# Polyphosphate and associated measurements from water and particulate samples collected from 13 locations in the Great Lakes in July-Aug 2018 on R/V Blue Heron cruise BH18-09

Website: https://www.bco-dmo.org/dataset/920878 Data Type: Cruise Results Version: 1 Version Date: 2024-03-17

#### Project

» <u>Ecosystem-scale responses of coupled carbon and nutrient cycles to dramatic shifts in benthic communities:</u> <u>The Upper Great Lakes</u> (Great Lakes CNP cycles)

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#### Abstract

Water and particulate samples were collected from 13 locations in the Great Lakes to quantify polyphosphate (polyP) under a range of phosphorus (P) levels. The dataset also includes other related parameters, including particulate phosphorus (PP), chlorophyll-a (ChI-a), activities of alkaline phosphatase (APase), soluble reactive phosphorus (SRP), particulate carbon (PC), and particulate N (PN). Ratios of polyP:PP reported in the literature are also included for comparison. The supplement files include CTD data (temperature, oxygen, ChI-a, and light transmission) and the phytoplankton abundance data. Phytoplankton data are originally from the Great Lakes Environmental Database (GLENDA) collected by the US Environmental Protection Agency Great Lakes National Program Office (EPA GLNPO).

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Location: Lake Superior, Lake Michigan, and Lake Huron covering pelagic (oligotrophic) to nearshore (mesotrophic) regions (water depth 35- 258 m) Spatial Extent: N:47.5562 E:-83.4033 S:43.8812 W:-91.3108 Temporal Extent: 2018-07-30 - 2018-08-10

#### Methods & Sampling

Water samples were collected using Rosette Niskin bottles attached to a Seabird 911plus conductivitytemperature-depth probe (CTD), which also profiled temperature, oxygen, light transmission, and chlorophyll (ChI-a) concentrations. Suspended particles in the water were collected by filtering water through various sizes of filters (0.2  $\mu$ m and 2.0  $\mu$ m polycarbonate membrane filters and 0.7 um GF/F). Particulate carbon and nitrogen (PC and PN) were analyzed by using an Elemental Analyzer on the particles collected on the 0.7  $\mu$ m GFF that have been pre-combusted at 550°C for 6 hours. Particulate phosphorus (PP) was quantified using the persulfate digestion and molybdenum blue method (Suzumura 2008, Grasshoff et al. 1999). Soluble reactive P (SRP) in the water column was also measured using the molybdenum blue method (Grasshoff et al. 1999).

Chl-a was extracted from the 0.2  $\mu$ m filters using 90% acetone for 12 hours at 4°C in the dark after homogenization and sonication, and determined for Chl-a fluorometrically at an excitation wavelength of 430 nm and an emission wavelength of 663 nm (APHA 1998). Alkaline phosphatase activity (APase) was determined using a fluorogenic substrate 3-O-methylfluorescein phosphate (MUF-P) (Martin et al. 2018). PolyP in the particles was extracted using an enzyme digestion and boiling method for marine plankton (Martin and Van Mooy 2013). Extracted PolyP was stained using 4',6-diamidino-2-phenylindole (DAPI) and fluorometrically quantified (Aschar-Sobbi et al. 2008; Diaz et al. 2010). We also used a polyP-specific dye JC-D7 (InvivoChem, USA, # V22869, 87% purity) to quantify polyP (Angelova et al. 2014): the polyP extracts were stained using JC-D7 (25  $\mu$ mol L-1) dissolved in 5% DMSO buffered with HEPES solution (pH = 7.4, 25 mmol L-1), and quantified for polyP concentrations by measuring fluorescence intensity at an excitation wavelength of 405 nm and an emission wavelength of 535 nm. The colorimetric and fluorescence measurements were conducted using a BioTek microplate reader.

PolyP data from other aquatic ecosystems (lakes and oceans) reported in the literature are also included in the dataset for comparison (Diaz et al. 2016, Hashihama et al. 2020, Li et al. 2019, Martin et al. 2014, Martin et al. 2018). The literature data were obtained by digitizing the figures in the original papers and can only be considered approximations (except for Li et al. 2019, for which the original values are reported).

