

Dissolved iodine speciation depth profile measurements from Siders Pond, Falmouth, Massachusetts, USA, Jul 2020 to Jul 2021

Website: <https://www.bco-dmo.org/dataset/1000740>

Data Type: Other Field Results

Version: 1

Version Date: 2026-06-10

Project

» [Collaborative Research: Manganese Cycling and Coupling Across Redox Boundaries within Stratified Basins of the Baltic Sea](#) (MnIONS)

Contributors	Affiliation	Role
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Abstract

The Siders Pond samples were collected and filtered on four sampling days of July 17, September 19, November 6, 2020 and July 31, 2021. We specifically used iodine exchange chromatography to separate and individually measure each total dissolved iodine, dissolved iodate, dissolved iodide, and dissolved organic iodine with inductively coupled plasma mass spectrometry, and reported the resulting concentrations together with sampling metadata. The samples were collected by researchers at Woods Hole Oceanographic Institution and iodine measurements made at Michigan State University. The iodine measurements contribute to a temporal study of the cycling of redox-sensitive elements in the salinity-stratified pond, including reactions between multiple biogeochemical cycles and sources and sinks.

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Coverage

Location: Siders Pond, Falmouth, Massachusetts, USA

Spatial Extent: N:41.551793 E:-70.6193716 S:41.5465985 W:-70.6239537

Temporal Extent: 2020-07-17 - 2021-07-27

Methods & Sampling

The samples were collected at discrete depth intervals at a single location within the pond. The samples were collected at temporal intervals intended to understand seasonal changes in pond chemistry. Samples collected for iodine analyses were filtered at 0.2µm with Sterivex filters and collected in 125ml or 60ml amber Nalgene bottles and were frozen immediately afterwards for iodine preservation (Campos, 1997). Iodine speciation analyses occurred within 1 year of sampling, mostly much sooner.

Glass columns for separating iodine species were washed by ultra-pure water produced from the Milli-Q® water purification system, followed by packing with PYREX glass wool and 1ml of Bio-rad AG® 1-X8 anion exchange resin. During the chromatography procedure, the I⁻ fraction is specifically adsorbed onto the surface

of the resin and eluted following the addition of a 2M HNO₃ (Fisher Scientific, TraceMetal™ Grade)-18% (v/v) Tetramethylammonium hydroxide (TMAH, Fisher Scientific) solution. The eluent containing IO₃⁻ and DOI fractions were collected separately during the elution steps prior to the I⁻ collection, followed by an acidification (pH <2) and reduction step which converts iodine to I⁻ by the addition of concentrated hydrochloric acid (HCl, Fisher Scientific) and sodium metabisulfite (Na₂S₂O₅, VWR Analytical) (Hardisty et al., 2020). The treated eluent was left overnight for reaction and was processed through the same chromatographic procedure to collect the I⁻ derived from the IO₃⁻ and DOI fractions. For independent measurement of total dissolved iodine (TI) and as a quality control, a separate split of seawater samples was treated with HCl/ Na₂S₂O₅ to reduce iodine completely to I⁻ and subsequently processed through the chromatographic method. The eluents from chromatography were collected in Savillex® PFA Teflon vials and then diluted 20 times in 0.2M HNO₃-0.9% TMAH before instrumental analysis. The iodine concentrations were measured using a Thermo Scientific iCAP Q triple-quadrupole inductively coupled plasma mass spectrometer (ICP-MS-TQ) using Qtegra software version 2.10.3324.131 in both single-quad (SQ) and triple-quad (TQ) modes with O₂ reaction cell gas.

For quality control, a 200ppb I⁻ solution, which was gravimetrically diluted from the Inorganic Ventures® 1000±4ug/ml I⁻ ICP-MS standard solution in 1% tetraethyl ammonium (TEA), was processed through chromatography alongside the seawater samples to estimate the yield of I⁻, DOI and TI fractions. Similarly, for estimating the yield of IO₃⁻ reduction and column separation, a 200ppb KIO₃ solution dissolved from gravimetrically weighed solid KIO₃ powder (Fisher Science Education) was reduced and chromatographically processed. Yields from standards were applied to corrections for unknown samples. For monitoring contamination, ultra-pure water was processed through the columns. Among each set of seawater samples (11 samples) which were processed at the same time, one of them was randomly selected as a replicate.

