Geochemical data collected from Whillans Subglacial Lake sediment cores in West Antarctica

Website: https://www.bco-dmo.org/dataset/822288
Version: 0
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Project
» Microbial carbon cycling beneath the West Antarctic Ice Sheet (SLW C Cycling)

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

Contributors	Affiliation	Role
Michaud, Alexander	Montana State University	Principal Investigator
Priscu, John C.	Montana State University	Contact
York, Amber D.	Woods Hole Oceanographic Institution (WHOI BCO-DMO)	BCO-DMO Data Manager

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Coverage

Spatial Extent: **N**:-83.79 **E**:-131.77 **S**:-85.02 **W**:-155.33

Dataset Description

These are the geochemical data collected from Whillans Subglacial Lake sediment cores. Values were determined using a number of analytical methods, please see the “Analytical Methods” section of the methodology.

Methods & Sampling

Location:
Samples were collected from Subglacial Lake Whillans, at the lower portion of the Whillans Ice Stream, West Antarctica. Samples were collected through a 800m borehole created with a clean, hot water drill system. Coordinates: 85°1'12"S and 83°47'24"S Latitude; 155°19'48"W and 131°46'12"W Longitude.

Detailed geochemical methods can be found at http://www.geosociety.org/datarepository/2016/2016110.pdf and in the Michaud et al., 2017, and Vick-Majors et al., 2016.

Design description:

A hot water drilling system was used between 23–27 January 2013 to melt through the ~801 m thick ice sheet, creating an access borehole (minimum diameter ~60 cm) for direct sampling and to conduct in situ

measurements of the SLW water column and sediments. Microbial cells in the drilling water and on exposed surfaces of the hose, cables, and deployed equipment were reduced and killed through the use of four complementary technologies: (1) filtration, (2) ultraviolet irradiation, (3) pasteurization, and (4) disinfection with 3% w/v H_2O_2. The drilling water, derived from the overlying ice sheet, was continuously circulated through a water treatment system that removed micron and sub-micron sized particles (>0.2 µm), irradiated the drilling water with two germicidal wavelengths of ultraviolet radiation (185 nm ~40,000 µW s^{-1} cm^{-2} and 254 nm ~175,000 µW s^{-1} cm^{-2}), and pasteurized the water at 90 °C to reduce the viability of persisting microbial contamination. Ports were plumbed along the system's flow path, allowing discrete water samples to be obtained before and after each stage. The drill hose and instrument cables were deployed at a rate no greater than 1 m s^{-1} through a custom borehole collar that contained 12 amalgam pellet ultraviolet lamps, providing a cumulative germicidal ultraviolet dosage of at least 40,000 µW s^{-1} cm^{-2} (Arapahoe SciTech). All borehole sampling tools and instruments were spray-saturated with 3% w/v H_2O_2 and staged in sealed polyethylene bags until tool deployment. Single-use protective apparel (Tyvek) was worn by all personnel during borehole science operations. The efficacy of the clean access technology and procedures were tested thoroughly before use in the field.

Sampling description

Water samples were collected and brought to the surface in Niskin bottles. Particulate matter for DNA sequence analyses was collected on 10.0, 3.0, 0.8, and 0.2 micron filters by an in situ filtration unit. Sediment samples were collected and brought to the surface using a shallow sediment multicorer.

Analytical Methods:

Sample Processing

SLW sediment porewater samples were collected from multicore 3B (MC-3B) using a Rhizon porewater sampler (Rhizosphere). Rhizon porewater samplers were prepared by soaking in MilliQ (18.2 MΩ) water prior to installation through a pre-drilled hole in the sediment core liner. A 10 mL syringe was attached to the outlet and the plunger locked to maintain a vacuum. After 14 h of porewater extraction through the 0.2 µm filter on the end of the Rhizon, the porewater was dispensed into cleaned bottles/vials (Major ions: 10% HCl acid washed and 6 MilliQ rinses; Trace elements: 10% trace metal grade HNO_3 acid washed and 6 MilliQ rinses; Water stable isotopes: new, filled with no headspace). SLW water samples for major ion analysis were filtered through a 0.4 µm polycarbonate filter (Whatman) and TE/REE samples were filtered through a 0.2 µm PTFE membrane syringe filter. The filtrate, or raw water in the case of the water stable isotope samples, was dispensed into a bottle cleaned as described above. Major ion and TE samples were frozen at -20°C and shipped to Montana State University and University of Venice, respectively, for analysis. Stable isotope samples were stored at 4°C and were shipped to University of Washington-Seattle for analysis. Procedural blanks were handled and processed in parallel to SLW samples. Briefly, MilliQ water from McMurdo Station was taken into the field in a 10% HCl acid washed and 6-time MilliQ rinsed HDPE bottle and was run through the same sampling set up for SLW water samples. For the porewater procedural blanks, rhizons were inserted into the MilliQ bottle and ~5 mL of water was pulled through the Rhizon into the syringe and allowed to reside in the syringe for 14 h to replicate syringe residence time. Procedural blank water was then dispensed into the three sample containers for subsequent analysis as described above.

