

# Molecular P characterization (by NMR) of initial dry POM added to each treatment from diatom samples collected on R/V Kilo Moana cruise KM1110 in the North Pacific Subtropical Gyre in 2011

**Website:** <https://www.bco-dmo.org/dataset/557572>

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## Project

» [Taxon-Specific Variability of Organic Matter Production and Remineralization Potential](#) (Taxon-Specific Organic P-C-N Production)

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## Dataset Description

Molecular P characterization (by NMR) of initial dry POM added to each treatment. Data published in Table 1 in Burkhardt et al. (2014).

### *Related Publications and References:*

Burkhardt, B., K. S. Watkins-Brandt, D. Defforey, A. Paytan and A. E. White. 2014. Remineralization of phytoplankton-derived organic matter by natural populations of heterotrophic bacteria. *Marine Chemistry* 162. doi: [10.1016/j.marchem.2014.03.007](https://doi.org/10.1016/j.marchem.2014.03.007)

See Related Datasets:

[Controls](#)

[Killed Controls](#)

[Diatoms](#)

[Trichodesmium](#)

[Prochlorococcus](#)

[OR POM](#)

[Tricho NMR](#)

## Methods & Sampling

All analytical and sampling methodologies are described in Burkhardt et al. (2014). However, summary of most relevant methods are included here:

To explore the relationship between POM source and remineralization rates and stoichiometry, the investigators conducted a suite of on-deck incubation experiments in the North Pacific Subtropical Gyre (NPSG) in March of 2011 near Station ALOHA. 20-L aliquots of seawater were collected from the 75-m depth horizon at Station ALOHA. Immediately after collection, seawater was stored in the dark in an incubator continually flushed with surface seawater for ~72 hours. Dried POM material (cultured *Trichodesmium* IMS 101, "TRICHO", *Prochlorococcus* MED4, "PRO", *T. weissflogii*, "DIATOM" and the natural POM from the Oregon coast, "OR-POM") was added to the carboys with aged Station ALOHA seawater. Each treatment was prepared in duplicate except for the OR-POM. Concentrations of ammonium (NH<sub>4</sub>) and SRP were obtained every 5 min for roughly the first half hour following POM addition to capture any solubilization trends. This initial phase was followed by discrete sampling every 3 hours. Nutrient samples were run at OSU, NMR samples were run at the University of California, Santa Cruz.

Nutrients were analyzed using flow-through colorimetric methods on a Technicon Auto Analyzer II. SRP was analyzed using the phosphomolybdic acid reduction; ammonium (NH<sub>4</sub>) was measured by the indophenol blue method (Gordon et al., 1993); and nitrate + nitrite (N+N) was analyzed using the cadmium reduction method of Armstrong et al. (1967). Detection limits were 55 nmol L<sup>-1</sup> for SRP, 22 nmol L<sup>-1</sup> for NH<sub>4</sub>, and 8 nmol L<sup>-1</sup> for N+N. Total dissolved P and N (TDP and TDN, respectively) were determined by the alkaline persulfate oxidation method (Valderrama, 1981) using a 1:10 oxidant to sample ratio. Dissolved organic P (DOP) and N (DON) were calculated as the difference of TDP and SRP and TDN less the sum of NH<sub>4</sub><sup>+</sup> + NO<sub>3</sub><sup>-</sup> + NO<sub>2</sub><sup>-</sup>, respectively.

Particulate C, N, and P content of each POM type was determined by collecting a subsample of the biomass onto combusted GFF filters, wrapping in foil, flash freezing, and storing at -80 degrees C. The filters were then thawed and dried at 60 degrees C overnight, folded into tin and silver boats, and run on a Carlo-Erba C/N Analyzer for particulate C (PC) and N (PN) content (Sharp (1974). For particulate P (PP) analyses samples were thawed and combusted at 450 degrees C for 4.5 hours, then extracted with 0.15 M HCl for 1 hour at 60 degrees C. PP was then analyzed as SRP in a 1.0 cm cell at 880 nm following Strickland and Parsons (1972).

