

## Strong margin influence on Arctic Ocean barium cycle revealed by Pan-Arctic Synthesis

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## Introduction

The information in this supplemental includes additional text, tables, and figures. Presented is an expansion on the methods presented in the manuscript, intercalibration of trace element data, and additional supportive figures toward the discussion in the manuscript. Details can be found in the sections below.

### **Text S1. Additional Sampling and Analytical Protocols for dBa**

Filtered seawater was collected into acid-washed 125 mL HDPE bottles. Acid washing procedures met GEOTRACES standards ([www.geotraces.org/cookbook](http://www.geotraces.org/cookbook)): bottles were filled with ~10% HCl (Reagent Grade) and soaked overnight at ~60°C (repeated 3 times). Bottles were then rinsed with DI water. Shipboard sampling was conducted by filtration through a 0.45 micron supor filter, each bottle was rinsed with seawater (3x) before collection of the sample.

#### **GN01**

At the University of Southern Mississippi (USM) Center for Trace Analysis, dissolved Ba was determined using an ICP-MS (ThermoFisher Element XR) in low resolution; samples were introduced with a PC3 spray chamber (Elemental Scientific). Prior to analysis, samples were acidified to 0.024 M HCl (Fisher Optima). In preparation for analysis, following isotope dilution methods (Klinkhammer and Chan, 1990), samples were diluted 30-fold with ultra-pure water and spiked with enriched  $^{135}\text{Ba}$  solution (Oak Ridge National Laboratory) to a target  $^{138}/^{135}\text{Ba}$  ratio between 0.5 and 1. Standards and GEOTRACES reference samples (GS & GD, distributed from the 2008 GEOTRACES Intercalibration Cruise) were analyzed in every run for reproducibility, which was within < 2% RSD (Table S1).

#### **GN02/3**

At Vrije Universiteit Brussel (VUB), a volume of 0.25 ml of sample was pipetted into an acid cleaned 15 mL polyethylene tube and acidified with 0.15 ml concentrated ultra-pure nitric acid to ensure the stability of Ba measurements. This acidified sub-sample was spiked with 0.15 ml of a  $^{135}\text{Ba}$ -spike solution yielding a  $^{138}/^{135}\text{Ba}$  ratio between 0.7 and 1 to minimize error propagation (Klinkenberg et al., 1996; Webster, 1960). Subsequently, the sample was diluted 30-fold with 7 ml Milli-Q grade water to reduce salt content to less than 0.2%. Quantities of sample, spike and dilution water were assessed gravimetrically. The same procedure was employed to prepare blanks (Milli-Q grade water) and reference waters: SLRS-5 & SLRS-3 (National Research Council Canada; Ba concentrations =  $14.0 \pm 0.5 \mu\text{g L}^{-1}$  and  $13.4 \pm 0.6 \mu\text{g L}^{-1}$ , respectively) and 'OMP' seawater (Mediterranean seawater prepared at Observatoire Midi Pyrénées, Toulouse, France; Ba concentration =  $10.4 \pm 0.2 \mu\text{g L}^{-1}$ ). Isotope ratios were measured by sector-field inductively-coupled plasma mass spectrometry (SF-ICP-MS; Element 2, Thermo Finnigan). Reproducibility of our method is within < 2 % (RSD) as tested on repeat preparation of SLRS-5 (Table S1).

## GN04

At the University of Alaska, Fairbanks, dissolved Ba was determined as done at the University of Southern Mississippi (see above GN01) with the following changes: The samples were diluted 100-fold with ultra-pure water (rather than 30-fold) and analyzed by ICP-MS (ThermoFisher Element 2). Furthermore, sample spikes target a  $^{138}/^{135}\text{Ba}$  ratio between 1 and 2. Standards and NRC NASS reference samples (NASS-6 and NASS-7, were analyzed in every run for reproducibility, which was within < 2% RSD (Table S1).

**Table S1.** Reproducibility and reference standards for dBa and  $\delta^{138}\text{Ba}$ .

Lab	Parameter	Standard	Referenced value	Measured value	RSD %	n	Detection limit
VUB	dBa	SLRS 5 (ppm)	14.0 +/- 0.5	14.02 +/- 0.21	1.53	15	4.7 nM
VUB	dBa	SLRS 3 (ppm)	13.4 +/- 0.6	13.25 +/- 0.33	2.49	15	7.2 nM
VUB	dBa	OMP (ppm)	10.4 +/- 0.2	10.43 +/- 0.24	2.30	7	5.3 nM
USM	dBa	GS (nmol/kg)		44.3 +/- 0.8	1.80	12	2.4 nmol/kg
USM	dBa	GD (nmol/kg)		54.1 +/- 0.9	1.70	12	2.7 nmol/kg
UAF	dBa	NASS-6 (nM)		48.1 +/- 0.8	1.70	18	1.6 nM
UAF	dBa	NASS-7 (nM)		33.1 +/- 0.7	1.43	14	2.1 nM
WHOI	dBa	GSP (nM)		35.4 +/- 0.8		5	
WHOI	dBa	GSC (nM)		41.8 +/- 0.9		8	
WHOI	dBa	D1 (nM)		101.1 +/- 2.0		13	
WHOI	$\delta^{138}\text{Ba}$	GSP (‰)		0.61 +/- 0.04		5	
WHOI	$\delta^{138}\text{Ba}$	GSC (‰)		0.54 +/- 0.04		8	
WHOI	$\delta^{138}\text{Ba}$	D1 (‰)		0.33 +/- 0.03		13	