The data for phytoplankton abundance were originally reported in the Great Lakes Environmental Database (GLENDA) by the US Environmental Protection Agency Great Lakes National Program Office (EPA GLNPO; <a href="https://cdx.epa.gov/">https://cdx.epa.gov/</a>; Barbiero et al. 2018). In the GLENDA dataset, phytoplankton were identified to species or the lowest practical taxonomic level, counted, and estimated for biovolume (µm<sup>3</sup> mL-1) for each identified species using the Utermöhl technique and the EPA Standard Operating Procedure for Phytoplankton Analysis (LG401). We extracted data from stations near our sites and only analyzed the data collected in the summer (August) for the past five years (2014-2019). Biovolume and biovolume per individual cell were averaged for each species identified. The GLENDA phytoplankton data were collected as part of a grant to Euan Reavie from the U.S. Environmental Protection Agency under Cooperative Agreements GL-00E23101 and GL-00E0198. Our data treatment and this document have not been subjected to the EPA's required peer and policy review and do not reflect the view of the Agency, so no official endorsement should be inferred.

#### **Data Processing Description**

The literature data reported here were obtained by digitizing the figures in the original papers using MATLAB R2020 and can only be considered approximations (except for Li et al. 2019, for which the original values are reported). In brief, data in scatter plots were digitized by referencing the the pixel locations of the data points to those of the coordinates (e.g., x and y axes) using linear interpolation; Values associated with color (e.g, data in colormaps) were obtained by referencing the RGB values of the data points to those of the colorbar using least square approximation.

#### **BCO-DMO Processing Description**

Imported data from source file "GreatLakes\_PolyP.xlsx" into the BCO-DMO data system. - Corrected latitude and longitude values and site names Imported supplemental file "GreatLakes\_CTD.xlsx"

- Added sampling dates based on Site ID
- Renamed S-240 to S232 as they are same site location

Imported supplemental file "GreatLakes\_Phytoplankton.xlsx"

- Rounded Biovolume and Biovolume\_perind values to 2 decimal places

- Taxonomic names in the dataset were checked using the World Register of Marine Species (WoRMS) taxa match tool. Added WoRMS information (including AphiaID and source) to phytoplankton data.

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# **Data Files**

#### File

Primary data: Great Lakes Polyphosphate data

filename: 920878\_v1\_great\_lakes\_polyphosphate.csv(Comma Separated Values (.csv), 19.40 KB) MD5:1e08054071b94f00ba1a72ad141c95da

Primary data file for dataset ID 920878, version 1

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# Supplemental Files

#### File

#### Supplemental\_files\_parameter\_descriptions.pdf

Parameter descriptions for Tables S1 and S2 (supplement to dataset 920878)

#### Table S1: Great Lakes CTD data

filename: 920878\_supplement\_great\_lakes\_ctd.csv

Supplemental CTD data for dataset ID 920878, version 1  $\,$ 

Site\_ID, Sample site identification, unitless Latitude, Sample site latitude, unitless Longitude, Sample site longitude, unitless Date, Date of sampling, YYYY-MM-DD Depth, Depth of sample collection, meters (m) Temp, Temperature, degrees Celsius (°C) Chl\_a, Chl-a measured by CTD, microgram per liter (ug L-1) O2, Dissolved oxygen, milligram per liter (mg L-1) Light, Light transmission, percent (%)

#### Table S2: Great Lakes Phytoplankton data

filename: 920878\_supplement\_great\_lakes\_phytoplankton.csv

Supplemental phytoplankton data for dataset ID 920878, version 1

Division, Division reported in GLENDA, unitless Genus, Genus reported in GLENDA, unitless Species, Species reported in GLENDA, unitless Site\_ID, description, unitless GLENDA\_site\_ID, Site identification for matching data in the Great Lakes Environmental Database (GLENDA), unitless Latitude, Sample site latitude, decimal degrees Longitude, Sample site longitude, decimal degrees Sample\_Type, Sample type where INT= Summer integrated epilimnion sample (2 to 4 depths in the epilimnion); DCL= sample of the summer deep chlorophyll layer, when present, unitless Biovolume\_perind, Biovolume of phytoplankton, square micrometer per millimeter (um3/mL) Biovolume\_perind, Biovolume per cell, square micrometer per millimeter (um3/mL) Scientific\_Name\_WoRMS, Scientific name in the World of Register of Marine Species (WoRMS): "ambiguous"=multiple choices were found; "not found = no match was found, unitless

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(Portable Document Format (.pdf), 190.45 KB) MD5:ce21493aae415e4624676bded0d2653a

> (Comma Separated Values (.csv), 155.22 KB) MD5:785806edf1f51d067693cd2a735bc414

> (Comma Separated Values (.csv), 579.45 KB) MD5:475d7f67477dbc83188d673cd2512b9f

# **Related Publications**

APHA (1998) Standard Methods for the Examination of Water and Wastewater. (20th Edition). American Public Health Association, American Water Works Association and Water Environmental Federation, Washington DC. https://isbnsearch.org/isbn/978-0875532998 Methods