Molecular characterization of PP compounds was performed using subsamples of each POM type with <sup>31</sup>P nuclear magnetic resonance (NMR) spectral analysis as per Cade-Menun et al. (2005). Samples were freeze-dried, extracted with a 25-mL solution of 0.25M NaOH 0.05M Na<sub>2</sub>EDTA for 4h, and then centrifuged. 1-mL aliquots of the supernatant and digested residue samples were analyzed for P concentrations via inductively coupled plasma optical emission spectroscopy (ICP-OES) to determine the extracted P and fraction that was not extracted. The remaining supernatant was analyzed for <sup>31</sup>P-NMR spectroscopy on a 600 MHz Varian Unity INOVA spectrometer equipped with a 10mm broadband probe at 20 degrees C and a 90 degrees pulse. Compounds were identified by their chemical shifts (ppm) relative to an external orthophosphoric acid standard. After standardizing the orthophosphate peak in all samples to 6 ppm, peak assignments were based on Tebbby and Glonek (1991) Cade-Menun and Preston (1996) and Turner et al. (2003b,c). Peak areas were calculated by integration of spectra processed with a 5 Hz line broadening, using NUTS software (Acorn NMR Inc.) as described in Paytan et al., (2003). Finally, the relative contribution of surface-adsorbed P was assessed for remaining TRICHO and DIATOM POM samples via the oxalate rinse method described in Fu et al. (2005); not enough material remained from PRO and OR-POM for similar analyses.

## Data Processing Description

All data processing is described in Burkhardt et al. (2014). In general, data processing for nutrients involved conversion of raw absorbance data to nutrient concentrations using standard curves.

BCO-DMO processing:

- Replaced blanks (missing data) with 'nd' to indicate 'no data'.
- Modified parameter names to conform with BCO-DMO naming conventions.

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## Data Files

**File****diatom\_NMR.csv**(Comma Separated Values (.csv), 573 bytes)

MD5:bf4750885ce4bfab21a7f04aba85f9ff

Primary data file for dataset ID 557572

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Parameter	Description	Units
taxon	Taxon name.	text
mean_abund_orthoP_pcmt	Mean abundance of orthophosphate, as a percentage.	%
mean_abund_monoesters_pcmt	Mean abundance of monoesters, as a percentage.	%
stdev_abund_orthoP_pcmt	Standard deviation of abundance of orthophosphate.	%
stdev_abund_monoesters_pcmt	Standard deviation of abundance of monoesters.	%
mean_total_P	Mean total P.	micromoles P per gram algae (umol P/g algae)
replicate	Replicate identifier.	1, 2, or 3
nature_monoesters	Description of the monoesters. Some of the monoesters measured in the sample may have been a product of the hydrolysis of diesters (DNA, RNA) due to the high pH of the NMR extractant solution. (a common issue with this solution)	text
abund_orthoP_pcmt	Abundance of orthophosphate, as a percentage.	%
abund_monoesters_pcmt	Abundance of monoesters, as a percentage.	%
rel_conc_orthoP	Relative concentration of orthophosphate.	micromoles P per gram algae (umol P/g algae)
rel_conc_monoesters	Relative concentration of monoesters.	micromoles P per gram algae (umol P/g algae)
total_P	Total P.	micromoles P per gram algae (umol P/g algae)

[\[ table of contents | back to top \]](#)**Instruments**

<b>Dataset-specific Instrument Name</b>	Carlo-Erba C/N Analyzer
<b>Generic Instrument Name</b>	Elemental Analyzer
<b>Dataset-specific Description</b>	A Carlo-Erba C/N Analyzer was used to determine particulate C (PC) and N (PN) content.
<b>Generic Instrument Description</b>	Instruments that quantify carbon, nitrogen and sometimes other elements by combusting the sample at very high temperature and assaying the resulting gaseous oxides. Usually used for samples including organic material.

<b>Dataset-specific Instrument Name</b>	inductively coupled plasma optical emission spectroscopy (ICP-OES)
<b>Generic Instrument Name</b>	Inductively Coupled Plasma Mass Spectrometer
<b>Dataset-specific Description</b>	Samples were analyzed for P concentrations via inductively coupled plasma optical emission spectroscopy (ICP-OES).
<b>Generic Instrument Description</b>	An ICP Mass Spec is an instrument that passes nebulized samples into an inductively-coupled gas plasma (8-10000 K) where they are atomized and ionized. Ions of specific mass-to-charge ratios are quantified in a quadrupole mass spectrometer.

<b>Dataset-specific Instrument Name</b>	nuclear magnetic resonance (NMR)
<b>Generic Instrument Name</b>	Nuclear Magnetic Resonance Spectrometers
<b>Dataset-specific Description</b>	Molecular characterization of PP compounds was performed using subsamples of each POM type with <sup>31</sup> P nuclear magnetic resonance (NMR) spectral analysis.
<b>Generic Instrument Description</b>	Instruments that identify and quantify magnetically active chemical entities by subjecting a sample to orthogonal magnetic and electrical fields.

<b>Dataset-specific Instrument Name</b>	Technicon Auto Analyzer II
<b>Generic Instrument Name</b>	Technicon AutoAnalyzer II
<b>Dataset-specific Description</b>	Nutrients were analyzed using flow-through colorimetric methods on a Technicon Auto Analyzer II.
<b>Generic Instrument Description</b>	A rapid flow analyzer that may be used to measure nutrient concentrations in seawater. It is a continuous segmented flow instrument consisting of a sampler, peristaltic pump, analytical cartridge, heating bath, and colorimeter. See more information about this instrument from the manufacturer.

