Geochemical data supporting from methanogenesis rates in subsurface sediments from R/V JOIDES Resolution IODP-385 drilling expedition in the Guaymas Basin between September and November, 2019

Website: https://www.bco-dmo.org/dataset/991374
Data Type: Cruise Results
Version: 1
Version Date: 2025-01-14

Project
» Pathways and regulation of transformation of low molecular weight carbon compounds in subseafloor sediments from the Guaymas Basin (Gulf of California) (Guaymas Basin Sediments)

Contributors	Affiliation	Role
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Abstract

Deep marine sediments are the largest reservoir of methane on Earth. Yet, the metabolic pathways and activity of methanogenesis in deep, hot sediments remain poorly understood. In this study, we quantified methanogenic activity using five different 14C-labeled substrates, and combined these potential rates with geochemical data to identify the dominant methanogenic pathways and their environmental controls in the subsurface sediments of the Guaymas Basin. Samples were collected during IODP Expedition 385 from September to November 2019. This dataset presents geochemical measurements from four drilling sites, including methane concentrations, methane to higher alkane (C_1/C_2^+) ratios, sulfate, methanol, trimethylamine, dissolved inorganic carbon, ammonium, temperature, water depth, and sediment depth.

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Coverage

Location: Guaymas Basin, Gulf of California
Spatial Extent: **N**:27.637 **E**:-111.48 **S**:27.472 **W**:-111.889
Temporal Extent: 2019-09-15 - 2019-11-15

Dataset Description

Study Results

Thermodynamic calculations and C1/C2+ ratios indicated that methane in the relatively cooler, shallower layers, was predominantly of biogenic origin. Radiotracer experiments provided direct evidence for the coexistence of multiple methanogenic pathways, hydrogenotrophic, acetoclastic, and methylotrophic, across the sediment column. Methanogenic activity from multiple methanogenic pathways occurred over a wide temperature range (3°C to 80°C), highlighting the unexpectedly high metabolic versatility of methanogens in deep, thermally heated sediments. High methanogenesis rates were detected in near-surface sediments driven predominantly by methylotrophic methanogenesis, followed by hydrogenotrophic pathways. However, these rates declined sharply with depth, particularly within the 40–60°C interval, indicating a transition from mesophilic to thermophilic microbial communities, due to rising temperatures, reductions in gene expression, and decreasing microbial cell densities. Methylotrophic methanogenesis remained detectable down to 320 meters below the seafloor and was the dominant methane-producing pathway at temperatures up to 60°C. In sediments increasingly influenced by sill intrusions, hydrogenotrophic and acetoclastic methanogenesis became the predominant modes of methane production.

Methanogenic activity rates from multiple substrates at 80°C were comparable to rates in near-surface sediments. This deep, hot activity is attributed to the presence of active microbial biomass and the enhanced reactivity and bioavailability of organic matter in deep, hydrothermally-heated sediments, which provided abundant substrates for methanogenesis. These findings expand the current understanding of methanogenesis in the deep biosphere and reveal the discovery of the contemporaneous activity of multiple methanogenic pathways in deep, hydrothermally-influenced sediments.

See related datasets for all data referenced in these findings.

Methods & Sampling

Subsurface sediment samples were collected from four drilling sites in the Guaymas Basin, Gulf of California, during IODP Expedition 385 “Guaymas Basin Tectonics and Biosphere” using the research vessel R/V JOIDES Resolution. Rates of methanogenesis were determined at four sites. Sites 1545 (27º38.230’N, 111º53.329’W) and 1546 (27º37.884’N, 111º52.781’W) were located roughly 52 km and 51 km, respectively, northwest of the axial graben of the northern spreading segment. Both sites are highly sedimented and a 75-meter thick inactive (~thermally equilibrated) basaltic/doleritic/gabbroic sill was present at site 1546 between ~355 to 431 meters below the seafloor (mbsf). Site U1545 is considered a reference site since it was free of sill intrusions and unaffected by active hydrothermal circulation. Samples to determine methanogenesis rates were collected from the "B" hole at every site (during IODP expeditions, several, closely-spaced holes are drilled at each site so that sufficient sediment is available for all of the proposed work.

