Sea ice control on winter subsurface temperatures of the North Iceland Shelf during the Little Ice Age: A TEX_{86} calibration case study

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Key Points:

1) Our local Icelandic GDGT calibration produces more realistic temperature estimates with substantially lower uncertainty over broader spatial calibrations
2) Periods of thick sea ice during the Little Ice Age likely warmed the subsurface as a result of winter insulation

Keywords: North Iceland Shelf, marine sediment, Little Ice Age, lipid biomarkers, IP_{25}, GDGTs

Abstract

Holocene paleoceanographic reconstructions along the North Iceland Shelf (NIS) have employed a variety of sea-surface temperature (SST) and sea-ice proxies. However, these surface proxies tend to have a seasonal bias toward spring/summer, and thus, only provide a discrete snapshot of surface conditions during one season. Furthermore, SST proxies can be influenced by additional confounding variables resulting in markedly different Holocene temperature reconstructions. Here, we expand Iceland’s marine paleoclimate toolkit with TEX_{86}^{L}; a temperature proxy based on the distribution of archaeal glycerol dibiphytanyl glycerol tetraether (GDGT) lipids. We develop a local Icelandic calibration from 21 surface sediment samples covering a wide environmental gradient across Iceland’s insular shelves. Locally calibrated GDGT results demonstrate that: 1) TEX_{86}^{L} reflects winter subsurface (0-200 m) temperatures on the NIS, and 2) our calibration produces more realistic temperature estimates with substantially lower uncertainty (S.E. ±4 °C) over global calibrations. We then apply this new calibration to a high-resolution marine sediment core (last millennium) collected from the central NIS (B997-316 GGC, 658 m depth) with age control constrained by ^{14}C-dated mollusks. To test the veracity of the GDGT subsurface temperatures, we analyze quartz and calcite wt% and a series of highly branched isoprenoid alkenes, including the sea ice biomarker IP_{25}, from the same core. The sediment records demonstrate that the development of thick sea ice during the Little Ice Age warmed the subsurface due to winter insulation. Importantly, this observation reflects a seasonal component of the sea-ice/ocean feedback to be considered for the non-linear cooling of the Little Ice Age in and around Iceland.

1. Introduction

The steep oceanographic (temperature, salinity, and nutrient) gradients caused by the presence of Arctic and Atlantic Ocean currents surrounding Iceland have made the insular shelves targets for northern North Atlantic climate change studies since the last deglaciation (Knudsen et al., 2004; Ólafsdóttir et al., 2010). Throughout the Holocene, the strength and latitudinal position of these currents has varied on centennial timescales, impacting terrestrial climate (Larsen et al., 2012; Geirsdóttir et al., 2013, 2019; Harning et al., 2018), as well as the status of Icelandic ice caps (Larsen et al., 2011; Brynjólfsson et al., 2015; Harning et al., 2016a, 2016b; Anderson et al., 2018). As the North Atlantic is the region that exhibits the largest meridional heat flux of the Northern Hemisphere (Wunsch, 1980), and the area of deep-water formation that drives the Atlantic Meridional Overturning Circulation (AMOC), changes in local climate also have widespread hemispheric relevance (Denton & Broecker, 2008; Buckley & Marshall, 2016). Thus, gaining a more comprehensive understanding of the past oceanographic conditions in this region of the North Atlantic is not only key to understanding past episodes of climate change, but also critical to contextualize circulation changes under a currently warming climate (Spielhagen et al., 2011; Caesar et al., 2018; Thornalley et al., 2018).

Over recent decades, numerous marine sediment core studies have generated surface and bottom water temperature proxy records based on Mg/Ca and δ^{18}O of benthic and planktic foraminifera, calcite wt%, the alkenone unsaturation index ($U^{k\prime}_{37}$) and biotic species assemblages, such as dinoflagellates and diatoms (Andersen et al., 2004; Castañeda et al., 2004; Giraudeau et al., 2004; Smith et al., 2005; Solignac et al., 2006; Bendle & Rosell-Melé, 2007; Justwan et al., 2008; Ran et al., 2008; Ólafsdóttir et al., 2010; Jiang et al., 2015; Moossen et al., 2015; Kristjánsdóttir et al., 2016). Sea surface temperature (SST) proxies derived from phytoplankton result in a bias toward spring/summer SST and are influenced by additional confounding variables (i.e., salinity, nutrients, and depth habitat of biota, e.g., Prahl et al., 2006; Chival et al., 2014), resulting in markedly different Holocene temperature reconstructions around Iceland (Kristjánsdóttir et al., 2016). As an example, the Little Ice Age (LIA, 1250-1850 CE) is believed to be the coldest multi-centennial climate anomaly of the Holocene in Iceland, yet the coldest Holocene conditions inferred from alkenones (Kristjánsdóttir et al., 2016), dinocysts (Solignac et al., 2006) and diatoms (Andersen et al., 2004; Justwan et al., 2008) occur earlier, between 4 and 2 ka. Although the cooling observed in some proxies between 4 and 2 ka may be linked to long-term changes in the AMOC (Hall et al., 2004) and/or North Atlantic Oscillation (Orme et al., 2018), expanding Iceland's quantitative proxy toolkit may help reconcile proxy discrepancies.

In this study, we focus on quantifying the distribution of archaeal glycerol dibiphytanyl glycerol tetraethers (GDGTs) archived in marine sediment from the North Iceland Shelf (NIS). Although yet to be used to reconstruct marine paleoclimate on the NIS, GDGT distributions have been shown to reflect modern winter subsurface temperature (subT, 0-200 m) around Iceland (Rodrigo-Gámiz et al., 2015), the North Sea (Herfort et al., 2006), Skagerrak (Rueda et al., 2009), and Antarctica (Kim et al., 2010, 2012). Assuming temperature is the dominant control on the distribution of GDGTs on the NIS (Schouten et al., 2013 and references therein), but acknowledging that at least part of the variability could also be explained by confounding effects such as ammonia oxidation rates (Hurley et al., 2016), we improve absolute temperature estimates by developing a local Icelandic calibration based on the analysis of surface sediments. We then apply this local calibration to our late Holocene marine sediment core record. A suite of additional oceanographic surface climate proxies from the same core allow us to test the veracity of and to explore controls on GDGTs-based temperatures around Iceland.

2. Regional Setting

Today, the NIS represents the boundary where Arctic and Atlantic Ocean currents intercept (Fig. 1a-b, Stefánsson, 1962; Hopkins, 1991; Belkin et al., 2009). This front separates the cool, low salinity and sea-ice-bearing East Icelandic Current (EIC, 1 to 4 ^{o}C) to the north from the warmer and more saline Atlantic waters carried by the North Iceland Irminger Current (NIIC, 5 to 8 ^{o}C) on the inner and mid-shelf (Orvik et al., 2001). The current density differences between the two water masses result in vertical stratification along the NIS, such that the NIIC overlies the denser and cooler Upper Arctic Intermediate Waters (<0 ^{o}C, UAIW) (Fig. 1c). Within 70-100 km from Iceland's northern coastline, freshwater run-off and summer heating modify the NIIC surface waters and form "coastal surface waters" (Fig. 1c), which then disintegrate during the following winter (Stefánsson, 1962; Ólafsson et al., 2008). The onset of this stratification in early spring triggers the spring bloom of phytoplankton (Zhai et al., 2012).

Atlantic waters provide the primary source of nutrients (i.e., phosphate, nitrate, silica) to the Icelandic shelves. Due to the greater influence of nutrient-deficient polar waters, NIS nutrient concentrations are considerably lower compared to those along the south of Iceland, where Atlantic waters dominate (Stefánsson, 1968; Stefánsson & Ólafsson, 1991). Although the freshwater run-off from Iceland is key for the seasonal stratification and phytoplankton blooms along the NIS, it has negligible direct effects on nitrate and phosphate concentrations throughout the water column (Stefánsson & Ólafsson, 1991). In terms of modern oxygen saturation, the eastern NIS has similar values to those of waters south of Iceland, which may suggest relatively high rates of productivity for both locations (Stefánsson & Ólafsson, 1991). However, given that the NIS is rather limited in available nutrients, the relatively high oxygen saturation on the NIS may also relate to higher solubility of the colder Arctic waters.