## Deployments

### KM1110

<b>Website</b>	<a href="https://www.bco-dmo.org/deployment/59056">https://www.bco-dmo.org/deployment/59056</a>
<b>Platform</b>	R/V Kilo Moana
<b>Report</b>	<a href="http://dmoserv3.bco-dmo.org/jg/serv/BCO-DMO/DIAZOTROPHS_CO2/726342.html1%7Bdir=dmoserv3.who.edu/jg/dir/BCO-DMO/DIAZOTROPHS_CO2/,info=dmoserv3.bco-dmo.org/jg/info/BCO-DMO/DIAZOTROPHS_CO2/CO2_experimental%7D?cruise_id_eq_km1110">http://dmoserv3.bco-dmo.org/jg/serv/BCO-DMO/DIAZOTROPHS_CO2/726342.html1%7Bdir=dmoserv3.who.edu/jg/dir/BCO-DMO/DIAZOTROPHS_CO2/,info=dmoserv3.bco-dmo.org/jg/info/BCO-DMO/DIAZOTROPHS_CO2/CO2_experimental%7D?cruise_id_eq_km1110</a>
<b>Start Date</b>	2011-03-12
<b>End Date</b>	2011-03-23

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## Project Information

### Taxon-Specific Variability of Organic Matter Production and Remineralization Potential (Taxon-Specific Organic P-C-N Production)

#### *Description from NSF award abstract:*

The marine phosphorus (P) cycle is characterized by tight coupling between the uptake and decomposition of dissolved inorganic P (DIP) and dissolved organic P (DOP). DIP is incorporated into a broad range of cellular compounds integral for energy storage, genetic material and cell structure. Cell death and autolysis, exudation, viral lysis and grazing all lead to the release of DOP into the environment where it can be depolymerized, hydrolyzed, reassimilated, removed by absorption onto sinking particles or accumulate in the surrounding environment. In this manner, the form and composition of P in the marine environment is largely controlled by the metabolic activity of microorganisms and is intimately linked to the cycling of carbon (C) and nitrogen (N) as particulate organic P (POP) and DOP is bound to C and N in multiple forms, including esters, phospholipids and phosphonates. Thus, a consideration of marine P cycling is most relevant when P transformations are viewed as part of the nutrient and energy flow in the oceanic water column. At the ecosystem scale, the balance of productivity and respiration in the open ocean is regulated by the availability of potentially limiting nutrients such as C, N and P. Therefore, understanding the coupling of C, N, and P cycles is central to the determination of the long-term controls of the magnitude and variability of primary production and particle export. Nonetheless, a paucity of simultaneous measures of dissolved organic carbon (DOC), dissolved organic nitrogen (DON) and DOP and a relative lack of information on production and decomposition processes have hindered progress in understanding the coupled dynamics of these pools. Recent studies of dissolved organic matter (DOM) dynamics show large departures from Redfield trajectories driven by alterations in phytoplankton species composition, the stoichiometry and chemical composition of organic matter production, differential lability of organic compounds and preferential remineralization of N and P by heterotrophic bacteria. Furthermore, there is mounting evidence of the potential liberation of greenhouse gases occurring via DOP hydrolysis.

In this research, the investigators will characterize the composition, lability and remineralization stoichiometry of organic P-C-N produced by ecologically significant photosynthetic genera. They will conduct a series of in situ and laboratory-based bio-assays where particulate (POM) and DOM isolated from *Prochlorococcus* and phosphonate-containing strains of *Trichodesmium* are added to natural microbial populations and incubated in the laboratory and at sea. Hypothesis driven experiments will address the following objectives:

(1) Determine the elemental (P-C-N) stoichiometry and biomolecular alterations (<sup>31</sup>P-nuclear magnetic resonance) occurring in response to exogenous additions of *Trichodesmium* and *Prochlorococcus* POM and DOM to natural populations of heterotrophic bacteria, estimate the labile and semi-labile fraction of organic material generated by ecologically significant genera and measure potential aerobic production of select greenhouse gases (methane and ethane).

(2) Initiate decomposition experiments in the NPSG at opposing phases of the seasonal cycle (summer/winter) in order to capture varying microbial assemblages having different initial metabolic status and community structure.

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## Funding

Funding Source	Award
<a href="#">NSF Division of Ocean Sciences (NSF OCE)</a>	<a href="#">OCE-0962362</a>
<a href="#">NSF Division of Ocean Sciences (NSF OCE)</a>	<a href="#">OCE-0961555</a>

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