The geothermal gradient at hole U1545B was 227ºC/km. The geothermal gradient at site U1546B was similar to that measured in hole U1545B, 221ºC/km, since the sill at 1546B was thermally equilibrated. Sites U1547 (27º30.413’N, 111º40.734’W) was located inside the periphery of an active, sill-associated hydrothermal mound located about 27 km northwest of the axial graben of the northern spreading segment. Temperatures in hole U1547B exceeded 50°C in the upper 50 mbsf. The geothermal gradient at this site was between 511ºC to 960ºC/km. Site U1549 was located near a cold seep sustained by a deeply buried, thermally equilibrated sill intrusion at several hundred meters depth. The geothermal gradient at hole U1549B was 194ºC/km.

Sediment samples were collected using an advanced piston coring system (APC) and a half-length APC (Teske et al. 2021). After retrieval on deck, sediment cores were sectioned in a designated core cutting area. Approximately 0.5 cm of the outer sediment layer was carefully removed to minimize potential contamination. For analysis of hydrocarbon gases, ~ 5 mL sediment was transferred into 21.5 mL clean, pre-combusted glass vials using a cut-off syringe. The vials were sealed immediately with polytetrafluoroethylene septa and aluminum caps and were incubated for 30 min at 70°C to allow dissolved hydrocarbon gases to equilibrate with the headspace before analysis, as described in Teske et al. (2021). For molecular hydrogen analysis, duplicate 3 mL sediment samples were collected immediately after core recovery using a cut-off syringe. The subsample was transferred into 21.5 mL glass vials and incubated and processed according to the procedures of Lin et al. (2012).

Data describing *in situ* geochemistry, pressure, and temperature were generated shipboard, as outlined in Teske et al. (2021). Porewater for geochemical analyses was extracted from subsurface sediments using a shipboard squeezing apparatus, following established protocols (Manheim and Sayles 1974, Teske et al. 2021). To prevent contamination, ~0.5 cm of the outer sediment was carefully removed prior to transferring 150–300 cm^3 of uncontaminated interior material into a titanium squeezing vessel. The vessel was placed in a Carver hydraulic press (Manheim and Sayles 1974), and the expressed porewater was filtered immediately through a 0.2 µm polyethersulfone membrane. Filtrates was collected in HCl-washed syringes and subsampled into acid-cleaned high-density polyethylene vials for onboard determinations of sulfate and ammonium, with samples stored at 4 °C following fixation (acidification with trace metal grade nitric acid for sulfate and phenol solution for ammonium).

Dissolved inorganic carbon (DIC) was collected into 2 mL glass vials and preserved with 10 µL of saturated $HgCl_2$ for shore-based analysis. For acetate analysis, 2 mL porewater subsamples were preserved immediately after collection and stored frozen at −20 °C in pre-combusted glass vials with Teflon-coated screw caps (Heuer et al. 2009). Samples for methanol and trimethylamine (TMA) measurements were transferred immediately into pre-combusted and N_2-purged 20 mL glass vails that were sealed with gas-tight butyl rubber stoppers, stored at −80°C until analysis.

Hydrocarbon gas analysis was performed by extracting about 5 mL of headspace gas from the 21.5 mL vials using a gas-tight syringe and directly injecting it into an Agilent 7890A gas chromatograph with a flame ionization detector (FID) (Teske et al. 2021b). Sulfate concentrations were measured by ion chromatography using a Metrohm 850 Professional IC system (Murray et al. 2000, Teske et al. 2021b). Ammonium concentrations were determined by a colorimetric method involving phenol diazotization followed by oxidation with bleach (Clorox) to produce a blue color, measured at 640 nm using a Cary Series 100 UV-Vis spectrophotometer (Agilent Technologies) (Solorzano 1969, Teske et al. 2021b). Samples were diluted beforehand to keep ammonium below 1000 µM. Total alkalinity was measured by Gran titration with a Metrohm 794 Basic Titrino autotitrator (Gieskes et al. 1991, Teske et al. 2021b). Methanol and TMA (~5 mL) in porewater were simultaneously quantified using a purge and trap pre-treatment system coupled with a gas chromatograph (Jiang et al. 2024). An Agilent Technologies Inc. (USA) 8890 gas chromatograph equipped with a purge and trap system (Acrichi PTC, Beiging Juxin Zhuifeng Technology, Ltd, China), an Agilent splitter based on proprietary capillary flow technology installed between the capillary column (HP-PLOT Q; 30m x 0.32mm, Agilent Technologies Inc., USA) and detectors, and dual nitrogen phosphorus (methylamine) and flame ionization (methanol) detectors was used for this analysis. The method followed the protocol outlined in Jiang et al. The molar fraction of H_2 in headspace gas was quantified using a Reduction Gas Analyzer (Peak Performer 1, Peak Laboratories LLC, USA). All geochemical data were retrieved from the IODP database (https://web.iodp.tamu.edu/OVERVIEW/) (Teske et al. 2021b), while DIC concentrations in interstitial waters were reported by Torres and Kim (2022).