Sea ice is also an integral component of the NIS. Iron oxide data on detrital grains suggest that drift ice is predominately sourced from east and southeast Greenland but also from as far as Canada and Russia, with the latter distal sources dramatically increasing in abundance over the last 1 ka (Andrews et al., 2009a; Darby et al., 2017). The presence of and correlation between quartz and the IP_{25} biomarker – proxies for sea ice extent - in core-top sediment along the NIS, and their absence from Iceland's southern and western shelves further supports the dominance of drift ice origins over local sea ice production (Axford et al., 2011; Cabedo-Sanz et al., 2016a). When present, sea ice limits the exchange of heat, gases and moisture between the ocean and atmosphere, in addition to insulating the colder polar atmosphere from the relatively warmer ocean during winter (Thorndike et al., 1975; Maykut, 1978, 1982). Due to Iceland's close proximity to the historical (post-1870 CE) sea ice edge (Divine & Dick, 2007), past changes in sea ice advection along the EIC have resulted in profound changes in local marine and terrestrial climate (Ogilvie & Jónsson, 2001; Moros et al., 2006; Massé et al., 2008; Miller et al., 2012; Cabedo-Sanz et al., 2016a).

3. Methods

3.1. Surface and marine core sediments

During July 1997, the cooperative USA/Icelandic *Bjarni Sæmundsson* B997 research cruise visited 30 locations across Iceland's western and northern shelves (Helgadóttir, 1997). At each location, marine surface sediments were collected using a grab sampler. Previous studies have used these surface samples to describe the regional distributions of foraminifera $\delta^{18}O$ (Smith et al., 2005), quartz wt % (Andrews & Eberl, 2007), and the sea ice biomarker IP_{25} (Axford et al., 2011; Cabedo-Sanz et al., 2016a). We selected a subset (n=11) of these marine surface sediment samples for GDGT analyses to help construct a local Icelandic calibration (Fig. 1). As many of the 30 surface sediment locations were spatially clustered, our selection provides a representative sample from each geographical location the cruise covered, and, thus optimizes our local calibration by spanning the full range of oceanographic conditions present around Iceland today. The B997 cruise also recovered a suite of piston and gravity sediment cores. In this study, we focus on giant gravity core B997-316 GGC (2.47 m long) from the central North Iceland Shelf (66.75°N, 18.79°W, 658 mbsl, Fig. 1) (Helgadóttir, 1997). Sediment (~1 cm^3) was subsampled every six cm for minerological and biomarker analyses. In order to minimize the degradation of biomarkers (e.g., Cabedo-Sanz et al., 2016b), samples were taken from cores stored at 4 °C. All samples were subsequently freeze-dried and kept in the freezer (-20 °C) prior to biomarker extraction.

3.2. Age control

Four radiocarbon-based age control points are derived from a combination of mollusks (*T. equalis*) and benthic foraminifera (*N. labradorica* and *G. auriculata arctica*) sampled from the B997-316 GGC core (Table 1). As the B997-316 GGC core lacked datable material in the uppermost sediment, two additional mollusks (*T. equalis*) were sampled from the near surface sediment of an adjacent short gravity core, B997-316 SGC (Table 1), to confirm that the tops of the sediment cores are modern and that no surface sediment was lost during coring. Samples were prepared for AMS radiocarbon dating at the Institute of Arctic and Alpine Research (INSTAAR) ^{14}C Preparation Lab and analyzed at the University of California Irvine.

3.3. Minerological analyses

Quantitative X-ray diffraction (qXRD) analysis was conducted on the <2 mm sediment fraction using the method developed by Eberl (2003) and used extensively on sediment samples on other B997- cores (Andrews & Eberl, 2007; Andrews et al., 2009a; Andrews, 2009). Comparison between qXRD weight percent estimates on known mineral mixtures and replicate analyses indicate that the errors on the weight percent estimates of both are in the range of ±1 %. For B997-316 GGC, we focus on the identification of quartz as a proxy to reconstruct the incursion of drift ice into Icelandic waters (i.e., sea ice and/or icebergs), and calcite as a proxy of ocean productivity (Andrews et al., 2001).

3.4. Biomarker analyses

At the University of Plymouth, freeze-dried subsamples (~1-2 g) from core B997-316 GGC were extracted for biomarkers by ultrasonication using dichloromethane:methanol (2:1, v/v). Samples were initially spiked with an internal standard (9-octylheptadec-8-ene, 9-OHD, 10 µL; 10 µg mL^{-1}) to permit quantification of highly branched isoprenoid (HBI) alkenes. Total lipid extracts (TLEs) were separated into three fractions (F1-F3) using silica column chromatography, after elution with hexane (6 mL), hexane:methylacetate (80:20, v/v, 6 mL), and methanol (4 mL), respectively. The F1 fraction contained aliphatic hydrocarbons including highly branched isoprenoids (HBIs; i.e., IP_{25}, $C_{25:2}$ and $C_{25:3}$), whereas F2 contained lipids with hydroxyl functional groups, including GDGTs. At the University of Colorado Boulder, freeze dried marine surface sediment samples (~3-7 g) were extracted three times on a Dionex accelerated solvent extractor (ASE 200) using dichloromethane:methanol (9:1, v/v) at 100 ^{o}C and 2000 psi, and kept as TLEs for the GDGT analysis.

The IP_{25} ($C_{25:1}$), diene II ($C_{25:2}$) and triene Z ($C_{25:3}$) biomarkers were analyzed at the University of Plymouth as described by Belt et al. (2012, 2015). Analysis of the F1 was performed via gas chromatography-mass spectrometry (GC-MS) following the methods and operating conditions of Belt et al. (2012) on an Agilent 7890A GC coupled to a 5975 series mass selective detector fitted with an Agilent HP-5ms column (30 m x 0.25 mm x 0.25 mm). Mass spectrometric analyses were carried out in selected ion monitoring mode. The identification of IP_{25} (Belt et al., 2007), diene II (Belt et al., 2007) and triene Z (Belt et al., 2000) was based on their characteristic GC retention indices (RI_{HP5MS} = 2081, 2082, and 2044 for IP_{25}, diene II, and triene Z, respectively) and mass spectra (Belt, 2018). Quantification of lipids was achieved by comparison of mass spectral responses of selected ions (IP_{25}, *m/z* 350; diene II, *m/z* 348; triene Z, *m/z* 346) with those of the internal standard (9-OHD, *m/z* 350) and normalized according to their respective response factors and sediment masses (Belt et al., 2012). Analytical reproducibility was monitored using a standard sediment with known abundances of biomarkers for every 14-16 sediment samples extracted (analytical error 4%, n = 31).

For GDGTs, we analyzed aliquots of the F2 from B997-316 GGC and aliquots of the TLE from marine surface sediment samples in the Organic Geochemistry Laboratory at the University of Colorado Boulder. Dry samples were dissolved in hexane:isopropanol (99:1, v/v), sonicated, vortexed, and then filtered using a 0.45 μm PTFE syringe filter. Prior to analysis samples were spiked with 10 ng of the C_{46} GDGT internal standard (Huguet et al., 2006). Isoprenoid GDGTs were identified and quantified via high performance liquid chromatography – mass spectrometry (HPLC-MS) following modified methods of Hopmans et al. (2016) on a Thermo Scientific Ultimate 3000 HPLC interphased to a Q Exactive Focus Orbitrap-Quadrupole MS. Rather than starting at 18% hexane:isopropanol (9:1, v/v) (Hopmans et al., 2016), we began our eluent gradient with 30% hexane:isopropanol (9:1, v/v) to shorten retention and overall run times without compromising the chromotographic separation of GDGTs. The HPLC was conditioned for 20 minutes between runs. Samples were analyzed on full scan mode with a mass range of 500-1500 *m/z* at 70,000 mass resolution. GDGTs were identified based on their characteristic masses and elution patterns. We adopt the TEX_{86}^{L} index to reflect relative changes in temperature, which is a modification of the original TEX_{86} index (Schouten et al., 2002) constructed for temperatures <15 ºC (Kim et al., 2010, 2012):

$$TEX_{86}^{L} = \log\left(\frac{[GDGT-2]}{[GDGT-1]+[GDGT-2]+[GDGT-3]}\right)$$

3.5. Local Icelandic TEX_{86}^{L} calibration

The largest uncertainty in the temperature relationship of GDGTs in global calibrations is at the low end of the temperature spectrum (<5 ºC, Kim et al., 2010, 2012), which may reflect a reduced sensitivity of Thaumarchaeota to temperature in cooler climates (Wuchter et al., 2004) or different regional oceanographic effects. Although a recent spatially-varying, Bayesian TEX_{86}-temperature calibration model was developed to capture regional oceanography variability (Tierney & Tingley, 2014), it excludes high-latitude settings and does not include any core top samples within a ~1000-km radius of Iceland. Hence, we targeted a network of local marine surface sediments (Fig. 1) to develop a local calibration that innately reflects the nuances of Icelandic oceanography and low local temperatures. We supplemented our 11 surface sediment samples with 10 previously published surface sediment samples from around Iceland (Table 2, Rodrigo-Gámiz et al., 2015) to generate a more comprehensive GDGT calibration that spans a larger geographical area and temperature gradient than obtainable using B997 samples alone. We note that although Rodrigo-Gámiz et al. (2015) sampled the surface of sediment cores, our samples were collected used a grab sampler, which may disturb the original sedimentary structure. However, natural factors such as sea floor mixing and variable sedimentation rates will always introduce uncertainty in the temperature embedded no matter how the sample is collected. Thus, we contend that the datasets can be merged for calibration purposes.

