

Supplemental Data from Frank et al. (2010): Cryogenic origin for brine in the subsurface of southern McMurdo Sound, Antarctica: *Geology*, v. 38, p. x- y.

Sampling and Analytical Methods

Thirty-five 5- to 10-cm-long whole-round core samples were collected from the AND-2A core for extraction and analysis of interstitial water. The sampling protocol called for obtaining 5 cm sections from the PQ core (0-229.12 mbsf) and 10 cm sections from the narrower gauge HQ core (229.12-1011.04 mbsf). These samples were taken at ~10 m intervals to 100 mbsf, and then every ~20 m to the bottom of the HQ hole. The shallowest sample was taken from 9.67 mbsf and the deepest from 963.44 mbsf, ensuring coverage of diagenetic processes throughout the PQ and HQ holes. Because of its relatively narrow diameter, the NQ core was not sampled for pore water analysis.

Whole-round sediment sections were cut immediately after each coring run and delivered to the Crary Lab within c. 14 hours of cutting. To prevent chemical alteration due to oxidation and temperature change before and during delivery from the drill site to Crary Lab, the cut samples were immediately wrapped in plastic shrinkwrap and refrigerated at ~4°C. Upon arrival at the Crary Lab, sediment samples were removed from the plastic wrap and the outside ~1 cm of each whole-round section was removed to minimise potential contamination. Prior to squeezing, lithified samples were placed into clean plastic bags and partially disaggregated using a rubber mallet. The samples were then placed into a titanium and stainless steel squeezing device and squeezed at ambient temperature (ca. 21°C) by applying pressure up to 40,000 lb (~4150 psi) with a Carver press (Manheim and Sayles, 1974). Interstitial water was extruded through a pre-washed Whatman no. 1 filter fitted above a titanium screen. All interstitial water samples were filtered through 0.45-micron disposable filters and collected into clean plastic syringes through a port in the base of the squeezing device. After collection of up to 30 ml of interstitial water, the syringe was removed from the squeezer, a fresh 0.45-micron filter was attached to the syringe, and the sample was dispensed into storage vials. Splits of the pore water samples were immediately analysed for pH, and, when more than ~8 ml of water was collected, alkalinity. Additional splits were stored for later analyses of major elements. Samples for stable isotopic analysis were placed into glass vials with poly-seal caps. Samples for major ion determinations were placed into hard plastic vials. All pore water samples were stored at ~4°C. Of the thirty-five whole rounds, 21 yielded pore water. Results are provided in Table DR-1.

The analysis of interstitial water samples followed the procedures outlined by Gieskes et al. (1991). Rough estimates for salinity were routinely determined with an optical hand-held refractometer. On samples with higher yields, conductivity, a proxy for salinity, was determined using an YSI 3100 Conductivity Instrument. Because the available conductivity meter and standards were designed mainly for use with freshwater samples, conductivity measurements became inaccurate once salinities exceeded ~38 PSU. On more saline samples, salinity was determined from Cl data using the relationship in Wooster et al. (1969). The pH was measured on the National Institute of Standards scale using a Beckman pH meter. Alkalinity was measured using a Brinkmann pH electrode and a Metrohm autotitrator. Note that although pH was also measured as part of the alkalinity titration, pH measurements obtained in this fashion are not always reliable because the algorithm employed for pH measurement before the start of

the alkalinity titration is strongly influenced by degassing. IAPSO (International Association for the Physical Sciences of the Ocean) served as the primary analytical standard for measurements of alkalinity, conductivity, salinity, and pH. For alkalinity measurements, a suite of sodium borate (Borax) based standards with alkalinities of 10 mM, 20 mM, and 40 mM were also prepared as outlined by Gieskes et al. (1991). Magnesium (Mg^{2+}), calcium (Ca^{2+}), potassium (K^+), sodium (Na^+), lithium (Li^+), chloride (Cl^-), bromine (Br^-), and sulfate (SO_4^{2-}) were measured to within 3%–5% on 1/200 to 1/1000 diluted aliquots in Milli-Q water using a Dionex DX-120 ion chromatograph. The same suite of analyses was also carried out on samples of formation fluid from *c.* 360 mbsf that was sampled at the drillsite and local seawater from the seawater tap at the Crary Laboratory. Although reported in Table DR-1, Ca^{2+} results are considered unreliable due to the presence of Ca^{2+} in the Crary Lab Milli-Q water that was used to prepare standards and dilute samples. The $\delta^{18}\text{O}$ values of pore water were measured using a GV Multiprep Autosampler attached to a GV mass spectrometer dual inlet in the Water Sciences Laboratory at the University of Nebraska-Lincoln. Oxygen-isotope ratios were corrected for ^{17}O contribution (Craig, 1957) and are reported relative to the V-SMOW standard. Precision is $\pm 0.1\text{‰}$ and was monitored through analysis of NIS and laboratory standards.