The eluents from the chromatography were collected in Savillex PFA Teflon vials and then diluted 20 times (0.2M HNO₃-0.9% TMAH) before instrumental analysis. The iodine concentrations were measured using a Thermo Scientific iCAP Q triple-quadrupole inductively coupled plasma mass spectrometer (ICP-MS-TQ) using Qtegra software version 2.10.3324.131 in both single-quad (SQ) and triple-quad (TQ) mode with O₂ reaction cell gas. The samples were introduced into the ICP-MS via a Teledyne Cetac ASX-520 autosampler. We used 5ppb internal standard solution diluted from 1001±3ug/ml indium (In) in 2% HNO₃, 999±5ug/ml Rhodium (Rh) in 15% HCl, and 1000±4ug/ml Cesium 0.1% HNO₃ (Cs) elemental standards from Inorganic Ventures® to calibrate the intensity of iodine and to correct for instrumental drift during the analytical run. The internal standard was either spiked into each sample or was introduced into the ICP-MS after being mixed with sample within an inline mixing chamber. Detection limits for samples were determined as 10 nmol/kg iodine.

Data Processing Description

Data were processed using Excel.

BCO-DMO Processing Description

- Loaded data from "SidersPond_iodine_depth_profiles.xlsx" (sheet 1) treating all values as strings, with "", "nd", and "N/A" set as missing values
- Combined ISO_DateTime_collected_UTC (format %m-%d-%y) and UTC_Time (%H:%M) into a single ISO 8601 datetime field ISO_DateTime_collected_UTC (output format %Y-%m-%dT%H:%MZ, type datetime)
- Deleted UTC_Time column
- Extracted non-numeric values from iodate_concentration_nM and DOI_concentration_nM into new flag columns iodate_concentration_nM_flag and DOI_concentration_nM_flag; original columns set to None where non-numeric
- Renamed column Total_I_concentration_nM to Total_I_concentration_nM and depth_m to depth to remove units from parameter names to align with BCO-DMO naming guidance
- Output written to 1000740_v1_siders_pond_iodine.csv

Parameters

Parameter	Description	Units
sample_ID	Sample ID	unitless
station_number	Station number	unitless
depth	Depth of sample collection	meters
iodide_concentration_nM	Iodide concentration; 10 nanomole/kg detection limit	nanomole/kg
iodate_concentration_nM	Iodate concentration; 10 nanomole/kg detection limit	nanomole/kg
iodate_concentration_nM_flag	Flag indicating the iodate concentration is <10	unitless
DOI_concentration_nM	Dissolved organic iodine concentration; 10 nanomole/kg detection limit	nanomole/kg
DOI_concentration_nM_flag	Flag indicating the DOI concentration is <10	unitless
Total_I_concentration_nM	Total iodine concentration; 10 nanomole/kg detection limit	nanomole/kg
ISO_DateTime_collected_UTC	Datetime of sampling event, UTC	unitless
Latitude	Latitude of sample, positive is North	decimal degrees
Longitude	Longitude of sample, negative is West	decimal degrees

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Instruments

Dataset-specific Instrument Name	Teledyne Cetac ASX-520 autosampler
Generic Instrument Name	Laboratory Autosampler
Dataset-specific Description	The samples were introduced into the ICP-MS via a Teledyne Cetac ASX-520 autosampler.
Generic Instrument Description	Laboratory apparatus that automatically introduces one or more samples with a predetermined volume or mass into an analytical instrument.

