Molybdate reactive phosphorus concentrations in NMR pretreatment extracts from sediment samples collected during cruises in the Arctic Ocean, California Margin, and Equatorial Pacific from 1992-1998

Website: https://www.bco-dmo.org/dataset/798995
Data Type: Cruise Results
Version: 1
Version Date: 2020-04-13

Project
» A new marine sediment sample preparation scheme for solution 31P NMR analysis (Marine Sediment Analysis 31P NMR)

Program
» Center for Dark Energy Biosphere Investigations (C-DEBI)

Contributors	Affiliation	Role
Paytan, Adina	University of California-Santa Cruz (UCSC)	Principal Investigator
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Abstract
Molybdate reactive phosphorus concentrations in nuclear magnetic resonance (NMR) pretreatment extracts from sediment samples collected during cruises in the Arctic Ocean, California Margin, and Equatorial Pacific from 1992-1998.

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Coverage

Spatial Extent: **N**:84.083 **E**:-125.017 **S**:-12 **W**:-174.967
Temporal Extent: 1992-10-30 - 1998-07-24

Dataset Description

Molybdate reactive phosphorus concentrations in nuclear magnetic resonance (NMR) pretreatment extracts from sediment samples collected during cruises in the Arctic Ocean, California Margin, and Equatorial Pacific from 1992-1998. These data were published in Defforey et al. (2017). See the related-resource page https://www.bco-dmo.org/related-resource/794727 for other datasets related to this publication. Sediment sample information for this dataset is available as a supplemental document (Sediment_Sample_Info.csv) which contains collection date, water depth, sediment depth, latitude, and longitude. Additional award information: *

NSF C-DEBI subaward # 156246 to Adina Paytan * NSF C-DEBI subaward # 157598 to Delphine Defforey

Methods & Sampling

Locations:

Arctic Ocean: P-1-94-AR P21, 84°5' N, 174°58' W
California margin: W-2-98-NC TF1, 41°5' N, 125°1' W
Equatorial Pacific: TT013-06MC, 12°00' S, 134°56' W

Methodology:

Prior to the extraction, we freeze-dried, ground and sieved sediment samples to less than 125 µm (Ruttenberg 1992). For a given sample, we weighed four sample replicates (2 g) and placed each in 250 mL HDPE bottles. Sodium dithionite (F.W. 147.12 g/mol; 7.4 g) was added to each sample split, followed by 200 mL of citrate-bicarbonate solution (pH 7.6). This step produces effervescence, so the solution should be added slowly to the sample. We shook samples for 8 h and then centrifuged them at 3,700 rpm for 15 min. We filtered the supernatants with a 0.4 µm polycarbonate filter. We took 20 mL aliquots from the filtrate for each sample split for MRP and total P analyses, and kept them refrigerated until analysis within 24 h. We added 200 mL of ultrapure water to the solid residue for each sample split as a wash step after the above reductive step, shook samples for 2 h, and then centrifuged them at 3,700 rpm for 15 min. We filtered the supernatants with 0.4 µm polycarbonate filters and set aside 20 mL of filtrate from each sample split for MRP and total P analyses. We then extracted the solid sample residues in 200 mL of sodium acetate buffer (pH 4.0) for 6 h. At the end of this extraction step, we centrifuged the bottles at 3,700 rpm for 15 min, filtered the supernatants with 0.4 µm polycarbonate filters and took a 20 mL aliquot of filtrate from each sample split for MRP and total P analyses. We added 200 mL of ultrapure water to the solid residue for each sample split as a wash step, shook samples for 2 h, and then centrifuged them at 3,700 rpm for 15 min. We filtered the supernatants with 0.4 µm polycarbonate filters and set aside 20 mL of filtrate from each sample split for MRP and total P analyses. We repeated the water rinse step, and collected aliquots for MRP and total P analyses as in the previous steps. The concentrations of MRP were determined as described below.

The MRP concentrations were measured on a QuikChem 8000 automated ion analyzer. Standards were prepared with the same solutions used for the extraction step to minimize matrix effects on P measurements. Sediment extracts and standards (0 30 µM PO_4) were diluted ten-fold to prevent matrix interference with color development. The detection limit for P on this instrument is 0.2 µM. We derived MUP concentrations by subtracting MRP from total P concentrations, which are included in a different dataset.

Data Processing Description

Data were processed in Excel.

BCO-DMO Data Manager Processing Notes:
* Data from originally submitted Excel file Data_MRP_sediments with pretreatment_v2.xlsx in sheet "Step 1" and "Step 2" were combined and exported as csv.
* added a conventional header with dataset name, PI name, version date
* modified parameter names to conform with BCO-DMO naming conventions
* blank values in this dataset are displayed as "nd" for "no data." nd is the default missing data identifier in the BCO-DMO system.
* PO4 values with eight decimal places in the Sheet "Step 2" were rounded to two decimal places to match the precision of other values in the column.
* Concentration_Units column with all values uM removed. This information is captured in the Parameter descriptions.