## Text S2. Additional Analytical Protocols for dissolved $\delta^{138}\text{Ba}$ (GN01)

Sample solutions were aspirated at 140  $\mu\text{L}/\text{min}$  with  $\sim 1$   $\text{L}/\text{min}$  Ar through a PFA micro-concentric nebulizer (Elemental Scientific) and desolvated in an Aridus II (CETAC). The resultant aerosol was introduced into the MC-ICP-MS and admixed with 3–5  $\text{mL}/\text{min}$   $\text{N}_2$  to reduce  $\text{BaO}^+$  formation (Miyazaki et al., 2014). Analyses were performed in static mode by simultaneously monitoring baseline-corrected ion currents corresponding to  $m/z$  131 (Xe; L3), 135 (Ba; L1), 136 (Xe, Ba, Ce; center cup), 137 (Ba; H1), 138 (Ba, Ce, La; H2), 139 (La; H3), and 140 (Ce; H4) for 30 integrations, each  $\sim 4.2$  s in duration. (Detector baselines were measured by deflecting the ion beam and measuring intensities for 30 s prior to each analysis.) Data reduction was performed using the three-dimensional geometric interpretation of the double spike problem (Siebert et al., 2001) whereby 138/135, 137/135, and 136/135 correspond to the x-, y-, and z-axes, respectively. Sample isotopic composition was solved iteratively—with additional nested loops for isobaric corrections—and reported relative to the nearest four bracketing measurements of NIST

standard reference material 3104a in the delta-notation:

$$\delta^{138}\text{Ba}_{\text{NIST}} (\text{‰}) = \left( \frac{\frac{^{138}\text{Ba}_{\text{sample}}}{^{134}\text{Ba}_{\text{sample}}}}{\frac{^{138}\text{Ba}_{\text{NIST}}}{^{134}\text{Ba}_{\text{NIST}}}} - 1 \right) \times 1000 \quad (\text{Eqn. 1})$$

## **Text S2. Additional Sampling and Analytical Protocols for pBa**

### **GN01**

Large and small fraction (> 51 µm & 1 - 51 µm) particulate barium (pBa) samples were collected via McLane Research in situ pumps (WTS-LV) during the GN01 section (following Cutter et al., 2014). This paper reported total pBa (the sum of both large and small fractions). Original data are available at BCO-DMO (Lam, 2020). Pump casts were set up as described in Xiang & Lam (2020). Briefly, filter holders on the McLane pumps were prepared for two flow paths (quartz fiber “QMA” and polyethersulfone “Supor” flow paths) with 142 mm-diameter filter holders. Each path housed a “pre-filter” (51 µm polyester mesh; Sefar 07-51/33). Following the prefilter, the “QMA” path had paired 1.0 µm quartz fiber filters (Whatman QMA) that had been pre-combusted at 450°C for 4 hours. The “Supor” path had paired 0.8 µm polyethersulfone (Pall Supor800) filters. At basin stations (GN01), dBa was collected from the clean rosette which conducted two casts with a total of 23 depths (one overlapping depth). Particulate samples were typically collected from two pump casts for a total of 16 depths; at three stations, three casts were conducted for a total of 24 depths. In comparing the dBa to pBa, sample depths are often not a match.

Particulate barium concentrations were obtained via a refluxing digestion method (Cullen & Sherrell, 1999; Ohnemus et al., 2014; Planquette & Sherrell, 2012). Briefly, the filter was placed onto the wall of a 15 mL flat-bottom screw-cap Savillex vial to avoid immersion. The digestion includes a 4-h refluxing at 110 °C with an ultrapure (ARISTAR® or Optima™ grade) 50% HNO<sub>3</sub>/10% HF (v/v) mixture and drying down of the acid mixture. By ICP-MS (Thermo Scientific Element XR) at the UCSC Plasma Analytical Facility, final pBa sample solutions were analyzed in low resolution in low resolution. Indium (1 ppb) was used as an internal standard for ICP-MS analysis.

### **GN02/3**

Detailed description of sampling and analysis are presented in Li (2017). Briefly, samples of particulate trace elements were collected from GO-FLO Bottles mounted on a trace metal clean rosette system. At all five stations, samples were collected between between 10 m and near bottom depth. Upon recovery, ~10 L of seawater were collected into LDPE cubitainers and was then filtered through a 0.45 µm Supor filter (47 mm diameter). The filters were subsequently dried, folded in half, and stored in clean poly bags until further analysis. Spaces, containers, and apparatuses were cleaned according to GEOTRACES protocols (Cutter et al., 2014).

