical Updates to the Bacterial Method for Nitrate Isotopic Analyses. *Analytical Chemistry*, 83(5), 1850–1856. doi:[10.1021/ac1028984](https://doi.org/10.1021/ac1028984)  
*Methods*

Sigman, D. M., Casciotti, K. L., Andreani, M., Barford, C., Galanter, M., & Böhlke, J. K. (2001). A Bacterial Method for the Nitrogen Isotopic Analysis of Nitrate in Seawater and Freshwater. *Analytical Chemistry*, 73(17), 4145–4153. doi:[10.1021/ac010088e](https://doi.org/10.1021/ac010088e)  
*Methods*

Wankel, S. D., Ziebis, W., Buchwald, C., Charoenpong, C., de Beer, D., Dentinger, J., ... Zengler, K. (2017). Evidence for fungal and chemodenitrification based N<sub>2</sub>O flux from nitrogen impacted coastal sediments. *Nature Communications*, 8(1). doi:10.1038/ncomms15595 <https://doi.org/10.1038/NCOMMS15595>  
*Methods*  
*Results*

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## Parameters

Parameter	Description	Units
N_number	Internal Lab ID	dimensionless
Study_Name	Study Identifier	dimensionless
Date_Local	Date Sample Collected	%Y-%m-%d
Bottle_Fill_Start	Time Water Sample Collection began	%H:%M
Bottle_Fill_Stop	Time Water Sample Collection ended	%H:%M
Gas_Bag_Start	Time Gas Sample Collection began	%H:%M
Gas_Bag_Stop	Time Gas Sample Collection ended	%H:%M
Time_Stamp	Time Registered for Sample	mm/dd/yy hh:mm
Elapsed	Time Since Incubation Initiation	Hours
Unique_ID	Unique Sample Identifier	dimensionless
Comment	Comment, Sample ID	dimensionless

Site_Type	Predominant Sediment Type	dimensionless
Core_ID	Unique Sediment Core ID	dimensionless
Duplicate_ID	Core A or B	dimensionless
Treatment	Experimental Treatment	unitless
Timepoint	Sequential Sampling Time point	dimensionless
Flow_Rate	Seawater Flow Rate through core	mL/min
Inflow_NO2	Inflow nitrite concentration	uM
Inflow_NH4	Inflow ammonium concentration	uM
Inflow_NO3	Inflow nitrate concentration	uM
Inflow_N2O	Inflow Nitrous oxide concentration	uM
Effluent_NO2	Effluent nitrite concentration	uM
Effluent_NH4	Effluent ammonium concentration	uM
Effluent_NO3	Effluent nitrate concentration	uM
Effluent_DON	Effluent dissolved organic nitrogen	uM
Effluent_TDN	Effluent total dissolved nitrogen	uM
Effluent_N2O	Effluent nitrous oxide concentration	nM
O2_Flux	Calculated Dissolved Oxygen Flux	mmol m <sup>-2</sup> d <sup>-1</sup>
NO2_Flux	Calculated Nitrite Flux	mmol m <sup>-2</sup> d <sup>-1</sup>
NH4_Flux	Calculated Ammonium Flux	mmol m <sup>-2</sup> d <sup>-1</sup>
NO3_Flux	Calculated Nitrate Flux	mmol m <sup>-2</sup> d <sup>-1</sup>
DON_Flux	Calculated DON Flux	mmol m <sup>-2</sup> d <sup>-1</sup>
N2O_Flux	Calculated N2O Flux	umol m <sup>-2</sup> d <sup>-1</sup>
NO3_d15N	Nitrate nitrogen isotope ratio	per mil
NO3_d18O	Nitrate oxygen isotope ratio	per mil
NO3_D17O	17O isotope excess in nitrate	per mil
NO2_d15N	Nitrite nitrogen isotope ratio	per mil
NO2_d18O	Nitrite oxygen isotope ratio	per mil
NO2_D17O	17O isotope excess in nitrite	per mil
TDN_d15N	Total dissolved nitrogen isotope ratio	per mil
TRN_d15N	Total reduced nitrogen isotope ratio	per mil
N2O_d15N	Nitrous oxide nitrogen isotope ratio	per mil
N2O_d18O	Nitrous oxide oxygen isotope ratio	per mil
N2O_SP	Nitrogen Isotope Site Preference N2O	per mil
N2O_D17O	17O isotope excess in N2O	per mil
ISO_DateTime_Local	Date/Time (Local) ISO formatted	%Y-%m-%dT%H:%M