BCO-DMO Processing Description

- Imported "OCE-2023575_Methanogenesis_geochemistry_csv.csv" into BCO-DMO system
- Created new field "methane_flag" and moved all "b.d.l." values to this new flag field and removed them from the "methane" field
- Created new field "methane_higher_alkane_ratio_bdl_flag" and moved all "b.d.l." values to this new flag field and removed them from the "methane/ higher alkanes" field
- Renamed fields to comply with BCO-DMO naming conventions, removing units, special characters, and spaces
- Exported file as "991374_v1_methanogenesis_geochem_guaymas_basin"

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Related Publications

Bowles, M. W., Samarkin, V. A., & Joye, S. B. (2011). Improved measurement of microbial activity in deep-sea sediments at in situ pressure and methane concentration. Limnology and Oceanography: Methods, 9(10), 499–506. doi:10.4319/lom.2011.9.499
Methods

Jiang, F., Zhou, Z., Wang, J.-Y., Guan, W.-J., Han, L.-G., Lin, X.-B., & Zhuang, G.-C. (2024). Simultaneous determination of seawater trimethylamine and methanol by purge and trap gas chromatography using dual nitrogen-phosphorus detector and flame-ionization detector. Frontiers in Marine Science, 11. https://doi.org/10.3389/fmars.2024.1356801
Methods

Krzycki, J. A., Kenealy, W. R., DeNiro, M. J., & Zeikus, J. G. (1987). Stable Carbon Isotope Fractionation by Methanosarcina barkeri during Methanogenesis from Acetate, Methanol, or Carbon Dioxide-Hydrogen. Applied and Environmental Microbiology, 53(10), 2597–2599. https://doi.org/10.1128/aem.53.10.2597-2599.1987
Methods

Manheim, F.T., and Sayles, F.L., 1974. Composition and origin of interstitial waters of marine sediments, based on deep sea drill cores. In Goldberg, E.D. (Ed.), The Sea (Vol. 5): Marine Chemistry: The Sedimentary Cycle:

New York (Wiley), 527–568. https://pubs.usgs.gov/publication/70207491
Methods

Murray, R. W., Miller, D. J., & Kryc, K. A. (2000). Analysis of Major and Trace Elements in Rocks, Sediments, and Interstitial Waters by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES). Ocean Drilling Program Technical Notes. https://doi.org/10.2973/odp.tn.29.2000
Methods

Solórzano, L. (1969). Determination of ammonia in natural waters by the phenolhypochlorite method 1 1.This research was fully supported by U.S. Atomic Energy Commission Contract No. ATS (11-1) GEN 10, P.A. 20. Limnology and Oceanography, 14(5), 799–801. doi:10.4319/lo.1969.14.5.0799
Methods

Summons, R. E., Franzmann, P. D., & Nichols, P. D. (1998). Carbon isotopic fractionation associated with methylotrophic methanogenesis. Organic Geochemistry, 28(7–8), 465–475. https://doi.org/10.1016/s0146-6380(98)00011-4 https://doi.org/10.1016/S0146-6380(98)00011-4
Methods

Teske, A., Lizarralde, D., Höfig, T. W., Aiello, I. W., Ash, J. L., Bojanova, D. P., Buatier, M. D., Edgcomb, V. P., Galerne, C. Y., Gontharet, S., Heuer, V. B., Jiang, S., Kars, M. A. C., Khogenkumar Singh, S., Kim, J., Koornneef, L. M. T., Marsaglia, K. M., Meyer, N. R., Morono, Y., … Zhuang, G. (2021). Expedition 385 summary. Guaymas Basin Tectonics and Biosphere. Internet Archive. https://doi.org/10.14379/iodp.proc.385.101.2021
Methods

Torres, M. E., & Kim, J. (2022). Data report: concentration and carbon isotopic composition in pore fluids from IODP Expedition 385. Guaymas Basin Tectonics and Biosphere. Internet Archive. https://doi.org/10.14379/iodp.proc.385.201.2022
Methods

Whiticar, M. J. (1999). Carbon and hydrogen isotope systematics of bacterial formation and oxidation of methane. Chemical Geology, 161(1–3), 291–314. https://doi.org/10.1016/s0009-2541(99)00092-3 https://doi.org/10.1016/S0009-2541(99)00092-3
Methods

