

# FT-ICRMS data from water samples collected at various sites around Puerto Rico during a Sargassum inundation event from May to June 2018

**Website:** <https://www.bco-dmo.org/dataset/1010617>

**Data Type:** Other Field Results

**Version:** 1

**Version Date:** 2026-10-22

## Project

» [Collaborative Research: Phlorotannins - An Important Source of Marine Chromophoric Dissolved Organic Matter?](#) (Sargassum DOM)

| Contributors                     | Affiliation                            | Role                   |
|----------------------------------|----------------------------------------|------------------------|
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| <a href="#">Powers, Leanne</a>   | Chesapeake Biological Laboratory (CBL) | Scientist              |

## Abstract

To compliment our work on Sargassum dissolved organic matter obtained during exudation experiments, we aimed to better understand the fate of Sargassum exudates and lysates in the environment. We sampled a Sargassum inundation event from 30 May to 6 June 2018 in Laguna Grande, Fajardo, Puerto Rico and other areas near Fajardo and Salinas, Puerto Rico. Laguna Grande is a 50-ha bioluminescent lagoon located in Las Cabezas de San Juan Nature Reserve in Fajardo, Puerto Rico. Sargassum had accumulated in the lagoon prior to sampling and dissolved oxygen (DO) was ~15 mg/L on May 30. On June 2, the lagoon was hypoxic (2.5 mg/L DO in the lagoon and 0.6 mg/L DO in the lagoon channel). From June 3 to 6, Sargassum had died (visible on seafloor) or was physically removed by humans and by June 6 the lagoon began to recover (4.5 mg/L DO). We also collected samples at a beach within the nature reserve and at Las Croabas, a beach adjacent to the lagoon channel where a dense accumulation of Sargassum was contained behind a barrier. Finally, we sampled a floating Sargassum mat in the coastal waters near Salinas, Puerto Rico. In all cases, we collected water samples underneath or at the mat's edge and at least ~15 m away from the mat. Filtered water samples were analyzed for dissolved organic carbon (DOC) and total dissolved nitrogen (TDN) concentrations directly. The remaining sample underwent solid phase extraction (SPE). Aliquots of these SPE methanolic extracts were dried and redissolved in ultrapure water for analysis of dissolved organic sulfur (DOS), -phosphorus (DOP), -bromine (DOBr), and -iodine (DOI) by inductively coupled plasma-mass spectrometry (ICP-MS). Additional aliquots of methanolic extracts dissolved in methanol were analyzed by Fourier transform-ion cyclotron mass spectrometry (FT-ICR MS).

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## Coverage

**Location:** Coastal waters of Puerto Rico

**Spatial Extent:** N:18.5868 E:-65.5708 S:17.8476 W:-67.3709

**Temporal Extent:** 2018-05-30 - 2018-06-06

## Methods & Sampling

### Sample collection

Grab water samples (surface, ~0m) were collected dockside or by small boat in 10-L low-density polyethylene cubitainers that had been pre-cleaned with 0.1 M NaOH for 24 h followed by several rinses of ultrapure Milli-Q water. Within 3 h of collection, samples for DOC and TDN analysis were 0.2- $\mu$ m filtered through cellulose acetate filters into 40-mL combusted amber glass vials. Single samples for DOC/TDN were collected at each site, unless otherwise noted. The remaining samples were 0.7  $\mu$ m-filtered through pre-combusted Whatman GF/F glass fiber filters into clean combusted 1-L bottles. SPE samples were collected in duplicate or triplicate. The 0.7  $\mu$ m-filtered samples were then acidified to pH 2 using ultrapure concentrated HCl and underwent solid phase extraction (SPE). *Sargassum* exudates were generated by placing fragments of *Sargassum* in clear 500-mL Teflon bottles with seawater collected on the Atlantic shore with no visible *Sargassum*. Bottles were placed in a cooler with running water to maintain ambient temperature for 5 h. Seawater in bottles without *Sargassum* served as a control. After 5 h, exudates and the control were 0.7  $\mu$ m-filtered as above and also underwent SPE.