Angelova, P. R., Agrawalla, B. K., Elustondo, P. A., Gordon, J., Shiba, T., Abramov, A. Y., Chang, Y.-T., & Pavlov, E. V. (2014). In Situ Investigation of Mammalian Inorganic Polyphosphate Localization Using Novel Selective Fluorescent Probes JC-D7 and JC-D8. ACS Chemical Biology, 9(9), 2101–2110. https://doi.org/<u>10.1021/cb5000696</u> *Methods* 

Aschar-Sobbi, R., Abramov, A. Y., Diao, C., Kargacin, M. E., Kargacin, G. J., French, R. J., & Pavlov, E. (2008). High Sensitivity, Quantitative Measurements of Polyphosphate Using a New DAPI-Based Approach. Journal of Fluorescence, 18(5), 859–866. https://doi.org/<u>10.1007/s10895-008-0315-4</u> *Methods* 

Barbiero, R. P., Lesht, B. M., Hinchey, E. K., & Nettesheim, T. G. (2018). A brief history of the U.S. EPA Great Lakes National Program Office's water quality survey. Journal of Great Lakes Research, 44(4), 539–546. https://doi.org/<u>10.1016/j.jglr.2018.05.011</u> *Methods* 

Diaz, J. M., Björkman, K. M., Haley, S. T., Ingall, E. D., Karl, D. M., Longo, A. F., & Dyhrman, S. T. (2015). Polyphosphate dynamics at Station ALOHA, North Pacific subtropical gyre. Limnology and Oceanography, 61(1), 227–239. Portico. https://doi.org/<u>10.1002/lno.10206</u> *Methods* 

Grasshoff, K., Kremling, K., & Ehrhardt, M. (Eds.). (1999). Methods of Seawater Analysis. doi:<u>10.1002/9783527613984</u> *Methods* 

Hashihama, F., Saito, H., Shiozaki, T., Ehama, M., Suwa, S., Sugiyama, T., Kato, H., Kanda, J., Sato, M., Kodama, T., Yamaguchi, T., Horii, S., Tanita, I., Takino, S., Takahashi, K., Ogawa, H., Boyd, P. W., & Furuya, K. (2020). Biogeochemical Controls of Particulate Phosphorus Distribution Across the Oligotrophic Subtropical Pacific Ocean. Global Biogeochemical Cycles, 34(9). Portico. https://doi.org/<u>10.1029/2020gb006669</u> *Methods* 

Li, J., Plouchart, D., Zastepa, A., & Dittrich, M. (2019). Picoplankton accumulate and recycle polyphosphate to support high primary productivity in coastal Lake Ontario. Scientific Reports, 9(1). https://doi.org/<u>10.1038/s41598-019-56042-5</u> *Methods* 

Martin, P., & Van Mooy, B. A. S. (2013). Fluorometric Quantification of Polyphosphate in Environmental Plankton Samples: Extraction Protocols, Matrix Effects, and Nucleic Acid Interference. Applied and Environmental Microbiology, 79(1), 273–281. https://doi.org/<u>10.1128/aem.02592-12</u> *Methods* 

Martin, P., Dyhrman, S. T., Lomas, M. W., Poulton, N. J., & Van Mooy, B. A. S. (2014). Accumulation and enhanced cycling of polyphosphate by Sargasso Sea plankton in response to low phosphorus. Proceedings of the National Academy of Sciences, 111(22), 8089–8094. https://doi.org/<u>10.1073/pnas.1321719111</u> *Methods* 

Martin, P., Lauro, F. M., Sarkar, A., Goodkin, N., Prakash, S., & Vinayachandran, P. N. (2018). Particulate polyphosphate and alkaline phosphatase activity across a latitudinal transect in the tropical Indian Ocean. Limnology and Oceanography, 63(3), 1395–1406. Portico. https://doi.org/<u>10.1002/lno.10780</u> *Methods* 

# Methods

Suzumura, M. (2008). Persulfate chemical wet oxidation method for the determination of particulate phosphorus in comparison with a high-temperature dry combustion method. Limnology and Oceanography: Methods, 6(11), 619–629. doi:<u>10.4319/lom.2008.6.619</u> *Methods* 

U.S. EPA. GLNPO. 2021. Standard Operating Procedure for Phytoplankton Analysis (LG401). Version 07, March 2021. Retrieved March 4th, 2024 from <u>https://www.epa.gov/system/files/documents/2021-12/lg401.v07-phytoplankton-analysis\_rfa.pdf</u>