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Data Files

File
nmr_sed_pre.csv(Comma Separated Values (.csv), 22.40 KB) MD5:3ef33f16f83763d7ac785233a5538e87 Primary data file for dataset ID 798995

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Supplemental Files

File
Sediment Sample Information filename: Sediment_Sample_Info.csv (Comma Separated Values (.csv), 657 bytes) MD5:4cfcfa04b5d6f41a024f3304d1598170 Sediment sample information: Region,Sample_ID,Latitude (decimal degrees),Longitude (decimal degrees),Water_depth (m),Sediment_Depth_Start (cm),Sediment_Depth_End (cm),Collection_Start_Date (yyyy-mm-dd),Collection_End_Date (yyyy-mm-dd) Cruises: P194AR (P-1-94-AR), W9807A (W-2-98-NC), TT013 (TT013-06MC)

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Related Publications

Defforey, D., Cade-Menun, B. J., & Paytan, A. (2017). A new solution 31 P NMR sample preparation scheme for marine sediments. Limnology and Oceanography: Methods, 15(4), 381–393. doi:10.1002/lom3.10166
Results

Ruttenberg, K. C. (1992). Development of a sequential extraction method for different forms of phosphorus in marine sediments. Limnology and Oceanography, 37(7), 1460–1482. doi:10.4319/lo.1992.37.7.1460
Methods

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Parameters

Parameter	Description	Units
Extract	Extraction solution	unitless
Step	Step in the sequential extraction scheme (1–4)	unitless
Dilution	Sample dilution or "None"	unitless
Date	Date the samples were analyzed in ISO 8601 format yyyy-mm-dd	unitless
Sample_ID	Unique sample identifier	unitless
Analyte_Name	Element analyzed	unitless
Peak_Concentration	Phosphate concentration (uncorrected)	micromolar (uM)
blank_corrected	Concentration adjusted after blank	unitless
Actual_PO4	Concentration corrected for dilution	micromolar (uM)
P_extracted	Amount of phosphorus extracted	micromoles (umol)
Sed_mass	Dried sediment mass	grams (g)
PO4	Micromoles of phosphorus per gram of sediment (ground dry weight)	micromoles per gram (umol/g)
Peak_Area	Peak area	volts
Peak_Height	Peak height	volts

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Instruments

Dataset-specific Instrument Name	QuikChem 8000 automated ion analyzer
Generic Instrument Name	Flow Injection Analyzer
Generic Instrument Description	An instrument that performs flow injection analysis. Flow injection analysis (FIA) is an approach to chemical analysis that is accomplished by injecting a plug of sample into a flowing carrier stream. FIA is an automated method in which a sample is injected into a continuous flow of a carrier solution that mixes with other continuously flowing solutions before reaching a detector. Precision is dramatically increased when FIA is used instead of manual injections and as a result very specific FIA systems have been developed for a wide array of analytical techniques.

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Deployments

P194AR

Website	https://www.bco-dmo.org/deployment/794741
Platform	USCGC Polar Sea
Start Date	1994-07-25
End Date	1994-08-30
Description	Exact dates in and out of port are not known. The start and end date listed are the start and end dates from cruise trackline and bathymetry data. The same dates are cited in several publications.

W9807A

Website	https://www.bco-dmo.org/deployment/795245
Platform	R/V Wecoma
Start Date	1998-07-17
End Date	1998-07-24
Description	Excerpt from https://pubs.usgs.gov/of/2001/0190/intro.html W9807A (w-2-98-nc) (metadata) The most recent cruise of the STRATAFORM project took place again aboard the R/V Wecoma in July 1998. The principle investigators were Homa Lee of the USGS and Clark R. Alexander from the Skidaway Institute of Oceanography. Also participating from the USGS were Gita Dunhill and Brad Carkin. Jacques Locat and Eric Boulanger from the University of Laval, Quebec, Canada, Harold Christian from GSC, and Brian McAdoo, of Vasser College, also participated in the cruise with their own scientific agendas. Sampling centered around obtaining box cores of the study area, along with Lehigh cores, CTDs, and piezometer readings. The cruise commenced on July 17 and was completed on July 24.

TT013

Website	https://www.bco-dmo.org/deployment/57732
Platform	R/V Thomas G. Thompson
Start Date	1992-10-30
End Date	1992-12-13
Description	Purpose: Benthic Survey, 12°N-12°S at 140°W TT013 was one of five cruises conducted in 1992 in support of the U.S. Equatorial Pacific (EqPac) Process Study. The five EqPac cruises aboard R/V Thomas G. Thompson included two repeat meridional sections (12°N - 12°S), 2 equatorial surveys, and a benthic survey (all at 140° W). The scientific objectives of this study were to observe the processes in the Equatorial Pacific controlling the fluxes of carbon and related elements between the atmosphere, euphotic zone, and deep ocean. As luck would have it, the survey window coincided with an El Nino event. A bonus for the research team. **Methods & Sampling** TT013-06MC