### Solid phase extraction (SPE)

We utilized 1 g Agilent Bond Elut PPL cartridges for SPE. Cartridges were activated with 10 mL ultrapure methanol and rinsed with 10 mL ultrapure 0.1% formic acid water prior to extraction. Samples were then loaded onto cartridges through clean Teflon tubing and extracted by gravity (final extraction volumes ranged from 0.4 to 1.2 L). After extraction, cartridges were rinsed with 30 mL 0.1% formic acid water and dried. Cartridges were then eluted in 10 mL ultrapure MeOH and these methanolic extracts were stored at -20 °C until ICP-MS and FT-ICR MS analysis.

### ICP-MS

Dissolved organic compound (DOS, DOP, DOBr, DOI) analysis followed the methods described in Gonsior et al. (2024) (doi:10.1039/D3VA00361B) for DOS and DOP and in Powers et al. (2024) (doi: 10.1016/j.scitotenv.2023.169292) and Powers et al. (2026) (doi: 10.1029/2025GL119062) for dissolved organic bromine (DOBr) and dissolved organic iodine (DOI). Briefly, methanolic extract aliquots were dried under a gentle stream of nitrogen gas and redissolved in ultrapure water. Samples for DOBr and DOI analysis were also passed through silver cartridges prior to analysis to remove residual halides. DOS and DOP were analyzed by ICP-MS/MS using oxygen as the reaction gas and DOBr and DOI were analyzed by ICP-MS/MS with no gas in the Octopole Reaction System. Final concentrations were corrected for both extraction and dilution volumes. Reported values are the average and standard deviation of three replicate injections per sample.

### BCO-DMO Curation Notes

Curation Actions Performed on Data File

- Loaded 13 XLSX files (sheet 1, header row 1, formatting preserved); empty strings and “nd” were treated as missing values. Each table was named according to its source file.
- Concatenated the 13 tables into 1010617\_v1\_sargassum\_inundation\_event\_ft-icrms\_data, mapping the shared columns and adding original\_source\_file\_name to identify each source file.
- Cleaned original\_source\_file\_name by replacing underscores with spaces, removing the “.xlsx” extension, and removing six-digit date strings.
- Renamed original\_source\_file\_name to Collection\_site\_description.
- Reordered the columns as follows: Sample\_ID, Date, lat\_dec, long\_dec, Collection\_site\_description, mz, actual\_mass, Int\_rep1, Int\_rep2, H, C, O, N, S, Br, OC, HC, DBE, Int\_rep3.
- Set column types: Sample\_ID and Collection\_site\_description as strings; Date as a date formatted %Y-%m-%d; lat\_dec, long\_dec, mz, actual\_mass, HC, and OC as numbers; and Int\_rep1, Int\_rep2, Int\_rep3, H, C, O, N, S, Br, and DBE as integers.
- Updated column metadata, including descriptions, BCO-DMO standard name IDs, and supplied units. Lat\_dec and long\_dec were flagged as primary parameters.
- Saved the final table as 1010617\_v1\_sargassum\_inundation\_event\_ft-icrms\_data.csv.

## Problem Description

No problems or issues have been reported for this dataset by the dataset authors.

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## Related Publications

Gonsior, M., Lahm, M., Powers, L., Chen, F., McCallister, S. L., Liang, D., Guinan, G., & Schmitt-Kopplin, P. (2024). Optical properties and molecular differences in dissolved organic matter at the Bermuda Atlantic and Hawai'i ALOHA time-series stations. *Environmental Science: Advances*, 3(5), 717–731.

<https://doi.org/10.1039/d3va00361b> <https://doi.org/10.1039/D3VA00361B>

*Methods*

Powers, L. C., Schijf, J., Schmitt-Kopplin, P., & Gonsior, M. (2025). Halogenated Organic Compounds: A Massive Halogen Reservoir and an Intriguing Component of the Marine Dissolved Organic Matter Pool. *Geophysical Research Letters*, 53(1). Portico. <https://doi.org/10.1029/2025gl119062>

<https://doi.org/10.1029/2025GL119062>

*Methods*

Powers, L. C., Schmitt-Kopplin, P., & Gonsior, M. (2024). Evaluating the photochemical reactivity of disinfection byproducts formed during seawater desalination processes. *Science of The Total Environment*, 912, 169292.

