Total phosphorus and metal concentrations from sediment extracts from samples collected during cruises in the Arctic Ocean, California Margin, and Equatorial Pacific from 1992-1998

Website: https://www.bco-dmo.org/dataset/805216
Data Type: Cruise Results
Version: 1
Version Date: 2020-06-23

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
Defforey, Delphine	University of California-Santa Cruz (UCSC)	Co-Principal Investigator
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Abstract
Total phosphorus and metal concentrations from sediment extracts from 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

Total phosphorus and metal concentrations from sediment extracts from 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

Location:

Arctic Ocean: P-1-94-AR P21, 84o5' N, 174o58' W
California margin: W-2-98-NC TF1, 41o5' N, 125o1' W
Equatorial Pacific: TT013-06MC, 12o00' S, 134o56' W

Methodology:

Solid sediment samples were transferred to two 50 mL centrifuge tubes (2 sample replicates combined per tube). We added 20 mL of 0.25 M NaOH + 0.05 M Na2EDTA solution to each tube, vortexed until all sediment was resuspended and then shook samples for 6 h at room temperature (Cade-Menun et al. 2005). We used a solid to solution ratio of 1:5 for this step to minimize the amount of freeze-dried material that will need to be dissolved for the 31P NMR experiments. Large amounts of salts from the NaOH-EDTA concentrated in NMR samples lead to higher viscosity and increase line broadening on NMR spectra (Cade-Menun and Liu 2013). We chose an extraction time of 6 h to improve total P recovery while limiting the degradation of natural P compounds in the sample. At the end of the extraction, samples were centrifuged at 3,700 rpm for 15 min and supernatants decanted into 50 mL centrifuge tubes. We collected a 500 µL aliquot from each sample, which we diluted with 4.5 mL of ultrapure water. These were refrigerated until analysis for total P content on the ICP-OES. The sample residues and supernatants were frozen on a slant to maximize the exposed surface area during the lyophilization step; this was done immediately after the removal of the 500 µL aliquot. Once completely frozen, the uncapped tubes containing supernatants and residues were freeze-dried over the course of 48 h. Each tube was covered with parafilm with small holes from a tack to minimize contamination. Freeze-dried supernatants from identical sample splits were combined and dissolved in 500 µL each of ultrapure water, D2O, NaOH-EDTA and 10 M NaOH prior to 31P NMR analysis. The D2O is required as signal lock in the spectrometer (Cade-Menun and Liu 2013). Sample pH was maintained at a pH > 12 to optimize peak separation (Cade-Menun 2005; Cade-Menun and Liu 2013). Sample pH was assessed with a glass electrode, and verified with pH paper to account for the alkaline error caused by the high salt content of our samples (Covington 1985).

Total P and metal concentrations in sediment extracts were measured using inductively coupled plasma optical emission spectroscopy (ICP-OES). Standards were prepared with the same solutions as those used for the extraction procedure in order to minimize matrix effects on P measurements. Sediment extracts and standards (0 µM, 3.2 µM, 32 µM and 320 µM) were diluted to lower salt content to prevent salt buildup on the nebulizer. Concentration data from both wavelengths (213 nm and 214 nm) were averaged to obtain extract concentrations for each sample. The detection limit for P on this instrument for both wavelengths is 0.4 µM.

Freeze-dried sample residues were ashed in crucibles at 550oC for 2 h and then extracted in 25 mL of 0.5 M sulfuric acid for 16 h (Olsen and Sommers 1982; Cade-Menun and Lavkulich 1997). We centrifuged samples at 3,700 rpm for 15 min, filtered supernatants with 0.4 µm polycarbonate filters, and measured P content on an ICP-OES.

Data Processing Description

Data were processed in Excel.

BCO-DMO Data Manager Processing Notes:
* Data from originally submitted Excel file Data_TP+metals_sediments with no pretreatment_v2.xlsx in sheets "NMR extract" and "Residue" were exported as csv.
* Column "Type" added with values "NMR extract" or "Residue", then the data form the two sheets were combined into one table.
* 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.

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Data Files

File
tp-metals-no-pre.csv (Comma Separated Values (.csv), 35.20 KB) MD5:75bffba84a5eb9502894c8031419328f Primary data file for dataset ID 805216

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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

Cade-Menun, B. (2005). Characterizing phosphorus in environmental and agricultural samples by 31P nuclear magnetic resonance spectroscopy. Talanta, 66(2), 359–371. doi:10.1016/j.talanta.2004.12.024
Methods

Cade-Menun, B., & Liu, C. W. (2013). Solution Phosphorus-31 Nuclear Magnetic Resonance Spectroscopy of Soils from 2005 to 2013: A Review of Sample Preparation and Experimental Parameters. Soil Science Society of America Journal, 78(1), 19–37. doi:10.2136/sssaj2013.05.0187dgs
Methods

Cade-Menun, B. J., & Lavkulich, L. M. (1997). A comparison of methods to determine total, organic, and available phosphorus in forest soils. Communications in Soil Science and Plant Analysis, 28(9-10), 651–663. doi:10.1080/00103629709369818
Methods

Covington, A. K. (1985). Procedures for testing pH responsive glass electrodes at 25, 37, 65 and 85 C and determination of alkaline errors up to 1 mol dm-3 Na+, K+, Li+. Pure and Applied Chemistry, 57(6), 887–898. doi:10.1351/pac198557060887
Methods

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

Olsen, S. R., & Sommers, L. E. (1982). Phosphorus. p. 403–430. In A.L. Page, R.H. Miller, and D.R. Keeney [eds.], Methods of Soil Analysis. Soil Science Society of America.
Methods

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Parameters

Parameter	Description	Units
Type	Type (NMR extract or Residue)	unitless
Extract	Extraction solution	unitless
Dilution	Sample dilution or "None"	unitless
Sample_ID	Unique sample identifier	unitless
Analyte_Name	Element analyzed	unitless
Int_Corr	Intensity (corrected)	unknown
RSD_Corr_Int	Relative standard deviation	unknown
Conc_Calib	Calibrated concentration of total phosphorous	parts per million (ppm)

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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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