Geochemical measurements of porewater from sediment push core samples in the Gulf of California during R/V Falkor cruise FK190211 in 2019

Website: https://www.bco-dmo.org/dataset/821665 Data Type: Cruise Results Version: 1 Version Date: 2020-08-21

Project

» <u>Collaborative Research: Microbial Carbon cycling and its interactions with Sulfur and Nitrogen</u> <u>transformations in Guaymas Basin hydrothermal sediments</u> (Guaymas Basin Interactions)

Contributors	Affiliation	Role
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Abstract

Geochemical measurements of porewater from sediment push core samples in the Gulf of California during R/V Falkor cruise FK190211 in 2019

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Coverage

Spatial Extent: N:27.5909 **E**:-111.40342 **S**:27.00643 **W**:-111.47512 **Temporal Extent**: 2019-02-27 - 2019-03-11

Dataset Description

Geochemical measurements of porewater from sediment push core samples in the Gulf of California during R/V Falkor cruise FK190211 in 2019.

Methods & Sampling

Location: Guaymas Basin, Gulf of California, 27 00.00 N, -111 20.00 W

Sampling and analytical procedures:

Sediment samples were collected by the ROV SuBastian using PVC push cores. Upon arrival at the surface the cores were described and cataloged prior to being sectioned into discrete depth intervals. Porewater was separated from the sediment using a manually-actuated porewater press as described by Joye et al. 2004.

Following the collection of porewater, the pressed sediment sample (mud-cake) was stored at -20°C for solid-phase analysis. Porewater and sediment samples were preserved and analyzed as follows:

1) pH: Sediment pH was determined using a sediment probe (Oakton, Prod. No. 35634-50) and measured immediately during core sectioning. Porewater pH was determined using a Ross electrode (Thermo Fisher, Prod. No. 8103BNUWP).

2) Salinity: Porewater salinity was determined using a handheld refractometer.

3) Nutrients (DOC, TDN, NOx, NO2, PO4, TDP): Porewater sample was filtered through a pre-rinsed 0.2 μm regenerated cellulose Target2 syringe filter (Thermo Scientific, Prod. No. F25047), collected into an HDPE bottle and stored frozen at -20°C until analysis. Individual analytes were analyzed as follows:

DOC was determined using high temperature catalytic combustion and an NDIR detector following the method described in Sugimura and Suzuki, 1988.

TDN was determined using high temperature combustion and a chemiluminescence detector following the method described in Watanabe et. al, 2007.

NOx was determined using chemical reduction and a nitric oxide detector following the method described by Garside, 1982.

NO2 was determined using the colorimetric method described by Bendschneider and Robinson, 1952 as reproduced by Parsons, Marta, and Lalli, 1984.

PO4 was determined using the colorimetric method described by Strickland and Parsons, 1972. TDP was determined using the colorimetric method described by Solorzano and Sharp, 1980.

4) NH4: Porewater sample was filtered through a pre-rinsed 0.2 μm regenerated cellulose Target2 syringe filter (Thermo Scientific, Prod. No. F25047), collected into a 15 mL centrifuge tube containing 100 μL of phenol reagent (cold; 4°C) per 1 mL of sample, and stored at 4°C until analysis. NH4 was determined using the colorimetric method described by Solorzano, 1969.

5) Alkalinity: Porewater sample was filtered through a pre-rinsed 0.2 μm regenerated cellulose Target2 syringe filter (Thermo Scientific, Prod. No. F25047). Alkalinity was determined using the spectrophotometric method described by Sarazin et al., 1999.

6) Methane, Ethane, and Propane: Sediment samples (3 cubic centimeters) were collected into a glass serum vial, preserved with N2-purged 2M NaOH (3 milliliters), crimp-sealed with a butyl rubber stopper and stored at room temperature until analysis. Concentrations were determined by headspace analysis using an SRI 8610C gas chromatograph equipped with a flame ionization detector and SRI Hayesep D 6'x1/8" column (Prod. No. 8600-PKDB).