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# Parameters

Parameter	Description	Units
Site_ID	Sample site identification	unitless
Latitude	Sample site latitude	decimal degrees
Longitude	Sample site longitude	decimal degrees
Date	Date of sampling collection	unitless
GLENDA_site	Site identification for matching data in the Great Lakes Environmental Database (GLENDA)	unitless
Total_Depth	Total water depth of the sample site	meters (m)
Sample_Depth	Depth where the sample was taken	meters (m)
APase	Activity of alkaline phosphatase	nanomole per hour per litre (nmol h-1 L-1)
SRP	Soluble reactive phosphorus	nanomolar (nM)
Chl_a	Chlorophyll a	microgram per litre (ug L-1)
Filter_size	The size of filters used to collect the particulate samples	micrometer (um)
РР	Particulate phosphorus	nanomole per litre (nmol L-1)
PolyP_DAPI	PolyP measured by using DAPI method	equivalent nanomolar (e.q. nM)
PolyP_JCD7	PolyP measured by using polyP specific dye JC-D7	nanomolar (nM)
PC	Particulate carbon	micromolar (uM)
PN	Particulate nitrogen	micromolar (uM)
ratio_PolyP_to_PP_JCD7	The ratio of polyP to particulate P; polyP was quantified using polyP specific dye JC-D7	unitless
ratio_PolyP_to_PP_DAPI	The ratio of polyP to particulate P; polyP was quantified using DAPI and the results are relative estimates	unitless
Reference	Data source	unitless
Region	Sampling region	unitless

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# Instruments

Dataset- specific Instrument Name	Seabird 911plus conductivity-temperature-depth probe
Generic Instrument Name	CTD Sea-Bird SBE 911plus
Dataset- specific Description	Water samples were collected using Rosette Niskin bottles attached to a Seabird 911plus conductivity-temperature-depth probe (CTD)
Generic Instrument Description	The Sea-Bird SBE 911 plus is a type of CTD instrument package for continuous measurement of conductivity, temperature and pressure. The SBE 911 plus includes the SBE 9plus Underwater Unit and the SBE 11plus Deck Unit (for real-time readout using conductive wire) for deployment from a vessel. The combination of the SBE 9 plus and SBE 11 plus is called a SBE 911 plus. The SBE 9 plus uses Sea-Bird's standard modular temperature and conductivity sensors (SBE 3 plus and SBE 4). The SBE 9 plus CTD can be configured with up to eight auxiliary sensors to measure other parameters including dissolved oxygen, pH, turbidity, fluorescence, light (PAR), light transmission, etc.). more information from Sea-Bird Electronics

Dataset- specific Instrument Name	CHNS Elemental Analyzer (Vario EL Cube, Elementar)
Generic Instrument Name	Elementar Vario EL Cube elemental analyzer
Dataset- specific Description	Particulate carbon and nitrogen (PC and PN) were analyzed by using an Elemental Analyzer
Generic Instrument Description	A laboratory instrument used for quantifying organic elements. It can measure C, H, N and S and optionally O, Cl and TIC. It was first developed in 2006 as a successor to the vario EL III. It uses a high-temperature combustion unit that is able to complete sample digestion at up to 1200 deg C (or 1800 deg C at the point of combustion when tin foil is used) and a jet injection of oxygen directly to the sample during combustion. Separation of gas components are performed on up to 3 gas-selective columns which trap gases until they are heated up and the prior gas peak has reached the baseline during detection. It uses a Thermal Conductivity Detector (TCD) as standard. An infrared (IR) detector for sulfur and oxygen and electrochemical detector for chlorine are optionally available. The instrument can measure C / N elemental ratios of up to 12,000:1 and provides an elemental detection limit of < 40 ppm (TCD).

Dataset- specific Instrument Name	Rosette Niskin bottles
Generic Instrument Name	Niskin bottle
Dataset- specific Description	Water samples were collected using Rosette Niskin bottles attached to a Seabird 911plus conductivity-temperature-depth probe (CTD)
Generic Instrument Description	A Niskin bottle (a next generation water sampler based on the Nansen bottle) is a cylindrical, non-metallic water collection device with stoppers at both ends. The bottles can be attached individually on a hydrowire or deployed in 12, 24, or 36 bottle Rosette systems mounted on a frame and combined with a CTD. Niskin bottles are used to collect discrete water samples for a range of measurements including pigments, nutrients, plankton, etc.