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## Instruments

<b>Dataset-specific Instrument Name</b>	Turner Designs Aquafluor Fluorometer
<b>Generic Instrument Name</b>	Fluorometer
<b>Generic Instrument Description</b>	A fluorometer or fluorimeter is a device used to measure parameters of fluorescence: its intensity and wavelength distribution of emission spectrum after excitation by a certain spectrum of light. The instrument is designed to measure the amount of stimulated electromagnetic radiation produced by pulses of electromagnetic radiation emitted into a water sample or in situ.

<b>Dataset-specific Instrument Name</b>	Isoprime 100 Isotope Ratio Mass Spectrometer
<b>Generic Instrument Name</b>	Mass Spectrometer
<b>Generic Instrument Description</b>	General term for instruments used to measure the mass-to-charge ratio of ions; generally used to find the composition of a sample by generating a mass spectrum representing the masses of sample components.

<b>Dataset-specific Instrument Name</b>	Shimadzu UV 2550 Spectrophotometer
<b>Generic Instrument Name</b>	Spectrophotometer
<b>Generic Instrument Description</b>	An instrument used to measure the relative absorption of electromagnetic radiation of different wavelengths in the near infra-red, visible and ultraviolet wavebands by samples.

<b>Dataset-specific Instrument Name</b>	Teledyne 200e NOx Analyzer
<b>Generic Instrument Name</b>	Chemiluminescence NOx Analyzer
<b>Generic Instrument Description</b>	The chemiluminescence method for gas analysis of oxides of nitrogen relies on the measurement of light produced by the gas-phase titration of nitric oxide and ozone. A chemiluminescence analyzer can measure the concentration of NO/NO <sub>2</sub> /NO <sub>x</sub> . One example is the Teledyne Model T200: <a href="http://www.teledyne-api.com/products/T200.asp">http://www.teledyne-api.com/products/T200.asp</a>

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## Project Information

### Collaborative Research: Nitrous Oxide Production and Fluxes in Coastal Sediments: Response to Environmental Change (Coastal\_Nitrous\_Oxide)

**Coverage:** Wadden Sea Field Station, Sylt, Germany; Santa Catalina Island, California, USA

## NSF Abstract:

Although marine sediments are known "hotspots" of nitrous oxide (N<sub>2</sub>O) production and emission, current estimates and future projections of this potent greenhouse gas from coastal areas, especially in response to lower levels of dissolved oxygen and increased nitrogen inputs, are an approximation at best. Scientists from the University of Southern California and Woods Hole Oceanographic Institute plan to improve upon these values by determining N<sub>2</sub>O dynamics at two coastal sites, Sylt, the German Wadden Sea, and Santa Catalina Island, a California Coastal lagoon. To attain their goal, they will carry out in-situ, high resolution microsensor measurements of N<sub>2</sub>O, oxygen, nitrate, nitric oxide, hydrogen sulfide, pH, redox potential, and temperature in conjunction with sediment and pore water analyses. Some of the sediment cores to be collected will be subjected to changes in oxygen content and nitrate concentrations in the overlying water to determine changes in nitrogen cycling activity and N<sub>2</sub>O flux as a function of low oxygen or nitrate addition. Using experimental incubations, the isotopic composition of N<sub>2</sub>O, nitrate, and ammonia will be measured to provide a quantitative estimate of net isotopic flux and N<sub>2</sub>O cycling processes. The combined use of microprofiling and multi-isotope approaches will provide not only detailed insight into N<sub>2</sub>O production and flux at these sites, but also yield data for a recently developed metabolic model to simulate and predict N<sub>2</sub>O dynamics under varying environmental conditions.

Broader Impacts: The research would strengthen the collaboration with German scientists. The proponents plan to create a webpage to discuss the technologies used in their project, as well as the activities taking place during their field trips. One postdoc and one undergraduate student from the University of Southern California would be supported and trained as part of this project.

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## Funding

Funding Source	Award
<a href="#">NSF Division of Ocean Sciences (NSF OCE)</a>	<a href="#">OCE-1259971</a>
<a href="#">NSF Division of Ocean Sciences (NSF OCE)</a>	<a href="#">OCE-1260373</a>

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