7) H2S: 2 mL porewater sample was collected into a 15 mL centrifuge tube containing 100 uL or 500 uL 20% zinc acetate w/v solution and stored at 5°C until analysis. H2S was determined using the colorimetric method described by Cline, 1969.

8) Sulfate (SO4): Porewater sample was filtered through a pre-rinsed 0.2 μm regenerated cellulose Target2 syringe filter (Thermo Scientific, Prod. No. F25047) into a 7 mL scintillation vial. Samples were acidified with 10 μL of concentrated HNO3 per 1 mL sample, sealed with a PTFE lined cap, and stored at room temperature until analysis. Sample analysis was performed using KOH eluent supplied by a Dionex EGC III KOH Eluent Generator Cartridge (Prod. No. 074532), Dionex CR-ATC Continuously Regenerated Trap Column (Prod. No. 060477), Dionex AERS 500 Electronically Regenerated Suppressor (Prod. No. 082541), Dionex IonPac AG19 Guard Column (Prod. No. 062888), Dionex IonPac AS19 Analytical Column (Prod. No. 062886) and Dionex CRD 200 RFIC Carbonate Removal Device (Prod. No. 062986). Reference - Weston et al. 2006, Biogeochemistry 77: 375-408.

9) Chloride (Cl): Porewater sample was filtered through a pre-rinsed 0.2 µm regenerated cellulose Target2 syringe filter (Thermo Scientific, Prod. No. F25047) into a 7 mL scintillation vial. Samples were acidified with 10 µL of concentrated HNO3 per 1 mL sample, sealed with a PTFE lined cap, and stored at room temperature until analysis. Sample analysis was performed using KOH eluent supplied by a Dionex EGC III KOH Eluent Generator Cartridge (Prod. No. 074532), Dionex CR-ATC Continuously Regenerated Trap Column (Prod. No. 060477), Dionex AERS 500 Electronically Regenerated Suppressor (Prod. No. 082541), Dionex IonPac AG19 Guard Column (Prod. No. 062888), Dionex IonPac AS19 Analytical Column (Prod. No. 062886) and Dionex CRD 200 RFIC Carbonate Removal Device (Prod. No. 062986). Reference - Weston et al. 2006, Biogeochemistry 77: 375-408.

10) Porosity: Sediment samples were collected into glass scintillation vials, capped and stored at 4°C until analysis. Porosity was determined by drying a sample of known mass at 80°C, weighing, and calculating the

moisture content.

11) SOM (LOI): Sediment samples were collected into glass scintillation vials, capped and stored at 4°C until analysis. SOM was determined by drying a sample at 80°C, weighing, ashing at 500°C, weighing and calculating weight lost on ignition.

12) TPN: The pressed sediment sample (mud-cake) was dried at 60°C and then homogenized via grinding. TPN was determined by the method described by Gordan, Jr., 1969 as reproduced by Sharp, 1974. Samples were analyzed on a ThermoFinnigan FlashEA 1112 series NC Soil Analyzer.

13) TPC: The pressed sediment sample (mud-cake) was dried at 60°C and then homogenized via grinding. TPC was determined by the method described by Gordan, Jr., 1969 as reproduced by Sharp, 1974. Samples were analyzed on a ThermoFinnigan FlashEA 1112 series NC Soil Analyzer.

14) POC: The pressed sediment sample (mud-cake) was acidified with 1 N HCl, dried at 60°C, and then homogenized via grinding. POC was determined by the method described by Gordan, Jr., 1969 as reproduced by Sharp, 1974. Samples were analyzed on a ThermoFinnigan FlashEA 1112 series NC Soil Analyzer.

15) TPP and TPP percent: The pressed sediment sample (mud-cake) was dried at 80°C, ground, and distributed into 20 mL scintillation vials. The samples were prepared using the method described by Zhou et. al., 2003. TPP and TPP percent were determined using the colormetric method described by Solorzano and Sharp, 1980.