Major Ion Analysis

Major anions and cations were measured using ion chromatography (Metrohm) with an ASupp 5 anion and C4 cation column (Metrohm). Porewater samples were diluted in MilliQ prior to analysis. The dilution factor varied by element/compound and also downcore depending on total solute concentration. The dilution factors that were used are as follows: sodium and chloride 200-250 fold, sulfate, potassium, magnesium and calcium, 15-25 fold. Sample concentrations were calculated from a 5-point standard curve for each analyte. Procedural blank samples were analyzed in parallel with samples and were negligible, but subtracted from sample concentrations, such that the values presented are blank-corrected. Precision for a 0.1 mg/L standard (2 to 8 ueq/L over the range of elements/compounds measured) was as follows: sodium ± 1%, potassium ± 4%, magnesium ± 6%, calcium ± 6%, chloride ± 1% and sulfate ± 1%. Bicarbonate concentration in the lake water was determined by infrared gas analysis as described in Christner et al., (2014). Bicarbonate concentration in the porewater samples was determined as the difference between the sum of the cations and the sum of the anions.

Silicon Analysis

Silicon was determined using the Molybdenum-blue colorimetric assay. The protocol was modified for a 1mL final reaction volume and the ammonium molybdate amount was doubled. Porewater was extracted from the sediment by transferring ~5 g of sediment to a 15 mL clean centrifuge tube and spinning at 4500 xg for 30 mins. The porewater was pipetted from the sediment surface and 0.22 µm syringe filtered through Durapore PVDF syringe filters (Millipore). Absorbance was measured at 810 nm. Filter blanks were analyzed in parallel and silicon from filtration was negligible. Concentrations in samples were determined from a 5-point standard curve made from a certified, NIST-traceable Si standard (Sigma-Aldrich). The methodological limit of detection was 0.5 µM following EPA methods.

Stable Isotope Analysis

Stable isotope measurements (δD and δ18O) on water samples were made using a Picarro cavity ring-down laser spectrometer at the Isolab (University of Washington, Seattle). The porewater samples (100 µL) were analyzed in random order (i.e., non-sequential depth and two separate sets of analyses were conducted for each depth on different days). There was good agreement between the values generated in the two separate runs. The results reported are the average of the two runs using standard δ notation in per mille relative to Vienna Standard Mean Ocean Water (VSMOW). Precision was ± 0.1 ‰.

Trace Element Analysis

Trace element measurements were performed by inductively coupled plasma sector field mass spectrometer (ICP-SFMS; Element2, Finnigan-MAT, Bremen, Germany). The instrument was installed in a dedicated laboratory with the sample introduction area protected by a laminar flow cabinet. The sample introduction system utilized a desolvation unit (Aridus, Cetac Technologies, Omaha, NE, USA) coupled with a PFA µ-flow nebulizer (100 µL min-1); the sample flows in a teflon spray chamber heated up to 95°C to prevent droplet accumulation, then it is swept by an Ar flow into a semi-permeable membrane (heated up to 165°C) to significantly reduce the formation of oxides. The introduction system was connected to the ESI SC2-E2 autosampler (Elemental Scientific, Omaha, NE, USA). The sample introduction system and the autosampler were maintained and handled under a laminar flow hood. Intensity optimization was carried out daily, using a tuning solution of ultrapure water containing 1 ng/ml of In. An accurate mass calibration was performed immediately prior to analysis in low, medium and high resolution mode using a solution containing elements with m/z values covering the whole mass range of interest. The accuracy of the measurements was determined using a certified reference material (TM-RAIN95) in which the trace element concentrations were measured. The vanadium concentration in TM-RAIN95 is 12.5 nM and the precision for its determination was ± 6%.

Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray (EDX) Spectroscopy

Bulk sediment was wet sieved sequentially through 1 mm, 0.589 mm, 250 µm, 125 µm, and 63 µm sieves. The size fractions were dried for 3 h at 90°C. Grains from the 63 – 125 µm fraction were sparsely distributed onto black, sticky carbon tape, mounted on a 50 mm SEM stub. The grains were coated with iridium using an Emitech K575x sputter coater (Quorum Technologies) operated at 20 mA for 60 s resulting in an Ir coating of approximately 10 nm. Mineral grain elemental composition was determined on a JEOL JSM-6100 SEM equipped with EDX spectroscopy and a NORAN SiLi detector. Elemental mapping was conducted using a RONTEC XFlash 1000 x-ray detector. Rontec electronics and software were used to collect elemental spectra and identify peaks. EDX was performed at 20 kV and spectra were identified using a dichotomous key. Two hundred and six grains from the 63 – 125 µm fraction were analyzed for their elemental composition.

Methane Geochemistry

Sediment from a gravity core (MC-2A) was sampled every 2 cm by extrusion and subsampling of each newly exposed layer. Sediment subsamples for methane (CH4) were collected using a sterile cut-off 5 ml syringe and immediately placed into 20 ml sterile serum vials and stoppered with a sterile butyl rubber stopper, then crimped with an aluminum cap. Three empty vials were sealed in the field to capture atmospheric air as procedural blanks. Ten ml of 2.5% NaOH was added by sterile syringe to each sample vial and the three blanks, stopping biological activity and creating a pressurized headspace within each vial. A CH4 sample from the SLW water column was collected from cast 1 from a Niskin bottle by placing the tube to the bottom of the serum vial and filling from top to bottom. The water sample was fixed with Lugol’s solution to prevent biological

activity. All vials were stored inverted at 4°C for transport back to Montana State University (MSU) for CH4 quantification. Headspace CH4 was quantified on a Hewlett-Packard 5890 Series II gas chromatograph (GC) equipped with a flame ionization detector (FID) with a detection limit of 3 nM for water column samples and 190 nM for the sediment samples. Headspace gas was introduced to the GC using a 10-port injection valve configured for back flushing of a precolumn (25 cm x 0.32 cm OD, packed with Porapak-T 80/100 mesh) to prevent water vapor from reaching the analytical columns. The vial overpressure was used to flush and fill a 1 cm3 sample loop using a syringe needle inlet; measured laboratory air temperature and pressure were used to calculate the total moles of gas contained within the loop, assuming gas ideality. Gases were separated on two analytical columns in series (both 183 cm x 0.32 cm OD, packed with Chromosorb 102 80/100 mesh and Porapak-Q 80/100 mesh, respectively). The columns were maintained at 55°C and the FID at 240°C. The carrier gas was an ultra-high purity N2, which was further purified through Molecular Sieve 5A, activated charcoal and an O2 scrubber. The carrier flow was 30 mL min-1; under these conditions, CH4 eluted to the FID at 1.97 min. Instrument calibration was performed using certified 500 and 51 ppmv CH4 in air standards (Air Liquide; ±1% accuracy), and volumetric dilutions thereof into carrier N2. Dissolved CH4 concentrations were calculated using Henry's Law based on measured headspace mole fractions and Bunsen solubility coefficients estimated from temperature and sample salinity (including added NaOH). Porewater volumes were determined from mass loss after drying the sediment at 95°C until the mass stopped decreasing (~24h), and dry sediment volume was similarly determined assuming a density of 2.60 g cm-3 for the sedimentary particles. The total volume of the vials was determined weighing the vials with sediment and NaOH fixative, then completely filling the headspace with deionized water and weighing again. The headspace volume was determined by difference. The extent of pressurization of the headspace was determined from total headspace volume and the volume of NaOH solution added. The total CH4 within each vial, after correction for the small amount of CH4 present in the headspace air when originally sealed (characterized by the blank vials), was then used to determine the initial CH4 concentration of the porewater.