To calibrate the TEX_{86}^{L} index, *in situ* decadal mean temperatures from 1995-2004 CE were obtained from the World Ocean Atlas (WOA09, Locarnini et al., 2010) at the quarter-degree pixel where each surface sediment site is located. Subsequently, seasonal (spring, summer, fall, winter) and annual SST, in addition to 0-10 m, 0-20 m, 0-30 m, 0-40 m, 0-50 m, 0-60 m, 0-70 m, 0-80 m, 0-90 m, 0-100 m, 0-125 m, 0-150 m, 0-175 m, 0-200 m depth subsurface temperature integrations, were each regressed against the 21 core top TEX_{86}^{L} index values (Rodgrio-Gámiz et al., 2015; this study) to assess which portion of the water column and which season the GDGT distributions most closely correspond to around Iceland. We calculated *p*-values for each regression to determine their significance.

4. Results and Interpretations

4.1. Age model

An age model for the B997-316 GGC sediment core was generated in the CLAM software using the Marine13 calibration curve (ΔR=0, Reimer et al., 2013) and a smooth spline regression over 1000 iterations (Blaauw, 2010). The calibrated benthic foraminifera date from 49.5 cm depth produced an age reversal in the initial model, and thus, was identified as an outlier and removed from the final age model (Fig. 2). The ~400-year difference between the calibrated age of the foraminifera and that estimated from the model may relate to changes in ΔR resulting from variable water masses (Eiríksson et al., 2004; Wanamaker et al., 2012). The two mollusks from the adjacent short gravity core (B997-316 SGC) both returned conventional ^{14}C ages ≤ 400 years (Table 1), confirming modern sediment at the core top of the SGC.

Based on several lines of reasoning, we argue that the modern ages of the SGC can be used to validate the extrapolation of the B997-316 GGC age model to the surface (Fig. 2). First, given that both cores were collected from the same location, we can exclude any impacts from geographic-dependent factors, such as variable sedimentation rates or oceanographic currents, that may cause the age-depth relationships to differ between the two cores. Second, coring-dependent factors can also be excluded as the two cores were collected in succession of each other using the same equipment (Helgadóttir, 1997). Even though coring operations can result in the loss of saturated or poorly consolidated surface sediment, the identical coring techniques employed suggests that if the SGC captures modern surface sediment, so should the GGC.

Given that our age model only uses the three lowermost mollusks from the GGC and the uppermost mollusk samples from the adjacent SGC, increased age uncertainty undoubtedly exists where no datable material could be obtained (i.e., ~1400-2000 CE). However, we argue that our age estimates throughout the entire GGC core are reasonably strong. First, NIS sedimentation rate slopes only change significantly between deglacial/nonglacial periods (Andrews et al., 2002; Xiao et al., 2017) due to the presence/absence of the Icelandic Ice Sheet. Following the rapid demise of the Icelandic Ice Sheet ~15 thousand years ago (Norðdahl and Ingólfsson, 2015; Patton et al., 2017), sedimentation rates have remained linear across the NIS (Castañeda et al., 2004), consistent with the linearity of B997-316 GGC's sedimentation rate over the last millennium (Fig. 2). Second, when our age model is applied to the proxy datasets (Fig. 3), the interpreted period of the Little Ice Age (LIA, see following section) is consistent with previous age ranges in Iceland that are derived from high-resolution and precisely-dated terrestrial archives (Geirsdóttir et al., 2009; Larsen et al., 2011, 2012).

4.2. Sediment core B997-316 GGC

4.2.1. Minerological analyses

In Icelandic waters, the two minerals quartz and calcite are qualitative indicators that reflect the incursion of drift ice (i.e., sea ice and/or icebergs) and marine surface productivity, respectively (Andrews et al., 2001, 2009a). In years when cold low-salinity Arctic water dominates, sea ice (% quartz) increases and surface productivity (% calcite) decreases due to the development of a well stratified water column. The opposite is seen in the proxies during years characterized by warm and saline Atlantic waters, which reduces sea ice presence and mixes the water column resulting in higher productivity. Not surprisingly, % quartz and calcite generally show an inverse relationship over the last millennium in B997-316 GGC, which can be interpreted as the relative dominance of Arctic versus Atlantic waters at this location (Fig. 3a-b).

Percent quartz ranges from 1.4 to 2.7 %, whereas calcite ranges from 5.4 to 8.3 % (Fig. 3a-b). Recent analyses using mineral mixtures with known quartz wt % of 3.5 and 1.5 (Andrews et al., 2018) confirm that these small amounts of quartz can be correctly measured. Prior to ~1250 CE, quartz is relatively low, and calcite is the highest of the record, suggesting a dominance of warmer Atlantic waters at this time. Subsequently, quartz begins a gradual yet quasi-episodic rise towards its peak abundance at ~1900 CE. On the other hand, calcite appears to decline more sharply to lower values after ~1250 CE and remain relatively low through ~1900 CE, when it rises to levels near its pre-1250 CE state. Based on these two minerals, the period between 1250 and 1900 CE was likely characterized by cooler Arctic waters that favored the advection of drift ice, vertical stratification and lower surface productivity on the NIS. Following 1900 CE, the conditions reverted back to a dominance of warmer Atlantic waters that favored restricted sea drift transport and higher surface productivity (Fig. 3).

4.2.2. Highly-branched isoprenoid (HBI) alkenes

The analysis of the biomarker IP_{25} (Belt et al., 2007), a monounsaturated C_{25} HBI biosynthesized by Arctic sea ice diatoms (Belt et al., 2008; Brown et al., 2014), has gained recent traction as a novel proxy for spring/summer sea ice conditions around Iceland (Massé et al., 2008; Andrews et al., 2009b; Sicre et al., 2013; Cabedo-Sanz et al., 2016a;

Xiao et al., 2017). Although the IP_{25} biomarker is well-preserved in Arctic and sub-Arctic marine sediment and routinely applied in paleo sea ice reconstructions as old as 5.3 Ma (Stein et al., 2016), questions remain regarding its vertical transport, degradation processes, and environmental controls (see reviews by Belt & Müller, 2013; Belt, 2018). Notably, the interpretation of its presence (or lack thereof) can be ambiguous. IP_{25} below the limit of detection has often been interpreted as reflecting either a lack of seasonal sea ice cover, or permanent and thick sea ice that inhibits light penetration needed for sea ice diatoms to photosynthesize and grow. However, this is likely an over-simplification of a broader range of scenarios that result in absent IP_{25} (Belt, 2018). In any case, further information may be obtained by the complementary analysis of certain open-water phytoplankton biomarkers (i.e., brassicasterol or dinosterol, Müller et al., 2011).

Based on a distinctively heavy stable carbon isotopic composition, in addition to similar concentration profiles to IP_{25} across Arctic marine surface sediment, the di-unsaturated HBI diene II also has an Arctic sea ice diatom source (Belt et al., 2008; Cabedo-Sanz et al., 2013; Brown et al., 2014), and is made by some Antarctic sea ice algae as well (Belt et al., 2016). In contrast, a tri-unsaturated HBI (hereafter, triene Z) is biosynthesized by certain open-water diatoms (Belt et al., 2000, 2008, 2015; Rowland et al., 2001), and sources for the Arctic and Antarctic have recently been identified (Belt et al., 2017). Importantly, the presence (or lack thereof) of triene Z, like certain phytoplankton sterols, may help differentiate between open-water or thick sea ice conditions inferred from IP_{25} and diene II in the Arctic (Cabedo-Sanz et al., 2013; Smik et al., 2016; Köseoğlu et al., 2018). However, since sterols may also be derived from other (e.g., terrestrial) sources in addition to sea ice algae (Huang & Meinschein, 1976; Volkman, 1986; Volkman et al., 1998; Belt et al., 2013, 2018), we limit our analysis here to triene Z only.

HBIs were detected in all downcore samples, with the exception of 96.5 cm depth (1509 CE), where no triene Z, diene II or IP_{25} were detected (Fig. 3c-e). Concentrations ranged from near detection up to 1.6 ng/g sediment for triene Z, up to 19 ng/g sediment for diene II, and up to 4 ng/g sediment for IP_{25}. Triene Z exhibited the highest concentrations prior to 1200 CE, while its abundance diminished to very low or undetectable between ~1200 and 1800 CE (Fig. 3c). Triene Z then rises to higher concentrations up through 2000 CE. The similar relative trends of diene II and IP_{25} concentrations suggest that both HBIs are likely sourced from sea ice algae around Iceland, similar to other Arctic (Brown et al., 2014) and Antarctic locations (Collins et al., 2013). Periods of synchronous reductions of diene II and IP_{25} concentrations occur at ~1170-1290 CE, 1450-1650 CE, and 1880 CE-present.

