# Sediment Nitrification Incubation data of a subterrarnian estuary core take in July 2020 in Gloucester Point, Virginia

Website: https://www.bco-dmo.org/dataset/915302

**Data Type**: experimental

Version: 1

Version Date: 2023-12-04

#### **Project**

» <u>Collaborative Research: Cryptic nitrogen cycling in the anoxic subterranean estuary</u> (Subsurface cryptic N cycle)

Contributors	Affiliation	Role
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#### **Abstract**

These data are the result of a sediment slurry incubation experiment conducted with 15N-labeled ammonium to quantify nitrification rates in the sediments of a shallow, sandy subterranean estuary in Gloucester Point, VA, USA. Triplicate sediment cores that were 50cm in length were collected and sectioned into 10cm intervals. Homogenized sediment was incubated with porewater from the same depth interval that had been amended with 15N-labeled ammonium. Sediments were incubated in the dark for zero, six and twelve hours. At each time point, samples were collected to analyze dissolved inorganic nitrogen (nitrate, nitrite, and ammonium) concentrations. Samples were analyzed with an isotope ratio mass spectrometer in order to assess the 15N enrichment of the nitrate in each sample resulting in a delta value (d15N) that allows for the calculation of the mole fraction of 15N-labeled nitrate produced by each slurry. This production over time constitutes the sediment nitrification rate for each depth interval.

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#### Coverage

Spatial Extent: Lat:37.2489 Lon:76.5053

Temporal Extent: 2020-07-22

#### Methods & Sampling

These data were collected during July 2020 from a sandy subterranean estuary (STE) located in Gloucester Point, Virginia, USA. A pole-corer was used to collect triplicate sediment cores roughly 50cm in length from the mid tide line of the beach during low tide. Cores were sectioned into ten centimeter increments. Porewater was collected from surface water and piezometers placed at 10cm increments to the same depth of 50cm. Core sections were used in isotope tracer incubations with 15N-labeled substrates to measure potential nitrification

rates. Briefly, 10 g of the composited sediment from each 10 cm section were used in sediment slurry incubation experiments with 40mL of porewater from the same depth interval. Samples were incubated for 0, 6, and 12 hours in in 100mL HDPE bottles and amended with 5 atom% 15  $\mu$ M 15NH4+ (Cambridge Isotope) at the measured *in situ* temperature. After the incubation period, samples were spun down, porewater was decanted and filtered with a 0.45  $\mu$ m syringe filter (Whatman GE) and were frozen (-20 °C) until analysis. Porewater nutrient concentrations were analyzed for NOx (nitrate + nitrite), nitrite, and ammonium with a Lachat autoanalyzer. The isotopic enrichment of nitrate and nitrite products were measured using a gas bench isotope ratio mass spectrometer (IRMS, Delta V Plus, Thermo Fisher Scientific, Waltham, MA) using the bacterial reduction method. A culture of *Pseudomonas aureofaciens* reduced NOx in collected samples to N2O. The isotopic composition of the N2O was measured with isotope-ratio mass spectrometry (IRMS) (Sigman et al., 2001).

#### **Data Processing Description**

Instruments used: Lachat QuikChem 8000 automated ion analyzer Lachat In- struments, Milwaukee, WI, USA; detection limits for NO3-, NH4+, and PO43- are 0.20, 0.36, and 0.16  $\mu$ M, respectively. Gas bench isotope ratio mass spectrometer (IRMS) Delta V Plus, Thermo Fisher Scientific, Waltham, MA.

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#### **Data Files**

File

**915302\_v1\_nitrificationincubation.csv**(Comma Separated Values (.csv), 6.72 KB)

MD5:ec5ce543d4eac180e7d0b2363fdf5fe7

Primary data file for dataset ID 915302, version 1

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#### **Parameters**

Parameter	Description	Units
Date	Date of core collection	unitless
Site	Site of core collection name	unitless
Latitude	Latitude of location that the cores were collected, south is negative	
Longitude	Longitude of location that the cores were collected, west is negative	decimal degrees
Depth_Interval	Depth section of core collected (cores were section in ten centimeter increments)	centimeter (cm)
Replicate	Three replicate cores (A, B, C) were collected at the site	unitless
Incubation_Time_Point	Sediment slurry incubation time point (T0, T1, T2)	unitless
Incubation_Hours	Hours that sediment slurry was incubated	hours (hr)
Sample_ID	Sample identifier (Depth Interval_Replicate_Time Point of sample; BKGD PW = background sample of porewater collected, INCUB PW = porewater collected after ammonium amendment)	unitless
NOX	Nitrate + Nitrite concentration in micromoles per liter (uM) measured by a lachat autoanalyzer	micromoles per liter (uM)
NO3	Nitrate concentration in micromoles per liter (uM) measured by a lachat autoanalyzer	micromoles per liter (uM)
NO2	Nitrite concentration in micromoles per liter (uM) measured by a lachat autoanalyzer	micromoles per liter (uM)
NH4	Ammonium concentration in micromoles per liter (uM) measured by a lachat autoanalyzer	micromoles per liter (uM)
d15N	Delta 15N measurement made by an isotope ratio mass spectrometer	parts per thousand (‰)
Mole_Fraction_15N	15N Mole Fraction calculated from the d15N value	unitless

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# Instruments

Dataset- specific Instrument Name	Gas bench isotope ratio mass spectrometer
Generic Instrument Name	Isotope-ratio Mass Spectrometer
Dataset- specific Description	Gas bench isotope ratio mass spectrometer (IRMS) Delta V Plus, Thermo Fisher Scientific, Waltham, MA.
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	Lachat QuikChem 8000 automated ion analyzer
Generic Instrument Name	Lachat QuikChem 8000 flow injection analyzer and Ion Chromatography (IC) system
Dataset- specific Description	Lachat QuikChem 8000 automated ion analyzer Lachat In- struments, Milwaukee, WI, USA; detection limits for NO3—, NH4+, and PO43— are 0.20, 0.36, and 0.16 μM, respectively.
	The Lachat QuikChem 8000 can operate flow injection analysis and ion chromatography simultaneously and independently on the same instrument platform. Instrument includes sampler, dilutor, sampling pump, electronics unit, and data station. Analysis takes 20-60 seconds, with a sample throughput of 60-120 samples per hour. Measurements are in the range of parts per trillion to parts per hundred.