Gravity core MC-3A was collected from SLW, capped and immediately frozen (-20°C). The frozen core was returned to MSU and thawed at 4°C overnight in a class 1000 clean, cold room in the MSU SubZero Science and Engineering Facility. The core was extruded and cut every 2 cm and sediment for CH4 stable isotope analysis was subsampled and fixed using the same method as for CH4 concentration analysis from MC-2A described above. One ml of room temperature headspace gas from the fixed sediment vials was transferred to a gas-tight laminated foil bag using a gas-tight, glass syringe and diluted 1:100 with CH4-free (zero grade) air. The bag was connected to the inlet of a Picarro G2201-i Cavity Ring-Down Spectrometer (CRDS) specific for high-precision concentration and δ13C analyses of CH4. Sample was introduced to the instrument at a flow rate of 100 ml min-1; δ13C-CH4 values were determined using factory calibrations and were averaged over ≥30 s of 1 Hz measurements. Between samples, atmospheric air was measured for at least 5 min to ensure lack of instrument drift. The δD-CH4 values were measured at the University of California Davis Stable Isotope Facility (UCD-SIF) using a PreCon concentration system (ThermoScientific) in line with a Delta V plus isotope ratio mass spectrometer (ThermoScientific). Two δ13C-CH4 samples (MC-3A samples from 18-20 cm and 34-36 cm depths) were also run at UCD-SIF to compare their independent results with our values obtained on the Picarro CRDS. There was a <4% difference in the δ13C-CH4 values reported from the two methods. The carbon and hydrogen stable isotope ratios are reported in δ-notation (δ13C, δD) with respect to VPDB and VSMOW standards, respectively. The running average (with depth) of the CH4 concentration and isotope values was calculated in SigmaPlot version 11 using a locally estimated scatterplot smoothing (loess) function with smoothing parameters set to first degree polynomial and a sampling frequency of 0.45, which determines the number of local data points used in the weighted regression carried out by the loess smoothing function.

Sediments used for dissolved NH4+ concentration measurements were collected from MC-3A. The sediment was transferred to acid washed (10% HCl), ultra-pure water-rinsed (6X), combusted (4h at 450 °C) glass vials with polytetrafluoroethylene lined caps, frozen at -20 °C and thawed prior to analysis. Sediments were transferred from the glass vials to acid washed and ultra-pure water rinsed 50 mL conical centrifuge tubes and centrifuged at 3500 x g for 20 minutes. The supernatant was transferred to acid washed and ultra-pure water rinsed 15 ml conical centrifuge tubes and spun for an additional 20 min at 4500 x g to pellet fine particulates. The clean supernatant from the 15 mL centrifuge tube was transferred to an acid washed and ultra-pure water rinsed glass vial. The supernatant was diluted (1:10) to a final volume of 5 mL with ultra-pure water for colorimetric analysis.

Particulate organic carbon and nitrogen values were determined with an elemental analyzer. Acetate, formate and oxalate concentrations were determined using ion chromatography.

Data Processing Description

BCO-DMO Data Manager Processing Notes:
* Extracted data submitted in Excel file "/data302/SLW_C_Cycling/orig/michaud_20200316/BCO-DMO_CDEBI Fellowship data.xlsx" to csv
* 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.
* Depth value "Lake" discussed with submitter. Value "Lake" changed to 0 so column could be typed as numeric.

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Related Publications

Michaud, A. B., Skidmore, M. L., Mitchell, A. C., Vick-Majors, T. J., Barbante, C., Turetta, C., ... Priscu, J. C. (2016). Solute sources and geochemical processes in Subglacial Lake Whillans, West Antarctica. Geology, 44(5), 347–350. doi:10.1130/g37639.1 https://doi.org/10.1130/G37639.1
Results

Vick-Majors, T. J., Mitchell, A. C., Achberger, A. M., Christner, B. C., Dore, J. E., ... Michaud, A. B. (2016). Physiological Ecology of Microorganisms in Subglacial Lake Whillans. Frontiers in Microbiology, 7. doi:10.3389/fmicb.2016.01705
Results

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Related Datasets

IsSupplementedBy

Michaud, A. (2020) **pmoA sequences amplified from DNA extracted from Whillans Subglacial Lake sediment in West Antarctica.** Biological and Chemical Oceanography Data Management Office (BCO-DMO). (Version 1) Version Date 2020-09-02 http://lod.bco-dmo.org/id/dataset/823123 [view at BCO-DMO]
Relationship Description: Related proA sequences (in GenBank) from the same samples.