<https://doi.org/10.1016/j.scitotenv.2023.169292>

*Methods*

Tziotis, D., Hertkorn, N., & Schmitt-Kopplin, Ph. (2011). Kendrick-Analogous Network Visualisation of Ion Cyclotron Resonance Fourier Transform Mass Spectra: Improved Options for the Assignment of Elemental Compositions and the Classification of Organic Molecular Complexity. *European Journal of Mass Spectrometry*, 17(4), 415–421. <https://doi.org/10.1255/ejms.1135>

*Methods*

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## Parameters

| Parameter                   | Description                                                                                                                           | Units           |
|-----------------------------|---------------------------------------------------------------------------------------------------------------------------------------|-----------------|
| Sample_ID                   | Unique identifier for the water or Sargassum exudate sample analyzed by FT-ICR MS.                                                    | unitless        |
| Date                        | Date on which the sample was collected.                                                                                               | unitless        |
| lat_dec                     | Latitude of the sample collection location in decimal degrees. Positive values indicate north latitude.                               | decimal degrees |
| long_dec                    | Longitude of the sample collection location in decimal degrees. Negative values indicate west longitude.                              | decimal degrees |
| Collection_site_description | Sampling site and collection context, including the site name and whether the sample was collected inside or outside a Sargassum mat. | unitless        |
| mz                          | Mass-to-charge ratio of an ion detected by FT-ICR MS.                                                                                 | Dalton          |

|             |                                                                                        |          |
|-------------|----------------------------------------------------------------------------------------|----------|
| actual_mass | Calculated exact mass of an ion based on its assigned molecular formula.               | Dalton   |
| Int_rep1    | Ion intensity measured for replicate FT-ICR MS analysis 1.                             | count    |
| Int_rep2    | Ion intensity measured for replicate FT-ICR MS analysis 2.                             | count    |
| H           | Hydrogen-related value reported for the molecular feature detected by FT-ICR MS.       | count    |
| C           | Carbon-related value reported for the molecular feature detected by FT-ICR MS.         | count    |
| O           | Oxygen-related value reported for the molecular feature detected by FT-ICR MS.         | count    |
| N           | Nitrogen-related value reported for the molecular feature detected by FT-ICR MS.       | count    |
| S           | Sulfur-related value reported for the molecular feature detected by FT-ICR MS.         | count    |
| Br          | Bromine-related value reported for the molecular feature detected by FT-ICR MS.        | count    |
| OC          | Oxygen-to-carbon ratio reported for the molecular feature detected by FT-ICR MS.       | unitless |
| HC          | Hydrogen-to-carbon ratio reported for the molecular feature detected by FT-ICR MS.     | unitless |
| DBE         | Double bond equivalent value reported for the molecular feature detected by FT-ICR MS. | unitless |
| Int_rep3    | Ion intensity measured for replicate FT-ICR MS analysis 3.                             | count    |