Dataset- specific Instrument Name	FlexStation 3 Multi-mode Microplate Reader, BioTek
Generic Instrument Name	plate reader
Dataset- specific Description	The colorimetric and fluorescence measurements were conducted using a BioTek microplate reader.
Generic Instrument Description	Plate readers (also known as microplate readers) are laboratory instruments designed to detect biological, chemical or physical events of samples in microtiter plates. They are widely used in research, drug discovery, bioassay validation, quality control and manufacturing processes in the pharmaceutical and biotechnological industry and academic organizations. Sample reactions can be assayed in 6-1536 well format microtiter plates. The most common microplate format used in academic research laboratories or clinical diagnostic laboratories is 96-well (8 by 12 matrix) with a typical reaction volume between 100 and 200 uL per well. Higher density microplates (384- or 1536-well microplates) are typically used for screening applications, when throughput (number of samples per day processed) and assay cost per sample become critical parameters, with a typical assay volume between 5 and 50 $\mu$ L per well. Common detection modes for microplate assays are absorbance, fluorescence intensity, luminescence, time-resolved fluorescence, and fluorescence polarization. From: <a href="http://en.wikipedia.org/wiki/Plate_reader">http://en.wikipedia.org/wiki/Plate_reader</a> , 2014-09-0-23.

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# Deployments

#### BH18-09

Website	https://www.bco-dmo.org/deployment/920899	
Platform	R/V Blue Heron	
Start Date	2018-07-30	
End Date	2018-08-08	

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# **Project Information**

# Ecosystem-scale responses of coupled carbon and nutrient cycles to dramatic shifts in benthic communities: The Upper Great Lakes (Great Lakes CNP cycles)

**Coverage**: Upper Laurentian Great Lakes (Lakes Superior, Michigan, Huron)

#### NSF Award Abstract:

This project will study the effects of invasive zebra and quagga mussels on the biology and chemistry of the Great Lakes. These mussels have caused unprecedented changes to lake ecosystems, including large shifts in water quality, altered fisheries and diminished recreational opportunities. The mechanisms by which the invasion has affected ecologically important aspects of lake chemistry remain uncertain, making it difficult to predict future changes. This project will quantify how mussels affect sediment and water nutrient chemistry in Lakes Michigan and Huron. Important missing data on altered chemical exchange between sediments and water will be collected to quantify the processes controlling nutrient cycles in the Great Lakes as water moves to the coastal ocean. Development of improved numerical models from this work will allow evaluation of other freshwater and marine systems affected by similar mussel invasions and other stressors. The project will train

a post-doctoral researcher and several graduate and undergraduate students in highly interdisciplinary research. Educators and the public at large will be served through creation of freely available, online multimedia content, as well as outreach events at the Duluth Great Lakes Aquarium, the Shedd Aquarium in Chicago, and the Discovery World Science Museum in Milwaukee.

Benthic communities can have large impacts on benthic-pelagic chemical exchanges, but the ecosystem-scale effects of benthos on coupled transformations of carbon, nitrogen, and phosphorus (C-N-P) are rarely addressed and virtually unguantified in freshwaters. The recent invasion of the Laurentian Great Lakes, the world's largest freshwater ecosystem, by dreissenid (zebra and guagga) mussels coincided with massive changes to ecology and water chemistry. Consequences of the invasion include a large and unexplained decline in phosphorus levels, enigmatic reversal of past trends in nitrate levels, and the near-disappearance of native bioturbating Diporeia amphipods. Modeling suggests that nutrient fluxes from sediments could have been strongly affected by shifts in benthic community. Mechanisms explaining these changes, however, remain obscure and thus hamper model predictions of future ecosystem trajectories. Researchers from the Large Lakes Observatory - University of Minnesota Duluth will determine the effects of established dreissenid populations on sediment geochemistry and nutrient dynamics in the Great Lakes by conducting detailed studies in dreissenid-invaded Lakes Michigan and Huron and dreissenid-free Lake Superior. A team of biologists and geochemists will (1) obtain field data to characterize sediment geochemistry of C-N-P, asses rates of biogeochemical processes, and dreissenid effects on chemical and physical properties of sediments, (2) conduct lab experiments to determine how functional traits of sessile dreissenids vs. burrowing Diporeia affect sediment characteristics and chemical fluxes, and (3) use reactive-transport and mass-balance models to understand the whole-lake geochemistry of carbon and nutrients in dreissenid-invaded lakes. This work will advance understanding of the ecology of the great lakes and generally improve models of sediment - water column interactions in aquatic ecosystems.

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# Funding

Funding Source	Award
NSF Division of Ocean Sciences (NSF OCE)	
Research Grant Council of the Hong Kong Special Administrative Region, China (RGC)	

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