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## **Project Information**

# Collaborative Research: Cryptic nitrogen cycling in the anoxic subterranean estuary (Subsurface cryptic N cycle)

**Coverage**: Temperate (Mid-Atlantic), Sandy Beach along the York River Estuary, Gloucester Point, Virginia, USA (37.24884N/76.505324W)

#### NSF Award Abstract:

Nitrogen is an important nutrient that maintains high coastal ecosystem productivity. Yet excess nitrogen delivery can cause serious water quality deterioration including harmful algal blooms, fish kills, and oxygen free dead zones. Numerous nitrogen transformations regulate the balance between nitrogen delivery and nitrogen removal in coastal environments and the majority of these reactions occur in sediments where seawater passes through the subsurface and mixes with groundwater transported from uplands. This mixing zone, referred to as the subterranean estuary, is characterized by very different geochemistry than either the seawater above it or the groundwater below it. Thus, it has the potential to host a variety of unique reactions that affect nitrogen availability to the overlying water. Scientists from the College of William and Marv, Virginia Institute of Marine Science (VIMS), and the University of Connecticut (UConn) propose to examine the importance of a cryptic nitrogen cycle, a novel and potentially widespread nitrogen cycling process in the subterranean estuary. The cryptic nitrogen cycle comprises anoxic ammonium oxidation to nitrite (anoxic nitrification) coupled with anaerobic ammonium oxidation (anammox) or denitrification producing harmless dinitrogen gas. The proposed project represents highly transformative science because it has the potential to change the current paradigm detailing operation of the biogeochemical nitrogen cycle in anoxic environments. Occurrence of the cryptic nitrogen cycle would have broad implications for the nitrogen budget of terrestrial and groundwater systems and the coastal ocean. Characterization of the cryptic nitrogen cycle will allow us to better understand interactions among the nitrogen, metals, and sulfur cycles, and potential impacts of ongoing human modification of coastal environments. Educational contribution of this project focuses on graduate and undergraduate student training. Two graduate students at VIMS and UConn will receive interdisciplinary training in microbiology, molecular ecology, and biogeochemistry while several undergraduates recruited through the VIMS REU (Research Experience for Undergraduates) Program and the UConn marine science programs will also participate in the project. In addition, three summer undergraduate interns will be recruited from Hampton University, a historically Black college, and trained to enhance minority education and research in marine science. Public outreach will be achieved through popular venues such as VIMS Marine Science Day, and the VIMS After Hours Public Lecture Series at VIMS. Tobias at UConn also provides educational contributions and outreach efforts through the UConn Marine Scholars and Early College Experience programs and an exhibit at Mystic Aquarium.

A cryptic nitrogen cycle is proposed as a new process coupling anoxic nitrification to microbial nitrogen removal pathways such as anammox and denitrification. Unlike anammox, which refers to the oxidation of

ammonium by nitrite to form dinitrogen (N2) gas, anoxic nitrification occurs by oxidation of ammonium in the absence of oxygen using other common chemical oxidants such as metal oxides (namely, Fe and Mn) or sulfate, abundant in many marine and coastal systems. The thermodynamic favorability of these reactions relies on coupling nitrite formed via these oxidants with anammox or denitrification. Due to the coupling, nitrite will not accumulate or be measurable in anoxic marine systems. Thus, a cryptic N cycle responsible for nitrite production can occur as a novel N transforming process in anoxic environments, serve as a vital link to N2 production, and attenuate N loads discharging from a subterranean estuary (STE). Preliminary results from a STE in the York River Estuary located in Virginia showed substantial N2 production, representing removal of 50-75% of the fixed groundwater N, in ferruginous and sulfidic zones where neither nitrite nor nitrate were detectable. Stable isotope incubation experiments using the 15N tracer and molecular analysis of microbial communities suggest that coupled anoxic nitrification and anammox processes are the dominant N2 production pathways rather than canonical denitrification in the STE. Therefore, coupled anoxic nitrificationanammox in coastal groundwater may be a major unrecognized sink for fixed nitrogen at the land-sea interface. In addition to coastal groundwater, the cryptic N cycle has potential importance in anoxic zones and ocean basins. This proposal focuses on the STE because geochemical conditions there appear optimal for the proposed reactions to occur, and our preliminary data show strong evidence for a cryptic N cycle. The proposed work uses a combined geochemical, 15N isotope tracer and microbiological approach to evaluate environmental controls on the cryptic N cycle as well as to estimate its contribution to reduction of fixed N fluxes to the coastal ocean. Four approaches are proposed: (1) Field characterization of anoxic nitrification reactions and associated microbial communities in a subterranean estuary; (2) Laboratory incubation experiments to identify hotspots of the cryptic N cycle; (3) Controlled microcosm experiments to determine geochemical controls on anoxic nitrification; and (4) in situ assessment of anoxic nitrification to estimate the importance of the cryptic N cycle in a coastal aquifer.

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# **Funding**

Funding Source	Award
NSF Division of Ocean Sciences (NSF OCE)	OCE-1657801

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