Digestion of the particle samples was conducted at the University of British Columbia in a HEPA-filtered fume hood within a class 100 cleanroom. Filters were digested following the Piranha method (Ohnemus et al., 2014). Filters were placed in 15 mL Teflon vials (Savillex) and digested using a mixture of concentrated  $\text{H}_2\text{SO}_4$  and concentrated  $\text{H}_2\text{O}_2$  (1.2 mL and 0.4 mL, respectively) at high heat, to digest organic matter and filter matrix. For total digestion, 0.4 mL of concentrated  $\text{H}_2\text{O}_2$  was added five times with a two hour reflux and slight drying between additions. Following refluxing, samples were dried, washed with 0.1 mL of 8N  $\text{HNO}_3$ , and dried again. The remaining materials were digested using a concentrated acid mixture of  $\text{HNO}_3$ : $\text{HCl}$ : $\text{HF}$  (i.e., 453  $\mu\text{L}$   $\text{H}_2\text{O}$ , 506  $\mu\text{L}$   $\text{HNO}_3$ , 687  $\mu\text{L}$   $\text{HCl}$  and 354  $\mu\text{L}$   $\text{HF}$ ) at 110°C for 4 h. After complete drying, 1 mL of concentrated  $\text{HNO}_3$  and 1 mL of concentrated  $\text{H}_2\text{O}_2$  were added to the vials and taken to dryness again. Following this step, if the digest was yellow, which was uncommon, remaining organic matter was suspected, and another 1 mL of concentrated  $\text{HNO}_3$  and 1 mL of concentrated  $\text{H}_2\text{O}_2$  were added, refluxed, and dried. To the ideal pellet, 0.1 mL of concentrated  $\text{HNO}_3$  was added and taken to dryness.

For analysis by ICP-MS (Element2, Thermo Scientific), the final digest was re-suspended in 1%  $\text{HNO}_3$  with 10 ppb Indium, as an internal standard. Instrumental blanks were monitored every 6 samples by measuring 1%  $\text{HNO}_3$  with Indium. Detection limits and blanks are reported in Li (2017).

#### **GN04**

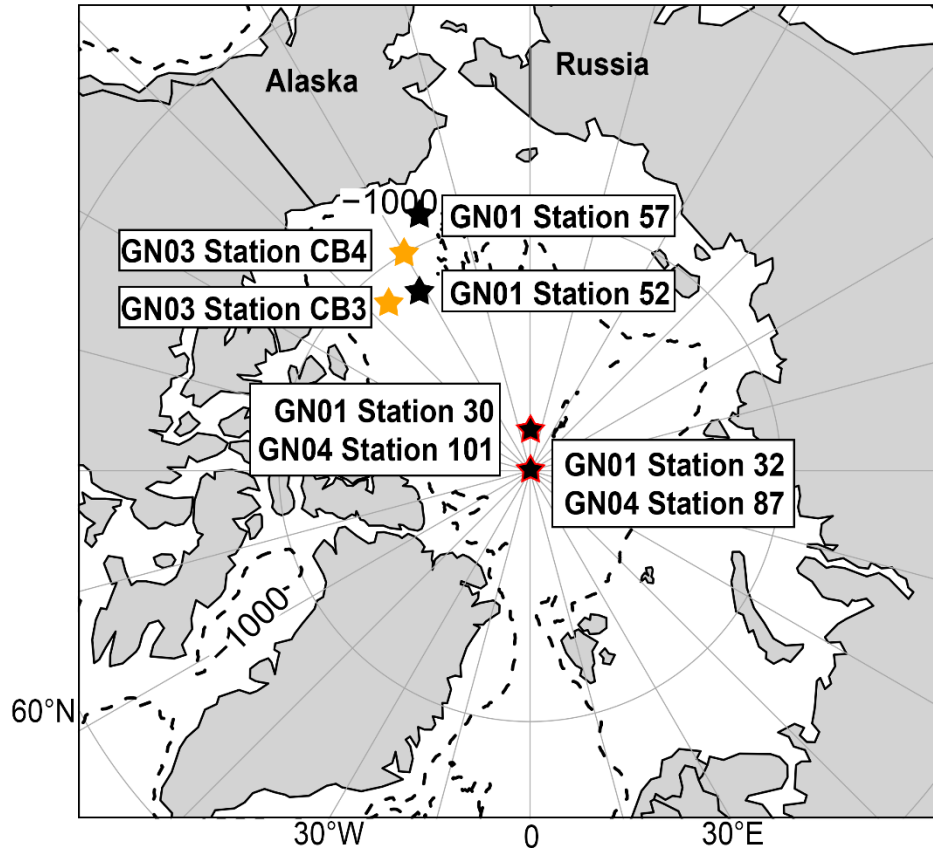
Suspended particles were sampled from the Dutch “ultraclean CTD” sampling system, Titan (de Baar et al., 2008), which consisted of 24 ultra-trace-metal clean polypropylene samplers of 24L each mounted on an all titanium frame with a SEABIRD 911 CTD system and deployed on a 11 mm Dyneema cable. After recovery, the complete “ultraclean CTD” was immediately placed in an ISO Class 6 clean room container, where samples for particulate trace elements were collected on 25mm diameter 0.45  $\mu\text{m}$  polyethersulfone filters (Pall Supor) mounted in swinnex filter holders under pressure of filtered  $\text{N}_2$  (0.7 bar) applied via the top-connector of the polypropylene sampler. Between 4 and 10L were filtered through the filters.

Particulate barium concentrations were obtained via a refluxing digestion method (Planquette & Sherrell, 2012). Briefly, the filter was placed onto the wall of a 15 mL flat-bottom screw-cap Savillex vial to avoid immersion. The digestion includes a 4-h refluxing at 110 °C with an ultrapure (Merck) 50%  $\text{HNO}_3$ /10%  $\text{HF}$  (v/v) mixture and drying down of the acid mixture. Residues were re-dissolved using a 3%  $\text{HNO}_3$  (v/v) solution then analyzed by SF-ICP-MS (Thermo Scientific Element XR) at the Pôle Spectrometrie Océans (France) in low resolution. Indium (1 ppb) was used as a drift monitor.