16) Calculated Values: Values for NO3, DIN, DON, DOP, PIC were calculated as follows:

NO3 = NOx - NO2 DIN = NOx + NH4 DON = TDN - DIN DOP = TDP - PO4PIC = TPC - POC

Known Issues:

PW NO2: The following samples were cloudy and most had no visible pink color development. Concentrations given are estimates made by visually comparing sample tubes to standards. Samples: 2, 3, 6-8, 11-14, 16-19, 39-41, 43-51, 65, 66, 68, 69, 72-76

PW PO4: The following samples were cloudy but had blue color development. Concentrations given are estimates made by visually comparing sample tubes to standards. Samples: 3, 6-8, 11-14, 18, 19, 41, 45-47, 49, 65, 66, 68, 69, 72-74, 76, 77

Data Processing Description

Microsoft Excel was used to tabulate the sediment and porewater geochemistry data.

BCO-DMO Data Manager Processing Notes:

* Extracted data submitted in Excel file Joye_OCE_1357360_Data_FK190211_Sediment_Geochemistry.xlsx to csv

* added a conventional header with dataset name, PI name, version date

* modified parameter names to conform with BCO-DMO naming conventions: only A-Za-z0-9 and underscore allowed. Can not start with a number. (spaces, +, and - changed to underscores).

* blank values in this dataset are displayed as "nd" for "no data." nd is the default missing data identifier in the BCO-DMO system.

* longitude and latitude rounded to five decimal places

- * Date converted to ISO 8601 format YYYY-MM-DD
- * Time converted to ISO 8601 format HH:MM

* ISO_DateTime_UTC column added in format YYYY-MM-DDThh:mmZ

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File fk190211-sed-geochem.csv(Comma Separated Values (.csv), 15.24 KB) MD5:d3e18e7e6cc1974bb1ffd3bc3dc43c2b Primary data file for dataset ID 821665

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Related Publications

Cline, J. D. (1969). Spectrophotometric Determination of Hydrogen Sulfide in Natural Waters. Limnology and Oceanography, 14(3), 454–458. doi:<u>10.4319/lo.1969.14.3.0454</u> *Methods*

Garside, C. (1982). A chemiluminescent technique for the determination of nanomolar concentrations of nitrate and nitrite in seawater. Marine Chemistry, 11(2), 159–167. doi:<u>10.1016/0304-4203(82)90039-1</u> *Methods*

Gordon, D. G. (1969). Examination of methods of particulate organic carVon analysis. Deep Sea Research and Oceanographic Abstracts, 16(6), 661–665. doi:<u>10.1016/0011-7471(69)90066-7</u> *Methods*

Joye, S. B., Boetius, A., Orcutt, B. N., Montoya, J. P., Schulz, H. N., Erickson, M. J., & Lugo, S. K. (2004). The anaerobic oxidation of methane and sulfate reduction in sediments from Gulf of Mexico cold seeps. Chemical Geology, 205(3-4), 219–238. doi:<u>10.1016/j.chemgeo.2003.12.019</u> *Methods*

Parsons, T. R., Y. Maita, and C. M. Lalli. "A Manual of Chemical and Biological Methods of Seawater Analysis", Pergamon Press (1984). ISBN: <u>9780080302874</u> *Methods*

Sarazin, G., Michard, G., & Prevot, F. (1999). A rapid and accurate spectroscopic method for alkalinity measurements in sea water samples. Water Research, 33(1), 290–294. doi:10.1016/s0043-1354(98)00168-7 https://doi.org/10.1016/S0043-1354(98)00168-7 Methods

Sharp, J. H. (1974). Improved analysis for "particulate" organic carbon and nitrogen from seawater1. Limnology and Oceanography, 19(6), 984–989. doi:<u>10.4319/lo.1974.19.6.0984</u> *Methods*

Solorzano, L. (1969). DETERMINATION OF AMMONIA IN NATURAL WATERS BY THE PHENOLHYPOCHLORITE METHOD Limnology and oceanography, 14(5), 799-801. https://pdfs.semanticscholar.org/7b24/b41d9b4e1ad507a06f282d0e0aed1e51e89e.pdf Methods

Solórzano, L., & Sharp, J. H. (1980). Determination of total dissolved phosphorus and particulate phosphorus in natural waters1. Limnology and Oceanography, 25(4), 754–758. doi:<u>10.4319/lo.1980.25.4.0754</u> *Methods*