Xu, L., Zhuang, G., Montgomery, A., Liang, Q., Joye, S. B., & Wang, F. (2020). Methyl-compounds driven benthic carbon cycling in the sulfate-reducing sediments of South China Sea. Environmental Microbiology, 23(2), 641–651. Portico. https://doi.org/10.1111/1462-2920.15110
Methods

Zhuang, G., Montgomery, A., Samarkin, V. A., Song, M., Liu, J., Schubotz, F., … Joye, S. B. (2019). Generation and Utilization of Volatile Fatty Acids and Alcohols in Hydrothermally Altered Sediments in the Guaymas Basin, Gulf of California. Geophysical Research Letters, 46(5), 2637–2646. doi:10.1029/2018gl081284 https://doi.org/10.1029/2018GL081284
Results

Zhuang, G., Montgomery, A., Sibert, R. J., Rogener, M., Samarkin, V. A., & Joye, S. B. (2018). Effects of pressure, methane concentration, sulfate reduction activity, and temperature on methane production in surface sediments of the Gulf of Mexico. Limnology and Oceanography, 63(5), 2080–2092. Portico. https://doi.org/10.1002/lno.10925
Methods

Zhuang, G.-C., Elling, F. J., Nigro, L. M., Samarkin, V., Joye, S. B., Teske, A., & Hinrichs, K.-U. (2016). Multiple evidence for methylotrophic methanogenesis as the dominant methanogenic pathway in hypersaline sediments from the Orca Basin, Gulf of Mexico. Geochimica et Cosmochimica Acta, 187, 1–20. https://doi.org/10.1016/j.gca.2016.05.005
Methods

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Parameters

Parameter	Description	Units
site	Drilling site identifier	unitless
hole	Hole Designation Letter, where "B" represents the second hole drilled at the site location	unitless
latitude	Sampling latitude	decimal degrees
longitude	Sampling longitude	decimal degrees
water_depth	Water column depth	m
sediment_depth	Depth below the seafloor in meters	mbsf
temperature	Temperature	unitless
methane	Methane concentration in interstitial water	mM
methane_flag	Flag field where "b.d.l." = below detection limit	unitless
methane_higher_alkane_ratio	Molar ratio of methane (C_1) concentration to the summed concentrations of higher alkanes (C_2^+; ethane and heavier hydrocarbons) in interstitial water gas	molar ratio (C_1/C_2^+)
methane_higher_alkane_ratio_bdl_flag	Flag field where "b.d.l." = below detection limit	unitless
sulfate	Sulfate concentration in interstitial water	mM
dissolved_inorganic_carbon	Dissolved inorganic carbon concentration in interstitial water	mM
ammonium	Ammonium concentration in interstitial water	mM
methanol	Methanol concentration in interstitial water	µM
trimethylamine	Trimethylamine concentration in interstitial water	nM

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Instruments

Dataset-specific Instrument Name	Advanced piston coring system (APC)
Generic Instrument Name	Advanced Piston Corer
Dataset-specific Description	Sediment samples were collected using an advanced piston coring system (APC) and a half-length APC (Teske et al. 2021).
Generic Instrument Description	The JOIDES Resolution's Advanced Piston Corer (APC) is used in soft ooze and sediments. The APC is a hydraulically actuated piston corer designed to recover relatively undisturbed samples from very soft to firm sediments. More information is available from IODP (PDF).

Dataset-specific Instrument Name	Peak Laboratories LLC (USA) Peak Performer 1 Reduction Gas Analyzer
Generic Instrument Name	Gas Analyzer
Dataset-specific Description	The molar fraction of H2 in headspace gas was quantified using a Reduction Gas Analyzer (Peak Performer 1, Peak Laboratories LLC, USA).
Generic Instrument Description	Gas Analyzers - Instruments for determining the qualitative and quantitative composition of gas mixtures.