#### **Text S3. Intercalibration of GEOTRACES Crossover Stations**

The suite of cruises was conducted such that crossover stations, whereby two cruises

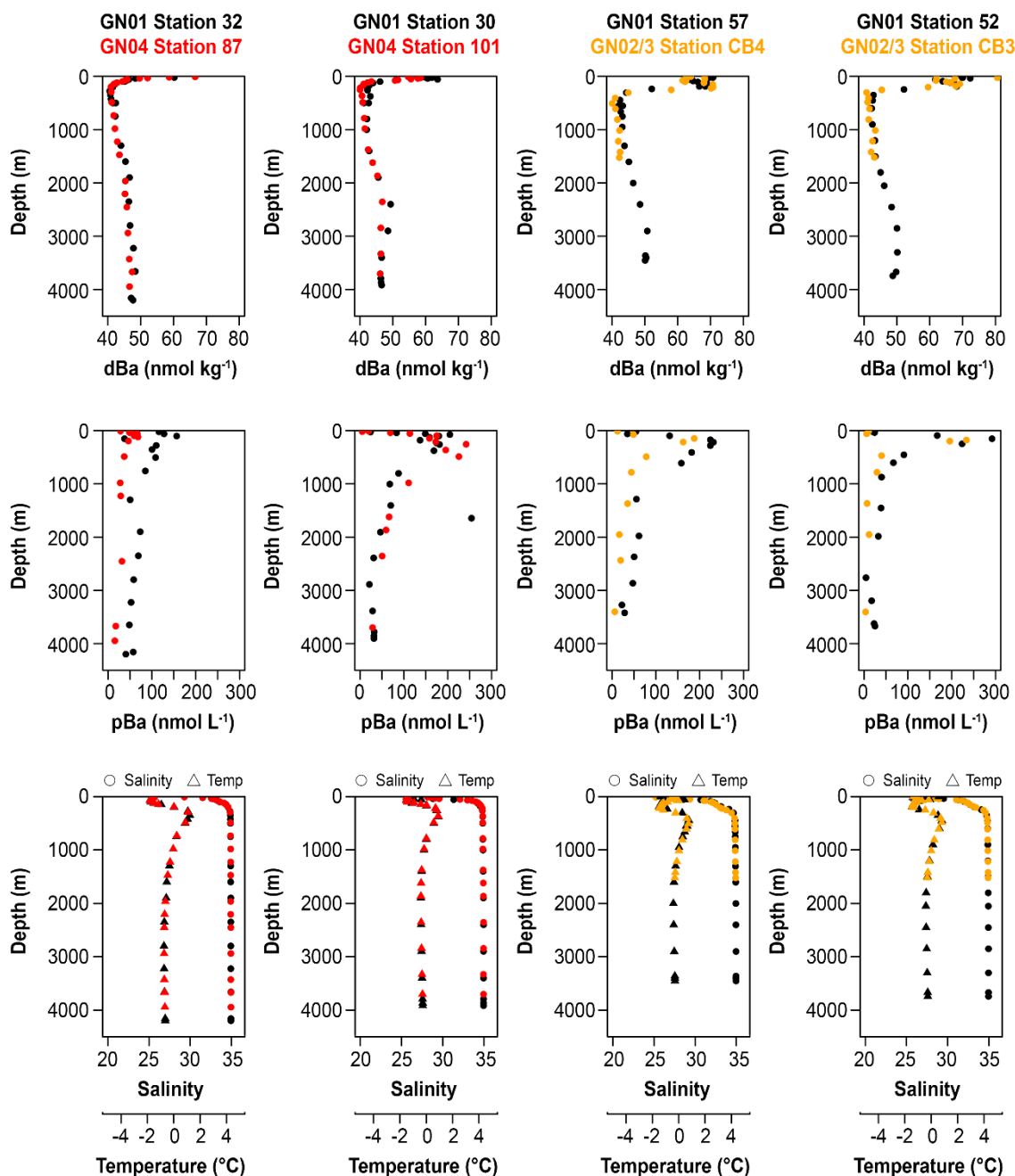
occupied the same station, could occur (Figure S1). We report the locations of each crossover station in Table S2. Generally, the stations compare well and there are acceptably low offsets (Figure S2a). For dissolved Ba, calibration offsets > 2.5 are only observed in the upper 500 m of the water column where there is the influence of a strong halocline. Thus, in the upper 500 m of the water column small differences in depth may result in large changes in dBa. Similarly to dBa, pBa may also be influenced by the halocline and pBa offsets > 50 pM are only observed in the upper 500 m of the water column.



**Figure S1.** Cross-over station conducted in three separate 2015 Arctic GEOTRACES cruises. Black colors indicate US stations, red colors indicate European stations, and orange indicates Canadian stations.

**Table S2.** Intercalibration exercise between cruises.

Cruise	Station	Latitude (°N)	Longitude (°E)	Approximate Distance between stations (km)	Dissolved Ba (nmol kg <sup>-1</sup> )			Particulate Ba (pmol L <sup>-1</sup> )		
					Median Offset	Min Offset	Max Offset	Median Offset	Min Offset	Max Offset
GN01	32	89.99	32.54	7	0.63	0.24	1.84	49.2	21.5	96.9
GN04	87	89.93	-120.19							
GN01	30	87.52	-179.81	3	2.01	0.20	8.10	19.2	0.6	188.1
GN04	101	87.50	179.80							
GN01	57	73.39	-156.53	266	1.94	0.12	18.07	33.8	13.8	82.6
GN02/3	CB4	75.00	-150.00							
GN01	52	77.50	-148.01	203	1.10	0.14	10.76	31.9	10.5	161.2
GN02/3	CB3	76.99	-140.05							
Summary					1.40	0.12	18.07	31.5	0.6	188.1



**Figure S2.** Cross-over station comparison. Following GEOTRACES Standards for intercalibration we compare for dBa (top row) and pBa (middle row) between stations conducted at roughly the same location on between cruises. The bottom row references temperature and salinity profiles for each station.