Dataset-specific Instrument Name	Thermo Scientific iCAP Q triple-quadrupole inductively coupled plasma mass spectrometer (ICP-MS-TQ)
Generic Instrument Name	Thermo Fisher Scientific iCAP TQ inductively coupled plasma mass spectrometer
Dataset-specific Description	The iodine concentrations were measured using a Thermo Scientific iCAP Q triple-quadrupole inductively coupled plasma mass spectrometer (ICP-MS-TQ) using Qtegra software version 2.10.3324.131 in both single-quad (SQ) and triple-quad (TQ) modes with O ₂ reaction cell gas.
Generic Instrument Description	A benchtop triple quadrupole (TQ) inductively coupled plasma mass spectrometer (ICP-MS) with a four channel peristaltic pump, three plasma gas flow controllers, and four QCell mass flow controllers. The iCAP TQ utilises triple quadrupole technology which allows the analyte signal to be isolated from interferences, enabling the analysis of complex or high-matrix samples. The high frequency (4 MHz) quadrupole mass analyser has pre and post filters for isolation of wanted ions. The instrument features Peltier cooled high purity quartz or perfluoroalkoxy alkane (PFA), and low volume, baffled cyclonic or double pass spray chambers to efficiently filter out larger aerosol droplets for improved plasma stability. A reaction finder method development assistant aids easy method development. The plasma system is designed to rapidly adapt to changing matrices and provide robustness for challenging samples such as highly volatile organic solvents. The argon ICP ion source has a digital, solid state radiofrequency generator, and dynamic frequency impedance matching the plasma at 27 MHz. The iCAP TQ has applications in trace element analysis of solid or liquid (particularly sediment or sea water) samples. It has a nebuliser default flow rate of 400 μ L/min.

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Project Information

Collaborative Research: Manganese Cycling and Coupling Across Redox Boundaries within Stratified Basins of the Baltic Sea (MnIONS)

Coverage: Baltic Sea

NSF Award Abstract:

The trace element manganese (Mn) is distributed widely throughout the global ocean where it cycles among three dominant oxidation states. Manganese in the higher oxidation states is highly reactive and thereby influences the cycling of nearly all other elemental cycles, including those of oxygen and nitrogen. The intermediate Mn species has only recently become recognized as an abundant component of the Mn pool, presenting now a previously unrecognized factor that may control the chemistry of the ocean. The Baltic Sea contains high Mn concentrations and preliminary investigations have pointed to the presence of an operationally defined "reactive" form of Mn but the composition and consequence of this Mn pool are unknown. This research will explore the cycling of Mn within the Baltic Sea enabled by an established collaboration with the Leibniz Institute for Baltic Sea Research in Warnemunde, Germany. By coupling field measurements and targeted shipboard incubations, this study will shed light on the processes controlling the Mn cycle and its link to the oxygen, iodine, and nitrogen cycles. This project will educate several undergraduate and graduate students and promote scientific exchange between research groups within the United States and Germany. Further, outreach efforts associated with this research will continue an existing collaboration between the PIs and the Boston Green Academy in South Boston to introduce high school students to chemical oceanography, and in particular biogeochemistry.

Manganese (Mn) is intricately linked to nearly all elemental cycles, and yet we know little about the processes governing its redox cycling within natural systems. Over the past decade a number of key scientific discoveries have provided greater insight into the diversity of processes and mechanisms involved in Mn redox cycling and introduced Mn(III) ligand complexes as important components of the dissolved Mn pool. The Baltic Sea is one of the most well studied stratified marine systems and reactive Mn has been implicated as a key factor in the formation and maintenance of suboxic zones. Thus, the goal of this research is to explore the cycling and

elemental coupling of Mn within stratified basins of the Baltic Sea. The PIs predict that reactive Mn, as Mn(III) ligand complexes and Mn oxide particles, is a primary control on the redox landscape of stratified marine waters, particularly at redox boundaries and within the suboxic zone. The PIs propose fieldwork in a local permanently stratified brackish pond to refine experimental procedures followed by two cruises to suboxic basins in the Baltic Sea enabled by an established collaboration with the Leibniz Institute for Baltic Sea Research in Warnemunde. Field measurements will be obtained using a combination of in situ sensors and ship/lab-based instrumentation at several Baltic Sea sites to define the distribution of Mn species and the rates of Mn redox transformations spanning the redoxcline along with a suite of chemical information. Further, a matrix of shipboard incubations will be conducted to constrain the underlying (a)biotic processes responsible for the observed Mn profiles. Specifically, across oxygen and Mn gradients spanning the redoxcline, the PIs will interrogate the link between the Mn cycle and iodine and nitrogen species, which will ultimately help constrain current gaps in the mass balance of these elements in Baltic Sea models.

This award reflects NSF's statutory mission and has been deemed worthy of support through evaluation using the Foundation's intellectual merit and broader impacts review criteria.

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Funding

Funding Source	Award
NSF Division of Ocean Sciences (NSF OCE)	OCE-1924236

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