The similar overall trends between % calcite and triene Z abundance suggest that both proxies indicate temperate water surface productivity (Fig. 3b-c). Hence, in years where warmer Atlantic waters dominate, both % calcite and triene Z abundance increase, while the opposite trend dominates during years characterized by cooler Arctic waters. The detection of both IP_{25} and % quartz throughout the record suggests that sea ice has been a persistent feature at this location of the NIS over the last millennium, even during intervals when elevated % calcite and triene Z suggest an increased influence of warmer Atlantic waters. We interpret the reduction of diene II and IP_{25} at ~1170-1290 CE and 1880 CE-present to reflect diminished seasonal sea ice because of higher concentrations of triene Z at the same time. In contrast, the reduction of diene II and IP_{25} from 1450-1650 CE likely reflects a period of thick, perennial sea ice as triene Z was mostly undetectable (Fig. 3c-e).

4.2.3. Glycerol dibiphytanyl glycerol tetraethers (GDGTs)

Changes in the degree of cyclization (number of cyclopentane moieties) in GDGTs have classically been interpreted to represent a physiological response of marine ammonia oxidizing Thaumarchaeota to changes in *in situ* temperature (e.g., Schouten et al., 2002). Thus, the TEX_{86} paleothermometer index has been empirically linked to annual or winter subT (0-200 m depth) in global data sets (Schouten et al., 2002; Kim et al., 2010, 2012). This presumption is supported by a recent study along a latitudinal transect in the western Atlantic Ocean, which demonstrated that the most likely water depths where GDGTs are produced from and exported to marine sediment is around 80-250 m (Hurley et al., 2018), similar to evidence for archaea abundance maxima at 200 m depths in the Pacific Ocean (Karner et al., 2001). Considering that Thaumarchaeota are chemolithoautotrophs that perform ammonia oxidation (conversion of NH_4^+ to NO_2^-), they are typically more abundant around the primary NO_2^- maximum near the base of the photic zone (Francis et al., 2005; Church et al., 2010; Hurley et al., 2018), and are thus most productive when there is minimized phytoplanktic competition over NH_4^+ (Schouten et al., 2013). In the case of the Arctic region, the latter occurs during the less productive winter months when photosynthesis for sea surface species is inhibited, which may explain the seasonal winter temperature bias of GDGTs previously observed in this region (Rodrigo-Gámiz et al., 2015). However, in addition to subT, recent studies have shown that several other environmental and geochemical factors can influence the degree of cyclization, such as growth phase (Elling et al., 2014), ammonia oxidation rates (Hurley et al., 2016), and oxygen concentrations (Qin et al., 2015). In contrast to some other marine temperature proxies, such as the $\delta^{18}O$ of planktic foraminifera, GDGTs do not seem to be influenced by variations in salinity (Wuchter et al., 2004, 2005; Elling et al., 2015). Finally, GDGTs appear to be relatively resistant to oxic degradation (Schouten et al., 2004), and thus, likely reflect original living conditions once deposited in the sedimentary record.

GDGTs were present above the detection limits in all marine sediment core samples, and substantially increase in concentration at the core top (Supplement Fig S1). The calculated TEX_{86}^{L} index ranged from -0.71 to -0.63 (Fig. 3f). The record displays high variability and a rather constant first order trend towards the present, in addition to the occurrence of two intervals of substantial decreases in TEX_{86}^{L} values during 1350-1530 CE and 1745-1975 CE. Both periods are preceded by periods of relatively higher TEX_{86}^{L} values during 1110-1350 CE and 1530-1745 CE, respectively. A full paleoceanographic interpretation of the TEX_{86}^{L} results is provided in the discussion.

4.3. Local Icelandic TEX_{86}^{L} calibration

GDGTs were also detected and above detection limits in all B997 marine surface sediments samples (n=11, Supplemental Fig S2). TEX_{86}^{L} values of these samples ranged from -0.72 to -0.61 (Table 2). The 10 marine surface sediment samples from Rodrigo-Gámiz et al. (2015) span a greater geographical and environmental range around Iceland, and hence exhibit a greater range of TEX_{86}^{L} values (-0.71 to -0.49, Table 2). When we use the combined set of Icelandic marine surface sediment samples (n=21), the regression analysis demonstrates that the integration of winter temperatures from the top 200 m of the water column provides the best regression coefficients (R^2=0.73, p<0.001) compared to the integration of other seasonal temperatures and the mean annual value (Figs. 4). Thus, sedimentary values around Iceland most likely represent winter subT that integrate a signal of the uppermost 200 m of the water column, consistent with the findings of Rodrigo-Gámiz et al. (2015).

5. Discussion

5.1. Local Icelandic TEX_{86}^{L} vs. regional Arctic calibration

If we supplement the combined Icelandic data set with more marine surface sediment samples from the greater northern North Atlantic region (Kim et al., 2010), the correlation coefficients of our winter subT (0-200 m) regression is substantially reduced (R^2 = 0.43 vs. 0.73, Supplemental Fig S3). This suggests that a local Icelandic calibration is optimal over larger regional calibrations, and perhaps, more accurately captures the nuances of local Icelandic oceanography. We hypothesize that the poorer performance of a more regional GDGT calibration for the North Atlantic region may relate to the inclusion of: 1) surface sediment samples from distal locations that feature different oceanographic environments than Iceland (e.g., Hudson Bay), and/or 2) samples from higher latitude (e.g., Svalbard and the Barents Sea) that are less "responsive" in terms of GDGT cyclization as they fall under the colder end of the spectrum in the global TEX_{86}^{L} calibration, which is characterized by a higher uncertainty and deviation from linearity (Kim et al., 2010). The standard error in our Icelandic winter subT calibration for 0-200 m (±0.4 °C), is also an order of magnitude lower than the error derived from global low temperature calibrations (e.g., 4.0 °C, Kim et al., 2010; 2.8 °C, Kim et al., 2012). The reduced uncertainty achieved in our Icelandic calibration highlights the growing need for the continued development and application of regional calibrations in future biomarker-based paleoclimate reconstructions (e.g., Kaiser et al., 2015; Foster et al., 2016; Russell et al., 2018). This is particularly important in areas where the temperature relationship of GDGTs deviates from the overall linear correlation observed in global calibrations (i.e., cold and warm regions).

Despite the reduced uncertainty compared to global calibrations, the regression coefficient for the Icelandic winter subT calibration (R^2=0.73) is comparatively lower than the global calibration (R^2=0.86-0.87; Kim et al., 2010, 2012). We hypothesize that the unconstrained confounding influence of ammonia-oxidation on the degree of GDGT cyclization (e.g., Hurley et al., 2016) may contribute to the scatter of our dataset (Fig. 4b). Although specific ammonia (NH_4^+) and nitrite (NO_2^-) information for this region is currently unavailable, reduced (enhanced) ammonia oxidation rates in the water column throughout the year would result in increased (decreased) degree of cyclization, thus yielding higher and lower temperatures, respectively (Hurley et al., 2016). If ammonia oxidation rates are driven by changes in ammonia supply and utilization (e.g., reduced nutrient availability in Arctic waters, or competition with phytoplankton), we cannot separate the influence of nutrient variability on the Icelandic TEX_{86}^L values with our current dataset. While oxygen availability has also been shown to influence the degree of cyclization in GDGTs (Qin et al., 2015), this factor is unlikely to affect the distribution of GDGTs around Iceland as these waters are relatively well-mixed and ventilated today (Stefánsson & Ólafsson, 1991), and presumably have been since the early Holocene (Kristjánsdóttir et al., 2016). With all known controlling factors considered, we suggest that our local TEX_{86}^L calibration improves the temperature estimates for Icelandic winter subsurface waters. Future work in constraining the effects of ammonia-oxidation around Iceland would undoubtedly benefit the application of TEX_{86} on the NIS.