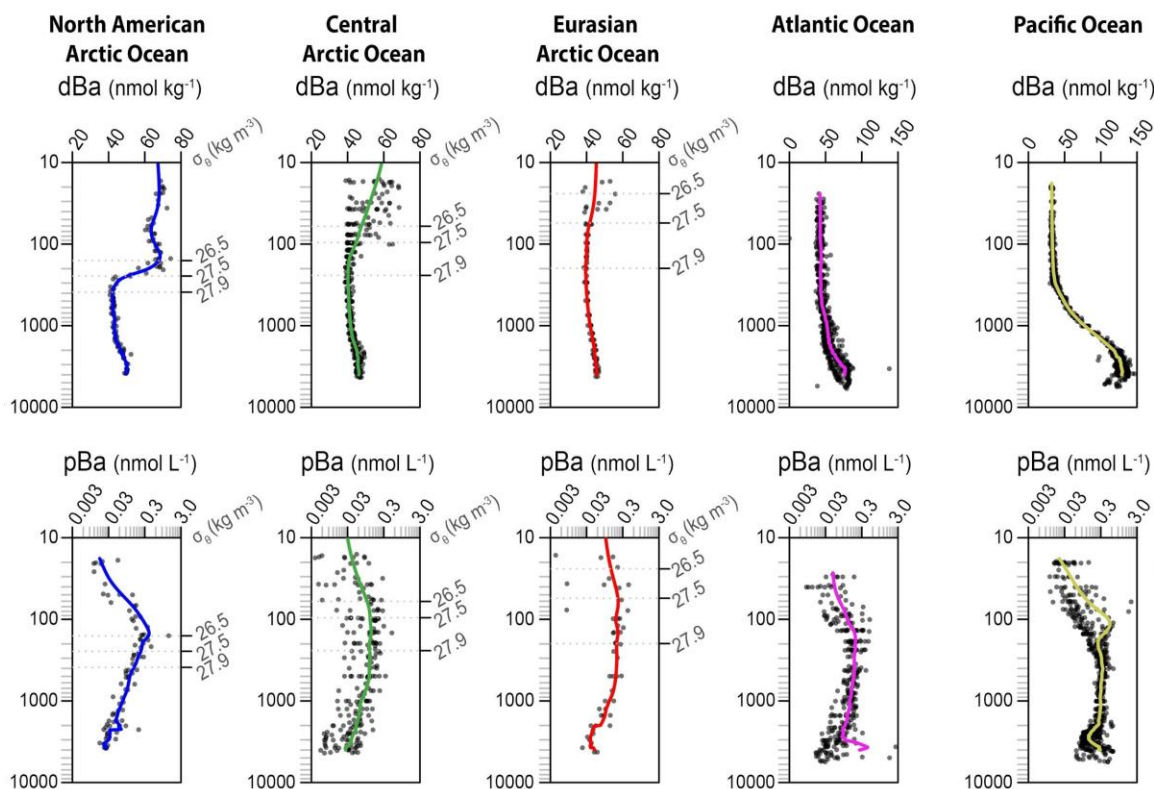
#### **Text S4. Comparison of dBa and pBa to the North Pacific and North Atlantic Oceans**

The distribution of dissolved and particulate Ba in the western Arctic Ocean is unique compared to vertical distributions in the North Pacific and North Atlantic Oceans



(Figure S3). Dissolved Ba distributions in the North Pacific and Atlantic follow a nutrient-like profile shape: low in the surface and generally increasing with depth. In the western Arctic Ocean basins, dBa is highest in the surface waters (< 300 m) and decreases between 300 and 2000 m depth before increasing toward the bottom.

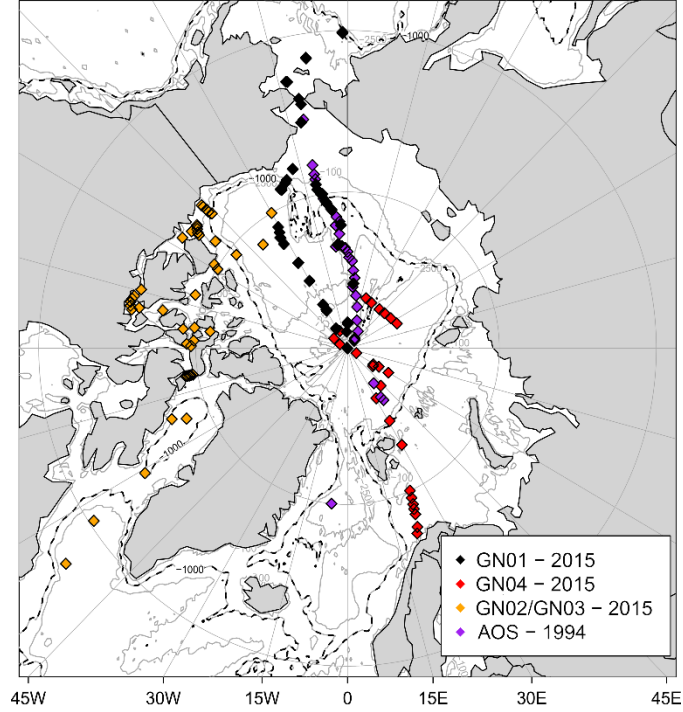
Particulate Ba usually has a mesopelagic maximum; in the western Arctic Ocean the maximum is slightly shallower than in other ocean basins.