Strickland, J. D. H. and Parsons, T. R. (1972). A Practical Hand Book of Seawater Analysis. Fisheries Research Board of Canada Bulletin 157, 2nd Edition, 310 p. *Methods*

Sugimura, Y., & Suzuki, Y. (1988). A high-temperature catalytic oxidation method for the determination of nonvolatile dissolved organic carbon in seawater by direct injection of a liquid sample. Marine Chemistry, 24(2), 105-131. doi:<u>10.1016/0304-4203(88)90043-6</u> *Methods*

Watanabe, K., Badr, E.-S., Pan, X., & Achterberg, E. P. (2007). Conversion efficiency of the high-temperature combustion technique for dissolved organic carbon and total dissolved nitrogen analysis. International Journal of Environmental Analytical Chemistry, 87(6), 387–399. doi:<u>10.1080/03067310701237023</u> *Methods*

Weston, N. B., Porubsky, W. P., Samarkin, V. A., Erickson, M., Macavoy, S. E., & Joye, S. B. (2006). Porewater Stoichiometry of Terminal Metabolic Products, Sulfate, and Dissolved Organic Carbon and Nitrogen in Estuarine Intertidal Creek-bank Sediments. Biogeochemistry, 77(3), 375–408. doi:<u>10.1007/s10533-005-1640-1</u> *Methods*

Parameters

Parameter	Description	Units
Date	Date of sample collection in ISO 8601 format YYYY-MM-DD	unitless
Time	Time of sample collection in (Zulu time UTC) in ISO 8601 format hh:mm	unitless
Site	Site where sample was collected	unitless
Latitude	Degrees North	Decimal degrees (DD)
Longitude	Degrees West	Decimal degrees (DD)
Operation_num	Operation number	unitless
Dive_num	Dive number	unitless
Core_num	Label on pushcore	unitless
Collection_Depth	Seafloor depth from which sample was collected	meters (m)
Sediment_Depth_Min	The minimum (shallowest) depth of the sediment layer from which the sample was taken. OWL = Overlying water sample.	centimeters (cm)
Sediment_Depth_Mid_point	The mid-point depth of the sediment layer from which the sample was taken. $OWL = Overlying$ water sample.	centimeters (cm)
Sediment_Depth_Max	The maximum (deepest) depth of the sediment layer from which the sample was taken. $OWL = Overlying$ water sample.	centimeters (cm)
Sample_ID	Sample identifier.	unitless
Sediment_pH	pH of the sediment layer.	pH scale
Porewater_pH	pH of the porewater.	pH scale
Salinity	Salinity	practical salinity units (psu)
NH4	Ammonium. Method detection limit = 0.1	micromolar (uM)
NO2	Nitrite. Method detection limit $= 0.1$	micromolar (uM)
NOx	Nitrate + Nitrite. Method detection limit = 0.1	micromolar (uM)
NO3	Nitrate. Method detection limit = 0.1	micromolar (uM)
DIN	Dissolved Inorganic Nitrogen. Method detection limit = 0.1	micromolar (uM)
TDN	Total Dissolved Nitrogen. Method detection limit = 1	micromolar (uM)
DON	Dissolved Organic Nitrogen. Method detection limit = 1	micromolar (uM)
PO4	Phosphate. Method detection limit = 0.1	micromolar (uM)
TDP	Total Dissolved Phosphate. Method detection limit $= 0.1$	micromolar (uM)
DOP	Dissolved Organic Phosphate. Method detection limit = 0.1	micromolar (uM)
DOC	Dissolved Organic Carbon. Method detection limit = 1	micromolar (uM)