By applying temperature calibrations to our down core TEX_{86}^{L} record, our data reveal rapid and abrupt temperature variability on the NIS during the last millennium (Fig. 5). If the existing annual SST (Kim et al., 2010) and annual subT TEX_{86}^{L} calibrations developed for polar regions (Kim et al., 2012) are applied, the GDGT distributions suggest that subT fluctuated up to 5 °C over the course of decades. These observations are considerably higher than expected, especially given that they are comparable to the magnitude of SST changes observed in other NIS proxy records over the entire Holocene (e.g., Andersen et al., 2004; Bendle & Rosell-Melé, 2007; Jiang et al., 2015; Kristjánsdóttir et al., 2016). As originally hypothesized, this exercise demonstrates that global calibrations that feature greater uncertainty for low temperatures and that do not include sites proximal to Iceland are not appropriate for the NIS. In contrast, by applying our local winter subT calibration, the magnitude of estimated subT is not only reduced to ranges more comparable to other proxy records for the last millennium but, importantly, also captures the modern instrumental winter subT (within calibration uncertainty) at the B997-316 GGC site (4 °C, Fig. 5), further reinforcing the application of our local Icelandic TEX_{86}^{L} calibration.

5.2. NIS surface and subsurface climate variability during the Little Ice Age

The NIS represents one of the few global examples where paleo-IP_{25} abundance in marine cores has been calibrated against observational and documentary records (Massé et al., 2008; Andrews et al., 2009b). As a result, the variability of IP_{25} has been routinely applied to marine sediment around Iceland as a robust indicator for seasonal sea ice (Massé et al., 2008; Andrews et al., 2009b; Sicre et al., 2013; Cabedo-Sanz et al., 2016a). Similar to these previous studies, IP_{25} concentrations in B997-316 GGC increase abruptly during the 13th century, and with the exception of the period 1450-1650 CE, remain elevated until the 19th century when concentrations begin to diminish (Fig. 6a). By employing statistical analyses on IP_{25} abundances and 11 other marine climate proxy datasets from marine sediment core MD99-2263, Andrews et al. (2009b) showed that a major regime shift in the marine climate off NW Iceland commenced after 1200 CE, possibly linked to a strengthening high-pressure ridge over Greenland in winter/spring that favored stronger north/northwesterly winds and increased drift ice export to Iceland. Our mineral and HBI records consistently reflect major shifts in surface conditions at a similar time and in the same direction (Fig. 3a-e), reinforcing the observed regime shift in marine climate, and increase of sea ice in particular, during the 13th century (Bergthórsson, 1969; Ogilvie & Jónsson, 2001; Massé et al., 2008; Andrews et al., 2009b; Sicre et al., 2013; Cabedo-Sanz et al., 2016a). The consistency of our surface productivity and sea ice proxy records in reflecting the established understanding of marine climate over the last millennium on the NIS supports the fidelity of the B997-316 GGC marine sediment record, and therefore, the interpretation of the GDGT record.

When the GDGT record is converted to winter subT, two pronounced centennial-scale cold anomalies exhibit mean winter subT below the record average of 4.34 °C; at 1350-1530 CE (3.99 °C) and at 1745-1975 CE (4.19 °C). These two cold anomalies are consistent with low surface productivity (% calcite and triene Z) and increased seasonal sea ice (% quartz, diene II, IP_{25}), which suggest greater dominance of colder Arctic surface waters between ~1250 and 1900 CE (Fig. 6a-b). In addition, alkenone-derived SST from marine core MD99-2275 50 km to the east (Fig. 1a) document steady cooling throughout this interval (Fig. 6c, Sicre et al., 2011), further supporting the presence of cool, Arctic surface waters in the vicinity of B977-316 at this time. However, the timing for the onset (1350 CE) and termination (1975 CE) of LIA cooling observed in the subsurface during winter appears to lag that of the surface (1250 and 1900 CE, respectively) (Fig. 6). A variety of model and data-based studies have demonstrated that the LIA was triggered by a combination of sustained stratospheric volcanic sulfate injection (Zhong et al., 2010; Miller et al., 2012; Sicre et al., 2013; Slawinska & Robock, 2018), low total solar irradiance (Shindell et al., 2001) and changes in the North Atlantic Oscillation, one of the major modes of internal climate variability in the North Atlantic (Trouet et al., 2009). On the NIS, these radiative forcings directly impact the ocean surface, as manifested in the immediate and abrupt increase in seasonal sea ice, reduced northward heat transport and suppression of SSTs (Miller et al., 2012). The phase relationship between the B997-316 GGC surface proxies and GDGTs suggests that it may have taken up to a century for the radiative forcing in contact with the surface to propagate to the subsurface.

The subsurface warming observed between the two subsurface cold anomalies (~1530-1745 CE, Fig. 6d) may suggest a reduced influence of the colder Arctic water mass that generally characterized the LIA. However, similar to our surface proxies (Fig. 6a-b) and the alkenone-derived SST from MD99-2275 (Fig. 6c), a local schlerochronological ^{14}C record (ΔR_{shell}) constructed from mollusk shells reflects the continued dominance of older Arctic waters in the benthos as well (Fig. 6e, Wanamaker et al., 2012). In fact, between 1450-1650 CE, the combination of our IP_{25} and triene Z datasets suggest thicker and more permanent sea ice above the B997-316 site (Fig. 6a-b), an interpretation supported by additional LIA sea ice proxy (IP_{25} and quartz) records from the NIS (Massé et al., 2008; Andrews et al., 2009b; Cabedo-Sanz et al., 2016a). If thick sea ice conditions are maintained throughout the year, the insulating effects of sea ice would warm the subsurface waters during winter, as reflected by our GDGT record (Fig. 6d). The thickening of sea ice that we observe at 1450 CE coincides with the local intensification of LIA terrestrial cooling manifested in the synchronous advance of local Icelandic ice caps (Larsen et al., 2011; Harning et al., 2016a), and reduced Icelandic lake productivity (Geirsdóttir et al., 2013, 2019; Harning et al., 2018). A previous data-modeling comparison showed that this LIA intensification was likely forced by another episode of high stratospheric sulfate loading from explosive tropical volcanism and sustained by sea-ice/ocean feedbacks that reflected incoming solar radiation during summer (Miller et al., 2012). We suggest that the winter sea ice insulation may be another important component of the sea-ice/ocean feedback to consider in the non-linear nature of Little Ice Age cooling around Iceland.

Following the dissipation of thick sea ice conditions at 1650 CE, rising IP_{25} and triene Z concentrations suggest the return to seasonal sea ice conditions that favored the co-productivity of sea ice and open water algae at the B997-316 site (Fig. 6a-b). The change in sea ice conditions is the likely mechanism for the return of lower subT at 1745 CE, inferred from low GDGT temperature anomalies (Fig. 6d). We hypothesize that as the sea ice thinned out during spring months, possibly spurred by the previously stored subsurface heat, the winter ice pack would have also thinned accordingly. Consequently, a more open winter sea ice pack would have facilitated increased heat flux from the ocean to the colder overlying atmosphere, as reflected by the lower GDGT-based subsurface temperatures.

6. Conclusion

Consistent with the community's growing comprehension of GDGT-based temperature records at high latitudes, we show that archaeal isoprenoid GDGT distributions (TEX_{86}^{L}) around Iceland predominately reflect winter subsurface temperatures (0-200 m). Furthermore, by developing a local calibration based on a network of surface sediment samples, reconstructed NIS subsurface temperature estimates and uncertainty are improved upon those obtained from regional and global calibrations. Our TEX_{86}^{L} subsurface paleotemperature record from the NIS captures the cooling likely associated with the LIA (1250-1900 CE), as seen in additional surface proxies (sea ice and marine productivity) from the same sediment core. However, the LIA onset, intensification, and termination of the subsurface lags those changes of the surface, suggesting that it may have up to a century for changes at the surface to propagate to the subsurface during the late Holocene. We propose that the development of thick sea ice conditions during the intensification of the LIA around 1450 CE insulated the subsurface in winter, resulting in apparently warmer seasonal subsurface waters. This mechanism likely represents another seasonal component of the sea-ice/ocean feedback to be considered in the abrupt cooling manifested in and around Iceland during the LIA.

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TABLES and FIGURES (8 units):

Table 1: Radiocarbon information. ^{14}C ages calibrated in Calib 7.1 (Stuiver et al., 2018) using the MARINE13 calibration curve (Reimer et al., 2013) and a ΔR of 0. Note that ^{14}C and calibrated ages are presented as BP (Before Present) in this table, and as CE (Common Era) in the main text.

Sediment core	Sediment depth (cm)	Lab ID	Material	$\delta^{13}C$ (‰)	Conventional ^{14}C date BP ± σ	ΔR	Calibrated age BP ± σ
B997-316 SGC	7.5	GRL-1691-S	mollusk (*T. equalis*)	0.62	294 ± 91	0	<400
B997-316 SGC	18	GRL-1690-S	mollusk (*T. equalis*)	-7.2	402 ± 38	0	<400
B997-316 GGC	49.5	CURL-18624	foraminifera (*N. labradorica* and *G. auriculata arctica)*	-14	1030 ± 15	0	600 ± 35
B997-316 GGC	135	CURL-19693	mollusk (*T. equalis*)	-9	1040 ± 15	0	620 ± 25
B997-316 GGC	160	CURL-19511	mollusk (*T. equalis*)	-8.5	1075 ± 15	0	645 ± 15
B997-316 GGC	212.5	CURL-20191	mollusk (*T. equalis*)	-5	1245 ± 15	0	780 ± 35

Table 2: Surface sediment calibration information. * indicates data from Rodrigo-Gámiz et al. (2015).