**Figure S3.** Comparison of Arctic Ocean dBa and pBa vertical distributions to the Pacific Ocean (GP16) and Atlantic Ocean (GA03). Data for the Atlantic and Pacific Oceans was extracted from the GEOTRACES Intermediate Data Product (Version 2) (Schlitzer et al., 2018).

#### Text S5. The Flux Balance Approach to the dissolved Ba Budget

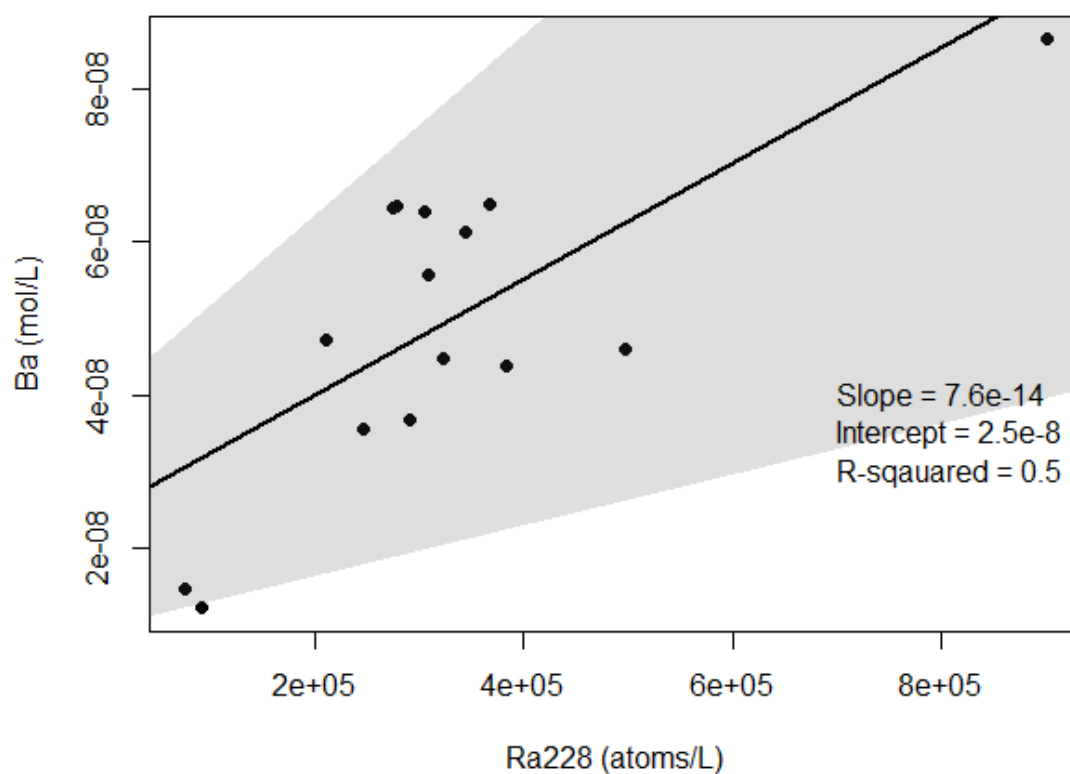
The box we consider in our elemental budget is the upper 500 m of the Arctic Ocean water column where bottom depths are greater than 1000 m (Figure S4). Two datasets are used separately to consider how the balance has changed since the early measurements of dBa in the Arctic Ocean: the 2015 Arctic GEOTRACES data and the 1994 Arctic Ocean Survey.



**Figure S4.** The spatial outer bounds of the box model are roughly identified in this figure. The 1000 m isobath is identified by the dashed line, the model is informed by all data points north of the Bering Strait, Fram Strait, and Canadian Arctic Archipelago (i.e., the Arctic Ocean Basins). Two scenarios were run using data from the 2015 GEOTRACES surveys and from the 1994 AOS survey.