Alkalinity	Alkalinity. Method detection limit = 0.1	millimolar (mM)
Methane	Dissolved methane. Method detection limit = 0.1	micromolar (uM)
Ethane	Dissolved ethane. Method detection limit = 0.1	micromolar (uM)
Propane	Dissolved propane. Method detection limit = 0.1	micromolar (uM)
H2S	Sulfide. Method detection limit = 0.001	millimolar (mM)
Sulfate	Sulfate. Method detection limit $= 0.1$	millimolar (mM)
Chloride	Chloride. Method detection limit = 1	millimolar (mM)
Porosity	Sediment moisture content. Method detection limit = 1	percent
SOM_LOI	Sediment Organic Matter/(Loss on Ignition). Method detection limit = 0.2	percent
TPN	Total Particulate Nitrogen. Method detection limit = 0.1	percent
ТРС	Total Particulate Carbon. Method detection limit = 0.1	percent
POC	Particulate Organic Carbon. Method detection limit $= 0.1$	percent
PIC	Particulate Inorganic Carbon. Method detection $limit = 0.1$	percent
ТРР	Total Particulate Phosphorus. Method detection limit = 0.1	micromole of Phosphorus per gram of sediment (uMol of P/g of sediment)
TPP_percent	Total Particulate Phosphorus Percentage. Method detection limit = 0.1	percent
ISO_DateTime_UTC	Date and time (UTC) in ISO 8601 format YYYY-MM- DDThh:mmZ	yyyy-MM-dd'T'HH:mm'Z'

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Instruments

Dataset- specific Instrument Name	Antek Instruments 7050 Nitric Oxide Detector with 745 Nitrate/Nitrite Reduction Assembly
Generic Instrument Name	Chemiluminescence NOx Analyzer
Generic Instrument Description	The chemiluminescence method for gas analysis of oxides of nitrogen relies on the measurement of light produced by the gas-phase titration of nitric oxide and ozone. A chemiluminescence analyzer can measure the concentration of NO/NO2/NOX. One example is the Teledyne Model T200: <u>https://www.teledyne-api.com/products/nitrogen-compound-instruments/t200</u>

Dataset- specific Instrument Name	ThermoFinnigan FlashEA 1112 series NC Soil Analyzer
Generic Instrument Name	Elemental Analyzer
Generic Instrument Description	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.

Dataset- specific Instrument Name	Shimadzu Instruments GC-2014 Gas Chromatograph with FID Detector and Methanizer
Generic Instrument Name	Gas Chromatograph
Generic Instrument Description	Instrument separating gases, volatile substances, or substances dissolved in a volatile solvent by transporting an inert gas through a column packed with a sorbent to a detector for assay. (from SeaDataNet, BODC)

Dataset- specific Instrument Name	SRI Instruments 8610C Gas Chromatograph with FID Detector
Generic Instrument Name	Gas Chromatograph
Generic Instrument Description	Instrument separating gases, volatile substances, or substances dissolved in a volatile solvent by transporting an inert gas through a column packed with a sorbent to a detector for assay. (from SeaDataNet, BODC)

Dataset- specific Instrument Name	DIONEX Corporation ICS-2000 Ion Chromatography System
Generic Instrument Name	Ion Chromatograph
Generic Instrument Description	Ion chromatography is a form of liquid chromatography that measures concentrations of ionic species by separating them based on their interaction with a resin. Ionic species separate differently depending on species type and size. Ion chromatographs are able to measure concentrations of major anions, such as fluoride, chloride, nitrate, nitrite, and sulfate, as well as major cations such as lithium, sodium, ammonium, potassium, calcium, and magnesium in the parts-per-billion (ppb) range. (from http://serc.carleton.edu/microbelife/research_methods/biogeochemical/ic)

Dataset-specific Instrument Name	ASI-V Autosampler
Generic Instrument Name	Laboratory Autosampler
Dataset-specific Description	Shimadzu Instruments TOC-Vcph Total Organic Carbon Analyzer with ASI-V Autosampler and TNM Total Nitrogen Analyzer
Generic Instrument Description	Laboratory apparatus that automatically introduces one or more samples with a predetermined volume or mass into an analytical instrument.