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## Instruments

|                                         |                                                                                                                                                                                                                                                                                                                                                  |
|-----------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <b>Dataset-specific Instrument Name</b> | Agilent 8900 ICP-QQQ triple-quadrupole inductively coupled plasma mass spectrometer                                                                                                                                                                                                                                                              |
| <b>Generic Instrument Name</b>          | Mass Spectrometer                                                                                                                                                                                                                                                                                                                                |
| <b>Dataset-specific Description</b>     | We utilized 1 g Agilent Bond Elut PPL cartridges for SPE. Cartridges were activated with 10 mL ultrapure methanol and rinsed with 10 mL ultrapure 0.1% formic acid water prior to extraction. Samples were then loaded onto cartridges through clean Teflon tubing and extracted by gravity (final extraction volumes ranged from 0.4 to 1.2 L). |
| <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> | Bruker Solarix 12 Tesla FT-ICR mass spectrometer                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |
| <b>Generic Instrument Name</b>          | Mass Spectrometer                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |
| <b>Dataset-specific Description</b>     | Mass spectrometric analyses were undertaken at the Helmholtz Zentrum, Munich, Germany using a Bruker Solarix 12 Tesla Fourier transform ion cyclotron resonance mass spectrometer (FT-ICR MS) using negative electrospray ionization at -3600 V. The flow rate was 120 µL/h, 500 spectra were averaged. Pre-calibration was performed with arginine clusters and spectra were post-calibrated in the Bruker Data Analysis software using known DOM m/z ions across the m/z range of 200-700, with signal-to-noise set to 10. |
| <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 Total Carbon Analyzer (TOC-Vcph)                                                                                                                                                                                                                                                                                                     |
| <b>Generic Instrument Name</b>          | Total Organic Carbon Analyzer                                                                                                                                                                                                                                                                                                                 |
| <b>Dataset-specific Description</b>     | DOC and TDN concentrations were measured using a Shimadzu TOC-V.                                                                                                                                                                                                                                                                              |
| <b>Generic Instrument Description</b>   | A unit that accurately determines the carbon concentrations of organic compounds typically by detecting and measuring its combustion product (CO <sub>2</sub> ). See description document at: <a href="http://bcodata.whoi.edu/LaurentianGreatLakes_Chemistry/bs116.pdf">http://bcodata.whoi.edu/LaurentianGreatLakes_Chemistry/bs116.pdf</a> |

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## Project Information

### Collaborative Research: Phlorotannins - An Important Source of Marine Chromophoric Dissolved Organic Matter? (Sargassum DOM)

**Coverage:** Mid-Atlantic Bight (July 2016), Sargasso Sea (July and September 2016), Coastal Bermuda (September/October 2016) and Coastal Puerto Rico (Laguna Grande, Fajardo; Las Croabas, Fajardo; Salinas; May/June 2018)

## NSF Award Abstract:

Chromophoric dissolved organic matter (CDOM), the sunlight absorbing components in filtered water, is important in the study of marine and freshwater ecosystems as it can be used to trace the mixing of surface waters, as a proxy for carbon cycles, and other biogeochemical processes. Although its importance in ocean studies has been firmly established over the last several decades, sources and structural composition of CDOM within the oceans remains unclear and continues to be a subject of debate. Sargassum, a brown alga, is widely distributed in temperate and subtropical marine waters and may be important source of CDOM to the Sargasso Sea and Gulf of Mexico where Sargassum is abundant. This project will investigate the contribution of macro brown algae-derived compounds to the marine CDOM pool. Results from this study will have implications for the marine carbon cycle and satellite remote sensing of ocean color to assess mixing of surface water masses and biogeochemical processes. The project will provide educational opportunities for a postdoctoral scholar, summertime undergraduate internships (through a local NSF-sponsored Research Experiences for Undergraduates (REU) program), and workshop and research opportunities for local high schools students.

Sources of marine CDOM remain debatable and a comprehensive understanding of its origins, distribution and fate have been difficult. Marine CDOM, and in particular the "humic-like" component, have been suggested to originate from terrestrial sources, primarily lignins. However, recent evidence indicates that the exudation of phlorotannins produced by macro brown algae may contribute significantly to the marine CDOM pool. Phlorotannins, a class of polyphenols that are only found in, and continuously exuded by macro brown algae such as Sargassum, strongly absorb ultraviolet light and may have been underestimated in their contribution to the marine CDOM pool within certain geographic locales. Upon partial oxidation, light absorption by these specific compounds extends into longer wavelengths in the visible creating an absorption spectrum similar to that of lignin. These phlorotannins and their transformation products absorb light that might explain in part the "humic-like" signatures observed in open ocean environments. This study aims to characterize the optical properties and molecular composition of Sargassum-derived CDOM including its aerobic oxidation and photochemical behavior, as well as quantify Sargassum-derived CDOM to better estimate its possible contribution to the CDOM pool in the Sargasso Sea and Gulf of Mexico.

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## Funding

| Funding Source                                           | Award                       |
|----------------------------------------------------------|-----------------------------|
| <a href="#">NSF Division of Ocean Sciences (NSF OCE)</a> | <a href="#">OCE-1536888</a> |
| <a href="#">NSF Division of Ocean Sciences (NSF OCE)</a> | <a href="#">OCE-1536927</a> |

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