IsSupplementTo

Michaud, A. (2020) **pmoA sequences amplified from DNA extracted from Whillans Subglacial Lake sediment in West Antarctica.** Biological and Chemical Oceanography Data Management Office (BCO-DMO). (Version 1) Version Date 2020-09-02 http://lod.bco-dmo.org/id/dataset/823123 [view at BCO-DMO]

References

Priscu J (2019). Geomicrobiology of Antarctic Subglacial Environments - Subglacial Lake Whillans. GBASE2020_part2. Version 1.1. SCAR - Microbial Antarctic Resource System. http://mars.biodiversity.aq/resources/198

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Parameters

Parameter	Description	Units
Depth	Sample depth in centimeters below the lake water-sediment interface.	centimeters (cm)
d18O_H2O	Isotope ratio of 18O to 16O of water in Whillans Subglacial Lake sediment porewater	permil (0/00)
dD_H2O	Isotope ratio of 2H to 1H of water in Whillans Subglacial Lake sediment porewater	permil (0/00)
Chloride	Chloride concentration in lake sediment porewater	milliequivalents per liter (meq/L)
Sulfate	Sulfate concentration in lake sediment porewater	milliequivalents per liter (meq/L)
Sodium	Sodium concentration in lake sediment porewater	milliequivalents per liter (meq/L)
Potassium	Potassium concentration in lake sediment porewater	milliequivalents per liter (meq/L)
Calcium	Calcium concentration in lake sediment porewater	milliequivalents per liter (meq/L)
Magnesium	Magnesium concentration in lake sediment porewater	milliequivalents per liter (meq/L)
Bicarbonate	Bicarbonate concentration in lake sediment porewater (calculated by charge balance)	milliequivalents per liter (meq/L)
Silicon	Silicon concentration in lake sediment porewater	micromolar (uM)
Vanadium	Vanadium concentration in lake sediment porewater	micromolar (uM)
Methane	Methane concentration in lake sediment porewater	micromolar (uM)
dD_CH4	Isotope ratio of 2H to 1H of methane in Whillans Subglacial Lake sediment porewater	permil (0/00)
d13C_CH4	Isotope ratio of 13C to 12C of methane in Whillans Subglacial Lake sediment porewater	permil (0/00)

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Project Information

Microbial carbon cycling beneath the West Antarctic Ice Sheet (SLW C Cycling)

Coverage: 800 m beneath the Whillans Ice Stream (84.24°S, 153.69°W)

Project description from C-DEBI (https://www.darkenergybiosphere.org/award/microbial-carbon-cycling-beneath-the-west-antarctic-ice-sheet/)

Subglacial lakes were discovered beneath the Antarctic Ice Sheet in the 1970's and, given the presence of liquid water and saturated sediments, it has been debated whether or not these deep, cold biosphere habitats harbor active microbial communities. Subglacial Lake Whillans (SLW) was cleanly sampled in January 2013 with the goal of establishing the habitability and presence of life beneath the Antarctic Ice Sheet. The aim of this graduate fellowship project was to further characterize the SLW carbon cycle, in particular, chemolithoautotrophic microbial processes through geochemical and microbiological methods. Geochemical analyses showed that sulfide oxidizing bacteria were active and contribute to mineral weathering in the surficial sediments of SLW. Long water residence times beneath the West Antarctic Ice Sheet (WAIS) create a mineral weathering regime in SLW that is distinctly different from subglacial habitats of mountain glaciers. Concentration and stable isotope measurements of methane confirm a reservoir of methane formed by methanogenic archaea beneath the WAIS. The modeling results show that this biological methane provides a source of energy to an active methane oxidizing population at the sediment-water interface. The methane also is modeled to be an important source of carbon for biomass synthesis in the methane oxidizing population,

with rates of biomass incorporation similar to that of ammonia oxidizing archaea in the SLW water column. These results provide evidence that the sub ice sheet environment provides favorable conditions and substrates to support an active microbial ecosystem, thus expanding the extent of the biosphere to include the area beneath the WAIS and, possibly, the entire Antarctic Ice Sheet.

This project was funded by a C-DEBI graduate fellowship.

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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
NSF Office of Polar Programs (formerly NSF PLR) (NSF OPP)	OPP-1023233
NSF Office of Polar Programs (formerly NSF PLR) (NSF OPP)	OPP-1115245

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