Site ID	Latitude	Longitude	Water depth (mbsl)	TEX_{86}^{L}
B997-313	66.617000°	-23.933000°	213	-0.67
B997-315	66.736000°	-24.332000°	217	-0.69
B997-316	66.746000°	-18.792000°	658	-0.68
B997-319	66.447000°	-18.843000°	422	-0.72
B997-324	66.527000°	-21.152000°	281	-0.65
B997-334	66.410000°	-21.880000°	112	-0.70
B997-329	65.965000°	-21.294000°	112	-0.68
B997-331	66.136000°	-21.591000°	165	-0.71
B997-344	64.836000°	-24.369000°	284	-0.61
B997-346	64.927000°	-24.129000°	320	-0.63

B997-347	63.928000°	-24.482000°	327	-0.64
Station 1*	62.000317°	-15.999183°	2255	-0.49
Station 7*	61.498550°	-24.172250°	1628	-0.51
Station 3*	63.366200°	-16.628267°	240	-0.59
Station 5*	63.583267°	-22.143733°	188	-0.62
Station 6*	63.238233°	-22.561417°	315	-0.61
Station 8*	64.293183°	-24.147083°	260	-0.62
Station 10*	66.677450°	-24.179500°	241	-0.64
Station 11*	66.633317°	-20.833433°	367	-0.63
Station 13*	67.501633°	-15.069217°	884	-0.71
Station 14*	66.303100°	-13.972817°	262	-0.68

Fig. 1: A) Overview maps of modern Icelandic oceanography. A) February 2014 and B) May 2014 50 m depth *in situ* temperature integrated from local CTD stations. Marine sediment cores (black dots) and used B997 surface sediment sample locations (black + and B997-316 GGC core site) are marked. C) May 2014 S-N trending cross section of NIS bathymetry and vertical *in situ* temperature structure along the Siglunes transect (A-A' in panels A and B) and through the B997-316 GGC marine sediment core site. Data from Hafrannsóknastofnun (Marine and Freshwater Research Institute, http://www.hafro.is/Sjora/).

Fig. 2: CLAM age model. Gray shaded area denotes the 95% confidence envelope (Blaauw, 2010). Teal and asterisked mollusk ages are from the adjacent short gravity core, B997-316 SGC. Radiocarbon information provided in Table 1.

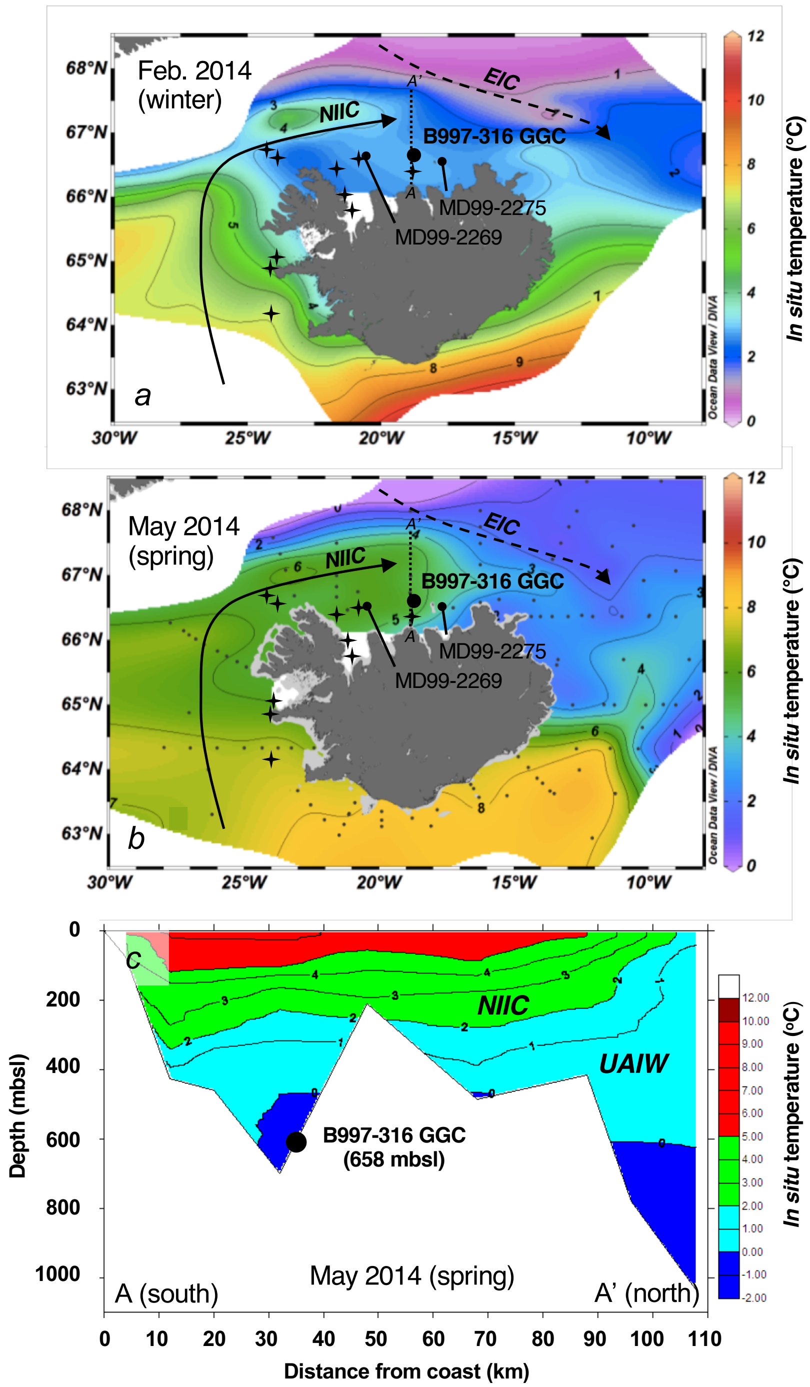
Fig. 3: B997-316 GGC marine sediment core climate proxies over the last millennium. A) % quartz, B) % calcite, C) triene Z concentrations, D) diene II concentrations, E) IP_{25} concentrations, and F) TEX_{86}^{L}. Blue boxes highlight colder, LIA-like conditions reflected in the surface climate proxies (A-E) and the subsurface proxy (F).

Fig. 4: Regression analysis summary of surface sediment GDGT calibration. A) Correlation coefficient (R^2) of all 21 surface sediment TEX_{86}^{L} values (Rodrigo-Gámiz et al., 2015; this study)

against seasonal and annual temperature depth integrations. B) Calibration of Icelandic marine surface sediment TEX_{86}^{L} values against winter 0-200 m temperature, where gray lines denote the 95% confidence envelope. Surface sediment data shown as closed circles (this study) and open circles (Rodrigo-Gámiz et al., 2015).

Fig. 5: Comparison of the available TEX_{86}^{L} temperature calibrations on the B997-316 GGC sediment record. Icelandic winter subsurface temperature (this study), annual SST (Kim et al., 2010) and annual subsurface temperature (Kim et al., 2012). Modern (1995-2004 CE) winter subsurface temperature at the B997-316 GGC site marked with gray dashed line (Locarnini et al., 2010).

Fig. 6: Comparison of select B997-316 GGC marine climate proxies to other well-dated Icelandic NIS marine climate records. A) B997-316 GGC IP_{25} concentrations (this study), B) triene Z concentrations (this study), C) MD99-2275 alkenone-inferred SST (Sicre et al., 2011), D) B997-316 GGC GDGT-inferred subsurface temperatures, with values below the record mean (4.34 ^{o}C) highlighted in blue (this study), and E) schlerochronological ΔR record, where increases in ΔR_{shell} values reflect the incursion of older, Arctic waters (Wanamaker et al., 2012). Vertical yellow bars highlight the period of interpreted thick sea ice, and the associated insulation/warming of the subsurface. Dashed blue lines bound the inferred periods of LIA-like conditions for the surface (A-C) and subsurface (D).