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Project Information

A new marine sediment sample preparation scheme for solution 31P NMR analysis (Marine Sediment Analysis 31P NMR)

Coverage: Equatorial Pacific, California Margin, Arctic Ocean

We developed and tested a new approach to prepare marine sediment samples for solution 31P nuclear magnetic resonance spectroscopy (31P NMR). This approach addresses the effects of sample pretreatment on sedimentary P composition and increases the signal of low abundance P species in 31P NMR spectra by removing up the majority inorganic P from sediment samples while causing minimal alteration of the chemical structure of organic P compounds. The method was tested on natural marine sediment samples from different localities (Equatorial Pacific, California Margin and Arctic Ocean) with high inorganic P content, and allowed for the detection of low abundance P forms in samples for which only an orthophosphate signal could be resolved with an NaOH-EDTA extraction alone. This new approach will allow the use of 31P NMR on samples for which low organic P concentrations previously hindered the use of this tool, and will help
answer longstanding question regarding the fate of organic P in marine sediments. We developed and tested a new approach to prepare marine sediment samples for solution 31P nuclear magnetic resonance spectroscopy (31P NMR). This approach addresses the effects of sample pretreatment on sedimentary P composition and increases the signal of low abundance P species in 31P NMR spectra by removing up the majority inorganic P from sediment samples while causing minimal alteration of the chemical structure of organic P compounds. The method was tested on natural marine sediment samples from different localities (Equatorial Pacific, California Margin and Arctic Ocean) with high inorganic P content, and allowed for the detection of low abundance P forms in samples for which only an orthophosphate signal could be resolved with an NaOH-EDTA extraction alone. This new approach will allow the use of 31P NMR on samples for which low organic P concentrations previously hindered the use of this tool, and will help
answer longstanding question regarding the fate of organic P in marine sediments.

NSF C-DEBI Award #156246 to Dr. Adina Paytan

NSF C-DEBI Award #157598 to Dr. Delphine Defforey

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Program Information

Center for Dark Energy Biosphere Investigations (C-DEBI)

Website: http://www.darkenergybiosphere.org

Coverage: Global

The mission of the Center for Dark Energy Biosphere Investigations (C-DEBI) is to explore life beneath the seafloor and make transformative discoveries that advance science, benefit society, and inspire people of all ages and origins.

C-DEBI provides a framework for a large, multi-disciplinary group of scientists to pursue fundamental questions about life deep in the sub-surface environment of Earth. The fundamental science questions of C-DEBI involve exploration and discovery, uncovering the processes that constrain the sub-surface biosphere below the oceans, and implications to the Earth system. What type of life exists in this deep biosphere, how much, and how is it distributed and dispersed? What are the physical-chemical conditions that promote or limit life? What are the important oxidation-reduction processes and are they unique or important to humankind? How does this biosphere influence global energy and material cycles, particularly the carbon cycle? Finally, can we discern how such life evolved in geological settings beneath the ocean floor, and how this might relate to ideas about the origin of life on our planet?

C-DEBI's scientific goals are pursued with a combination of approaches:
(1) coordinate, integrate, support, and extend the research associated with four major programs—Juan de Fuca Ridge flank (JdF), South Pacific Gyre (SPG), North Pond (NP), and Dorado Outcrop (DO)—and other field sites;
(2) make substantial investments of resources to support field, laboratory, analytical, and modeling studies of the deep subseafloor ecosystems;
(3) facilitate and encourage synthesis and thematic understanding of submarine microbiological processes, through funding of scientific and technical activities, coordination and hosting of meetings and workshops, and support of (mostly junior) researchers and graduate students; and
(4) entrain, educate, inspire, and mentor an interdisciplinary community of researchers and educators, with an emphasis on undergraduate and graduate students and early-career scientists.

Note: Katrina Edwards was a former PI of C-DEBI; James Cowen is a former co-PI.

Data Management:
C-DEBI is committed to ensuring all the data generated are publically available and deposited in a data repository for long-term storage as stated in their Data Management Plan (PDF) and in compliance with the NSF Ocean Sciences Sample and Data Policy. The data types and products resulting from C-DEBI-supported research include a wide variety of geophysical, geological, geochemical, and biological information, in addition to education and outreach materials, technical documents, and samples. All data and information generated by C-DEBI-supported research projects are required to be made publically available either following publication of research results or within two (2) years of data generation.

To ensure preservation and dissemination of the diverse data-types generated, C-DEBI researchers are working with BCO-DMO Data Managers make data publicly available online. The partnership with BCO-DMO helps ensure that the C-DEBI data are discoverable and available for reuse. Some C-DEBI data is better served by specialized repositories (NCBI's GenBank for sequence data, for example) and, in those cases, BCO-DMO provides dataset documentation (metadata) that includes links to those external repositories.

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Funding

Funding Source	Award
NSF Division of Ocean Sciences (NSF OCE)	OCE-0939564

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