Dataset-specific Instrument Name	Agilent Technologies Inc. (USA) 8890 gas chromatograph
Generic Instrument Name	Gas Chromatograph
Dataset-specific Description	An Agilent Technologies Inc. (USA) 8890 gas chromatograph equipped with a purge and trap system (Acrichi PTC, Beiging Juxin Zhuifeng Technology, Ltd, China), an Agilent splitter based on proprietary capillary flow technology installed between the capillary column (HP-PLOT Q; 30m x 0.32mm, Agilent Technologies Inc., USA) and detectors, and dual nitrogen phosphorus (methylamine) and flame ionization (methanol) detectors was used for this analysis.
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	Agilent 7890A gas chromatograph with a flame ionization detector (FID)
Generic Instrument Name	Gas Chromatograph
Dataset-specific Description	Hydrocarbon gas analysis was performed by extracting about 5 mL of headspace gas from the 21.5 mL vials using a gas-tight syringe and directly injecting it into an Agilent 7890A gas chromatograph with a flame ionization detector (FID) (Teske et al. 2021b).
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	Metrohm 850 Professional IC system
Generic Instrument Name	Ion Chromatograph
Dataset-specific Description	Sulfate and chloride concentrations were measured by ion chromatography using a Metrohm 850 Professional IC system (Murray et al. 2000, Teske et al. 2021b).
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	Cary Series 100 UV-Vis spectrophotometer (Agilent Technologies)
Generic Instrument Name	Spectrophotometer
Dataset-specific Description	Ammonium concentrations were determined by a colorimetric method involving phenol diazotization followed by oxidation with bleach (Clorox) to produce a blue color, measured at 640 nm using a Cary Series 100 UV-Vis spectrophotometer (Agilent Technologies) (Solorzano 1969, Teske et al. 2021).
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	Metrohm 794 Basic Titrino autotitrator
Generic Instrument Name	Titrator
Dataset-specific Description	Total alkalinity was measured by Gran titration with a Metrohm 794 Basic Titrino autotitrator (Gieskes et al. 1991, Teske et al. 2021).
Generic Instrument Description	Titrators are instruments that incrementally add quantified aliquots of a reagent to a sample until the end-point of a chemical reaction is reached.

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Deployments

IODP-385

Website	https://www.bco-dmo.org/deployment/869491
Platform	R/V JOIDES Resolution
Start Date	2019-09-16
End Date	2019-11-16
Description	Guaymas Basin Tectonics and Biosphere - International Ocean Discovery Program Expedition 385, General information: https://iodp.tamu.edu/scienceops/expeditions/guaymas_basin_tectonics_bio...

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Project Information

Pathways and regulation of transformation of low molecular weight carbon compounds in subseafloor sediments from the Guaymas Basin (Gulf of California) (Guaymas Basin Sediments)

Coverage: Guaymas Basin (Gulf of California)

NSF Award Abstract:
This research will explore carbon cycling in one of the largest carbon reservoirs on Earth, marine sediments, located at bottom of the ocean. This carbon is recycled gradually over time through interacting geological, chemical, and biological processes. This project will document how each of these processes transforms carbon in marine sediments from the Guaymas Basin (Gulf of California). This setting offers the chance to study carbon cycling across a broad range of chemical and temperature gradients, providing an opportunity to tease apart the factors regulating carbon cycling in marine sediments. This project will investigate the role of ocean sediments in the global carbon cycle. These research objectives represent key science priorities in a time of global environmental change. For outreach activities, the scientist, in collaboration with Jim Toomey Education, would continue the "Adventures of Zack and Molly" educational video series. In this instance, the video would document results from this study and its broader significance. The scientist also would create a learning guide for teachers. Both the video and the learning guide would be disseminated to educators. One graduate and one undergraduate student would be supported and trained as part of this project.

Subsurface sediments in the Guaymas Basin (Gulf of California) offer an accessible window for investigating carbon cycling in a dynamic, yet tractable, marine environment. This work will study how heating of subsurface sediments affects the production, consumption, and fate of low molecular weight dissolved organic carbon. The research will track the fate of key carbon species – including formate, acetate, and methanol – as they are processed through a gauntlet of microbial-mediated processes. Samples were collected during Expedition 385 of the International Ocean Discovery Program in September-October 2019. Some experiments were conducted on the research vessel and additional experiments will be conducted in the laboratory. The study will constrain the magnitudes of transformation and the fate of low molecular weight carbon substrates using a combination of direct rate, pool size, and stable isotopic measurements coupled to thermodynamic modeling and probative laboratory experiments. Key topics for investigation include: (1) What is the dominant production mode for organic compounds in subsurface sediments? (2) What are the dominant pathways of methanogenesis along geochemical and temperature gradients? (3) What are the temperature limits of microbially-driven carbon cycling processes? (4) How does the fate of organic compounds change along geochemical and/or temperature gradients?

This award reflects NSF's statutory mission and has been deemed worthy of support through evaluation using the Foundation's intellectual merit and broader impacts review criteria.

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Funding

Funding Source	Award
NSF Division of Ocean Sciences (NSF OCE)	OCE-2023575

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