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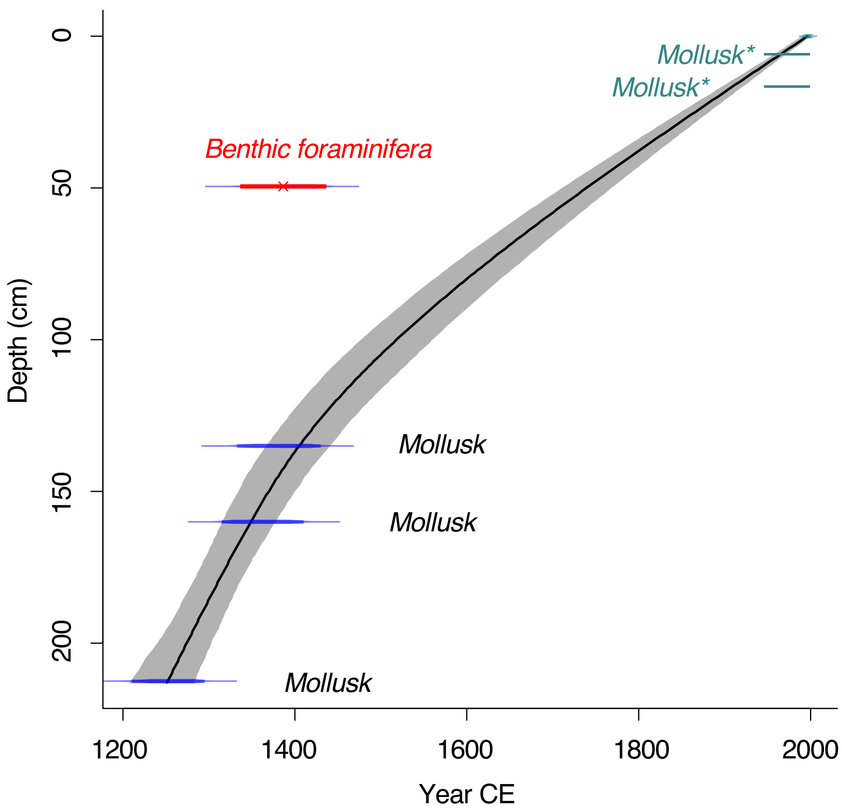

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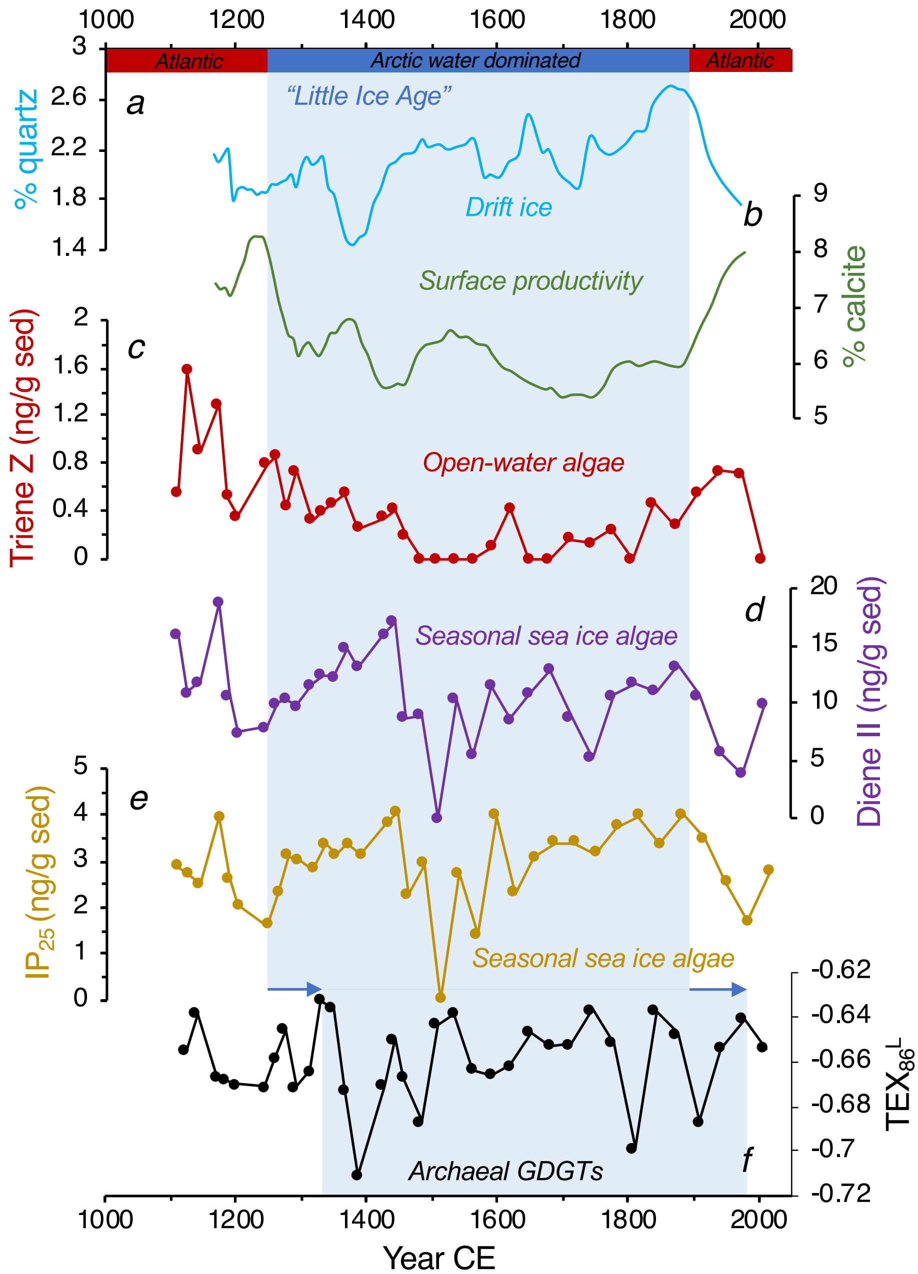

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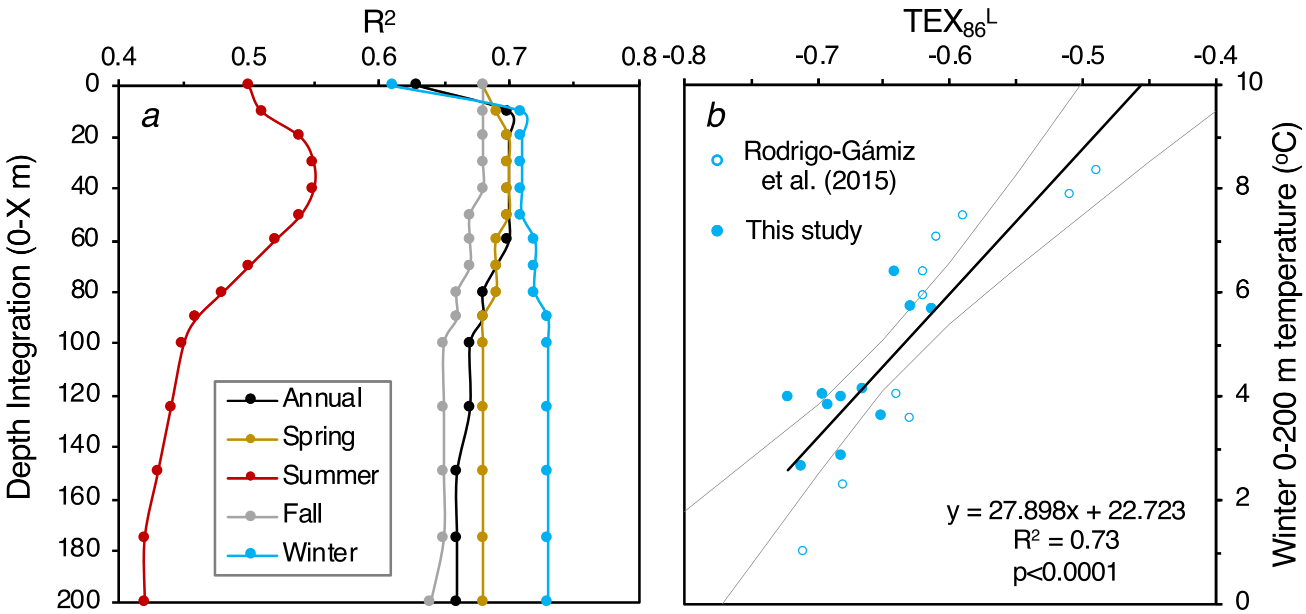