#### **Text S6. Comparison of the box model results to Ra-flux predicted Ba fluxes**

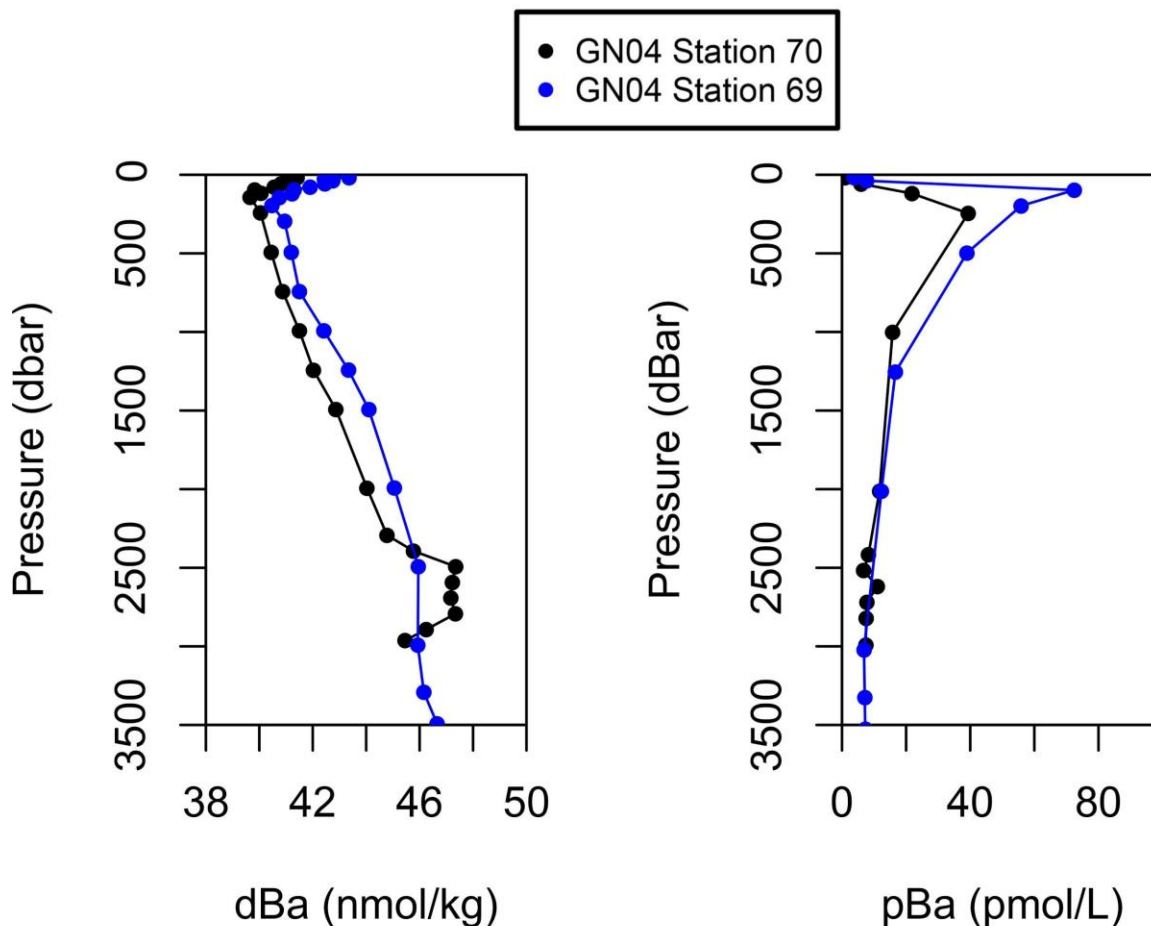
In the manuscript text we describe the results of our box model; which indicated that approximately 50% of the dBa budget is sourced from the shelves. Here, we use the dBa:Ra relationship on the shelf to predict the fluxes of dBa from shelf sediments such that:  $F_{Ba} = \frac{dBa}{dRa} \times F_{Ra}$ . Where F indicates flux (with the superscript representing the element) and dBa and dRa indicating the ratio of those elements on the shelves. The flux of radium ( $F_{Ra}$  in atoms/y) was directly from Kipp et al. (2018). We determined the dBa:dRa ratio using shelf dBa (nmol/L) data from this study and shelf  $^{228}\text{Ra}$  data from Kipp et al. (2018). The ratio used is the regression of the two parameters (Figure S5).



**Figure S5.** The observed relationship between dBa and dissolved  $^{228}\text{Ra}$ . Data points are from the western Arctic shelves (Bering and Chukchi Sea) sampled during the 2015 GN01 expedition. The black line is a type II linear regression and the gray shaded area is the 95% confidence interval.

#### **Text S7. Evidence of hydrothermal Ba in the Eurasian Arctic**

Two stations in the GN04 transect sit near the Nansen-Gakkel Ridge Crest. One station has dissolved distributions of dBa that reflect hydrothermal input (i.e., deep water maxima between 2000 and 3000 m; Figure S6).