Dataset-specific Instrument Name	Shimadzu Instruments TOC-Vcph Total Organic Carbon Analyzer
Generic Instrument Name	Shimadzu TOC-V Analyzer
Dataset-specific Description	Shimadzu Instruments TOC-Vcph Total Organic Carbon Analyzer with ASI-V Autosampler and TNM Total Nitrogen Analyzer
Generic Instrument Description	A Shimadzu TOC-V Analyzer measures DOC by high temperature combustion method.

Dataset-specific Instrument Name	Hach Company DR 2800 Spectrophotometer
Generic Instrument Name	Spectrophotometer
Generic Instrument Description	An instrument used to measure the relative absorption of electromagnetic radiation of different wavelengths in the near infra-red, visible and ultraviolet wavebands by samples.

Dataset-specific Instrument Name	Shimadzu Instruments UV1601 Spectrophotometer
Generic Instrument Name	Spectrophotometer
Generic Instrument Description	An instrument used to measure the relative absorption of electromagnetic radiation of different wavelengths in the near infra-red, visible and ultraviolet wavebands by samples.

Dataset- specific Instrument Name	TNM Total Nitrogen Analyzer
Generic Instrument Name	Total Nitrogen Analyzer
Dataset- specific Description	Shimadzu Instruments TOC-Vcph Total Organic Carbon Analyzer with ASI-V Autosampler and TNM Total Nitrogen Analyzer
Generic Instrument Description	A unit that accurately determines the nitrogen concentrations of organic compounds typically by detecting and measuring its combustion product (NO). See description document at: http://bcodata.whoi.edu/LaurentianGreatLakes_Chemistry/totalnit.pdf

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Deployments

FK190211

Website	https://www.bco-dmo.org/deployment/820900	
Platform	R/V Falkor	
Start Date	2019-02-11	
End Date	2019-03-14	
Description	Start and end port: Manzanillo, Mexico	

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Project Information

Collaborative Research: Microbial Carbon cycling and its interactions with Sulfur and Nitrogen transformations in Guaymas Basin hydrothermal sediments (Guaymas Basin Interactions)

Coverage: Guaymas Basin, Gulf of California, 27.00 N, 111.00W

Description from NSF award abstract:

Hydrothermally active sediments in the Guaymas Basin are dominated by novel microbial communities that catalyze important biogeochemical processes in these seafloor ecosystems. This project will investigate genomic potential, physiological capabilities and biogeochemical roles of key uncultured organisms from Guaymas sediments, especially the high-temperature anaerobic methane oxidizers that occur specifically in hydrothermally active sediments (ANME-1Guaymas). The study will focus on their role in carbon transformations, but also explore their potential involvement in sulfur and nitrogen transformations. First-order research topics include quantifying anaerobic methane oxidation under high temperature, in situ concentrations of phosphorus and methane , and with alternate electron acceptors; sulfate and sulfur-dependent microbial pathways and isotopic signatures under these conditions; and nitrogen transformations in methane-oxidizing microbial communities, hydrothermal mats and sediments.

This integrated biogeochemical and microbiological research will explore the pathways of and environmental controls on the consumption and production of methane, other alkanes, inorganic carbon, organic acids and organic matter that fuel the Guaymas sedimentary microbial ecosystem. The hydrothermal sediments of Guaymas Basin provide a spatially compact, high-activity location for investigating novel modes of methane cycling and carbon assimilation into microbial biomass. In the case of anaerobic methane oxidation, the high temperature and pressure tolerance of Guaymas Basin methane-oxidizing microbial communities, and their potential to uncouple from the dominant electron acceptor sulfate, vastly increase the predicted subsurface habitat space and biogeochemical role for anaerobic microbial methanotrophy in global deep subsurface diagenesis. Further, microbial methane production and oxidation interlocks with syulfur and nitrogen transformations, which will be explored at the organism and process level in hydrothermal sediment microbial communities and mats of Guaymas Basin. In general, first-order research tasks (rate measurements, radiotracer incorporation studies, genomes, in situ microgradients) define the key microbial capabilities, pathways and processes that mediate chemical exchange between the subsurface hydrothermal/seeps and deep ocean waters.

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Funding

Funding Source	Award
NSF Division of Ocean Sciences (NSF OCE)	<u>OCE-1357360</u>

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