References

- Craig, H., 1957, Isotopic standards for carbon and oxygen and correction factors for mass spectrometric analysis of carbon dioxide: *Geochimica et Cosmochimica Acta*, v. 12p. 133–149.
- Gieskes J.M., Gamo T., and Brumsack H., 1991, Chemical methods for interstitial water analysis aboard JOIDES Resolution. Ocean Drilling Program, Technical Note 15, 60 pp.
- Hassan, A.A., 1982, Methodologies for extraction of dissolved inorganic carbon for stable carbon isotope studies: Evaluation and alternatives: US Geological Survey Water Resources Investigations, v. 82-86, p. 51.
- 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.
- Wooster, W.S., Lee, A.J., and Dietrich, G., 1969, Redefinition of salinity: *Limnological Oceanography*, v. 14, p. 437-438.

Table DR1: Pore water geochemical results for core AND-2A, Southern McMurdo Sound (SMS) Seawater, and Formation Water (Borehole Flow-in) from c. 360 mbsf.

Depth (mbsf)	Cl (meq/L)	Salinity (PSU)	Na (meq/L)	K (meq/L)	Mg (meq/L)	Br (meq/L)	SO ₄ (meq/L)	Ca (meq/L)	Alkalinity (mM)	δ ¹⁸ O (‰, VSMOW)
9.67	654	41.90	557	30.65	58.69	1.00	29.57	26.82	2.38	-1.3
30.09	576	36.91	490	13.98	56.51	1.01	27.92	37.67	17.39	-2.7
37.41	612	39.20	523	14.30	67.10	1.09	26.66	46.28	30.93	-5.6
43.72	659	42.20	563	17.22	76.76	1.11	25.29	23.43	44.14	-8.1
51.30	693	44.40	598	17.28	81.65	1.15	26.22	27.65	40.81	-9.8
57.21	692	44.30	598	17.09	81.11	1.15	25.87	48.02	42.00	-10.0
62.66	691	44.25	598	18.03	78.36	1.20	25.20	17.87	38.34	-10.6
73.15	821	52.57	722	21.11	99.28	1.42	30.37	34.52	41.64	-10.3
81.03	722	46.22	621	19.56	81.76	1.16	25.41	14.67	38.80	
92.97	740	47.37	640	17.69	88.08	1.18	26.02	65.89	44.51	-10.9
116.22	804	51.47	693	17.79	95.09	1.32	29.36	46.51	35.76	-5.2
155.76	1117	71.51	884	26.11	108.86	1.56	29.70	33.73	53.02	-8.5
235.66	1974	126.44	1498	46.86	188.81	3.01	26.95	53.35	51.00	-9.7
293.30	2100	134.50	1605	42.64	211.25	3.25	24.74	48.87	54.92	-10.6
336.18	2303	147.51	1723	47.49	212.72	3.27	27.04	125.23	46.04	-10.2
353.53	2253	144.26	1696	53.52	204.12	3.35	26.60	109.45	-	-
545.01	2771	177.48	2139	66.43	275.65	4.24	29.32	84.45	13.48	-9.3
619.35	2728	174.70	2047	75.96	238.69	4.17	25.82	145.19	-	-
779.69	2895	185.41	-	-	-	4.52	26.24	-	-	-
809.84	2722	174.32	-	-	-	4.11	24.01	-	-	-
963.44	3091	197.98	-	-	-	5.22	29.92	-	-	-
SMS seawater	559	35.81	483	10.86	51.37	0.86	29.81	16.87	-	-1.0
Borehole flow-in	2085	133.55	1556	52.98	186.06	2.87	28.56	71.51	-	-

Note: mbsf is meters below sea floor.