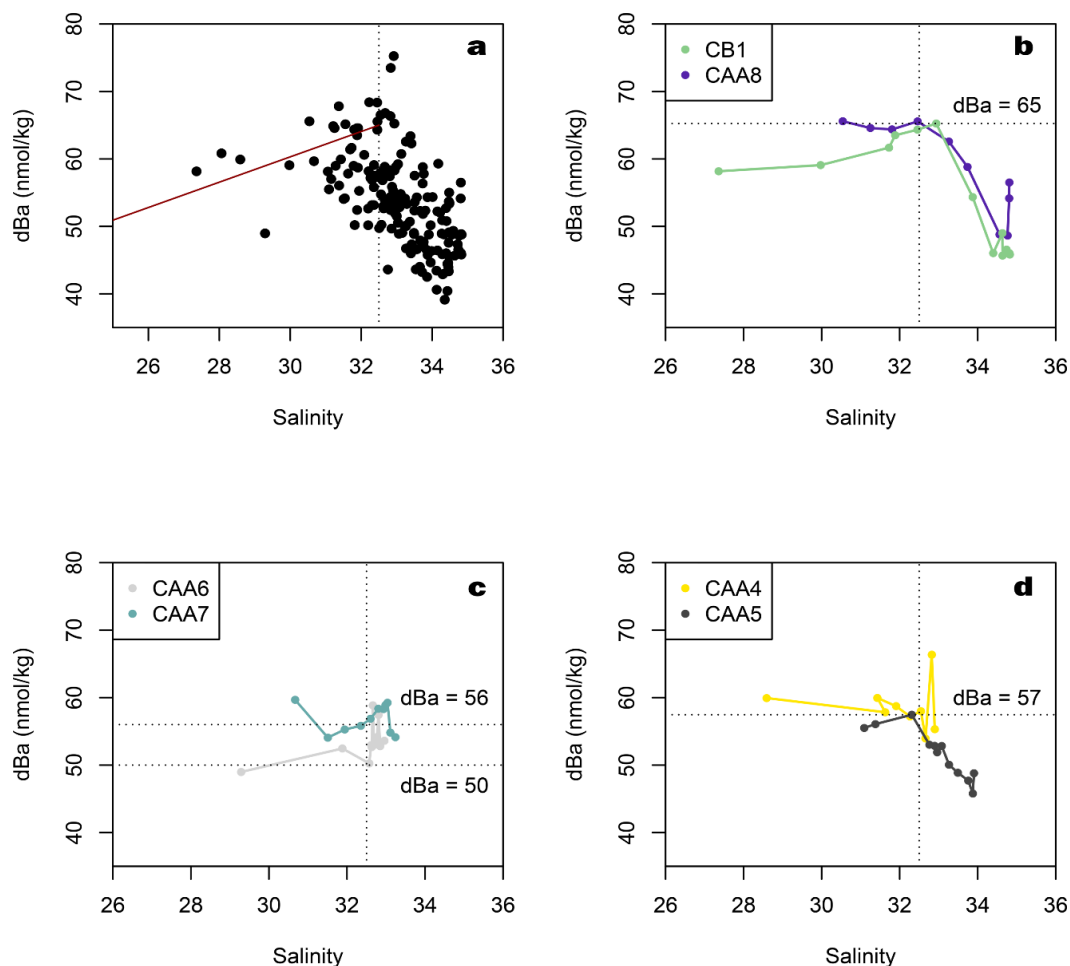
**Figure S6.** Nansen-Gakkel Ridge Crest Stations. The black dots and lines represent station 70, which appears to be influenced by a dBa source between 2000 and 3000 dbar (hydrothermal input); there also may be a slight input of pBa to the water column. Blue lines are the nearby station 69; which does not appear to be influenced by the hydrothermal plume.

#### **Text S8. Dissolved Ba Salinity Relationships in the Canadian Arctic Archipelago**

We investigated the dBa-Salinity relationships to probe how rivers or sea ice melt might influence dBa concentrations. Broadly through the Archipelago there are two salinity patterns. At high salinities ( $S > 32.5$ ), dBa decreases with increasing  $S$ . This is consistent with mixing of Atlantic-source water with Pacific-origin waters in the Arctic Ocean basins. However, we note that there is a large amount of scatter in the CAA trend, which is not observed in the Arctic Ocean. At low salinities ( $S < 32.5$ ) dBa decreases slightly, which is roughly in line with how a slight contribution of sea ice would dilute the seawater concentrations. We suspect there is not a large river influence as at low salinities ( $S < 32.5$ ), dBa decreases; generally, rivers have high dBa signatures and would drive dBa up at low salinity. We note that the concentrations of dBa in CAA rivers is not well constrained, but studies show a broad range of possible endmember (Colombo et al.,

2019). Most of the possible endmembers are higher than seawater, but a few do fall below the seawater concentration. Thus, it is possible there is slight river influence in addition to sea ice melt at the low salinity range.

Here, we diagnose the reason for the scatter at the high salinity range by looking at stations in the CAA (Parry Channel) moving from the Arctic Ocean eastward to Lancaster Sound. We follow the dBa at each station and highlight the dBa at  $S = 32.5$ , the salinity of Pacific-derived seawater. Moving eastward, dBa at  $S = 32.5$  decreases, furthermore, the dBa on the north side of the channel (CAA4 and CAA6) can be substantially lower than on the south side of the channel (CAA5 and CAA7). We note that CAA7 is tucked just south of the Parry Channel, in a northward flowing channel of the Archipelago. Due to its position, it may not be perfectly representative of waters flowing from the Arctic through Parry Channel. On the south side of the Parry Channel, dBa decreased to  $\sim 56$  nmol/kg at  $S = 32.5$  (from  $\sim 65$  nmol/kg in the Canada Basin and western extent of Parry channel). On the north side of the channel, dBa reached as low as 50 nmol/kg at  $S = 32.5$ . We suggest this erosion of the high dBa signal is due to mixing of Atlantic-like waters in Baffin Bay with the eastern extent of the Parry Channel.



**Figure S7.** dBa-Salinity patterns in the CAA. In all panels the dashed vertical line is  $S = 32.5$  and is representative of Pacific-derived seawater; the dashed horizontal line is the dBa at  $S = 32.5$ . a) all stations and samples in the Parry Channel. The red line denotes mixing with sea ice melt. b) Stations on the Arctic Ocean (western) side of the Parry Channel; CB1 is in the Canada Basin and CAA8 is in the Parry Channel. Both of these stations have a “western Arctic Ocean-like” signal, where Pacific-derived seawater is high in dBa ( $\sim 65$  nmol/kg). c) Stations CAA6 and CAA7 (just west of the Barrow Strait in Parry Channel). Dissolved Ba has decreased to 56 nmol/kg on the south side of the Channel and 50 nmol/kg on the north side of the channel. d) Stations CAA4 (north side of channel) and CAA5 (south side of channel) are located just east of the Barrow Strait. They have roughly equivalent dBa (57 nmol/kg).

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