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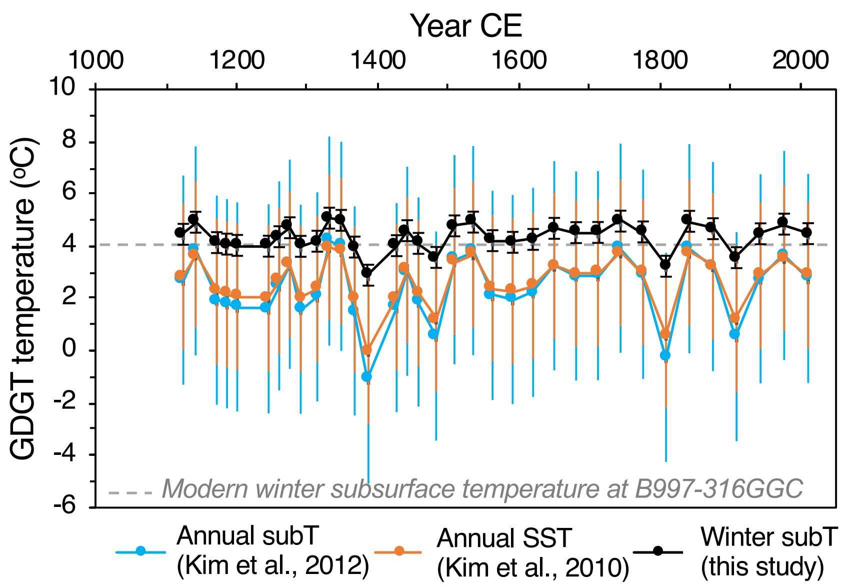

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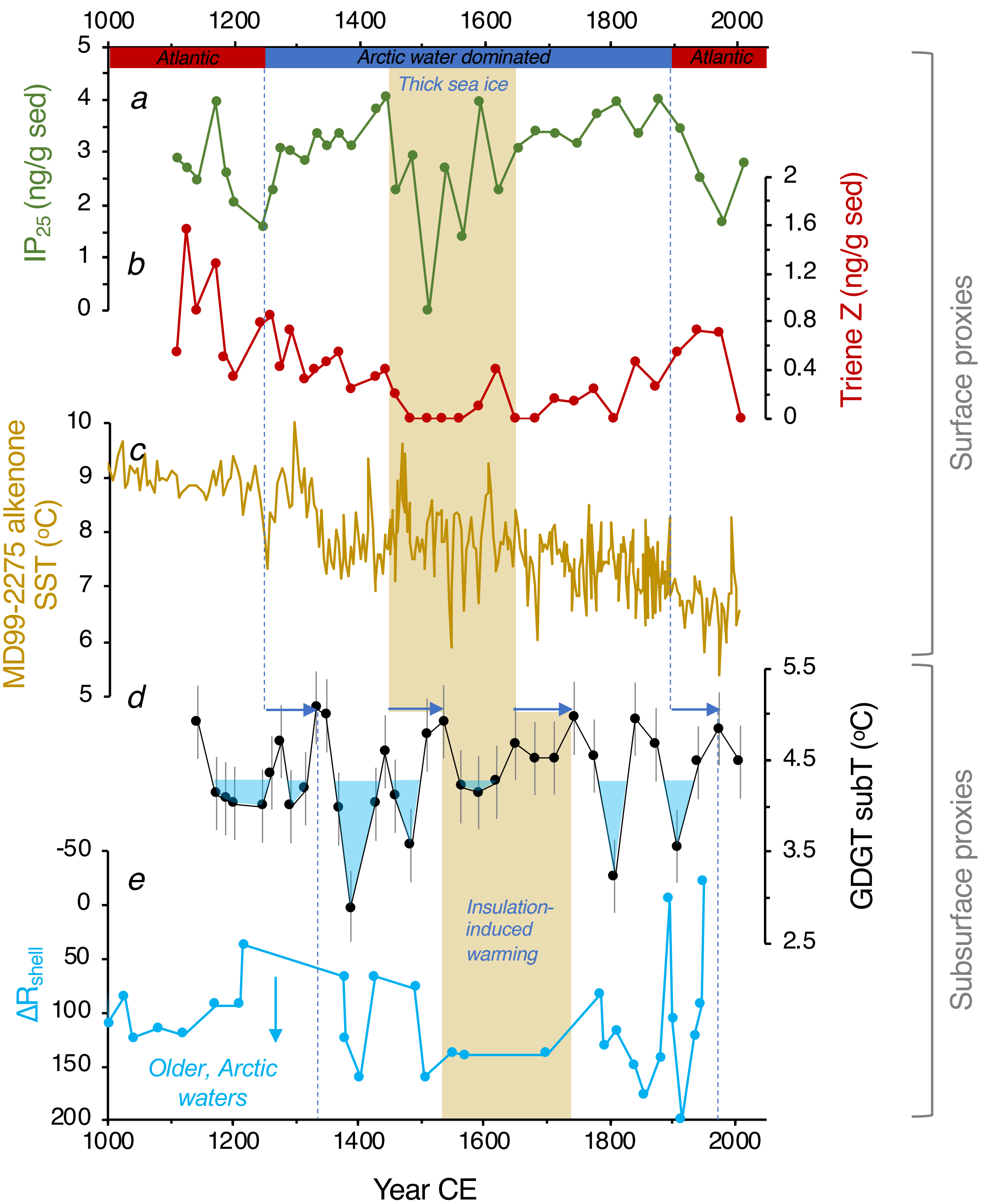
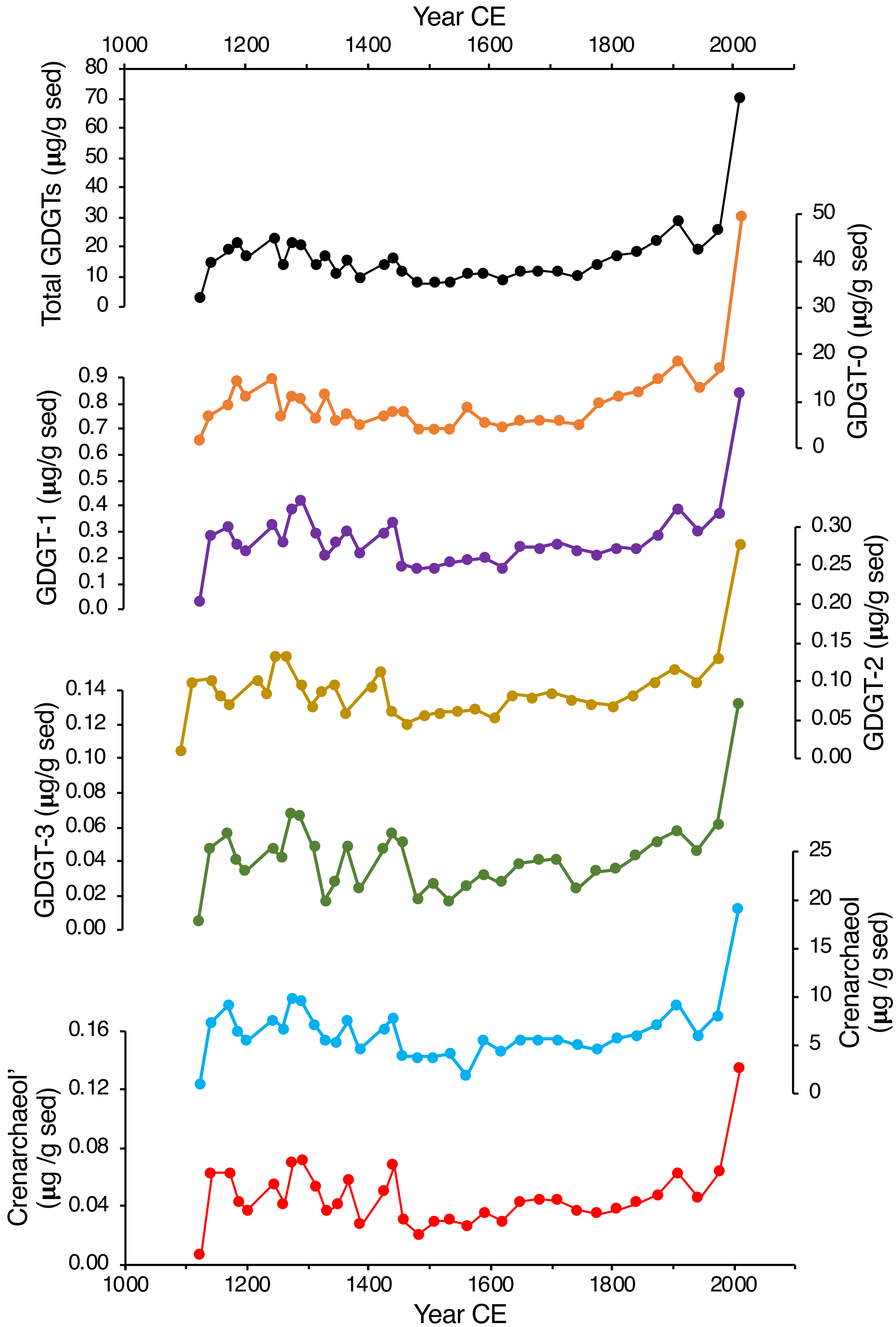
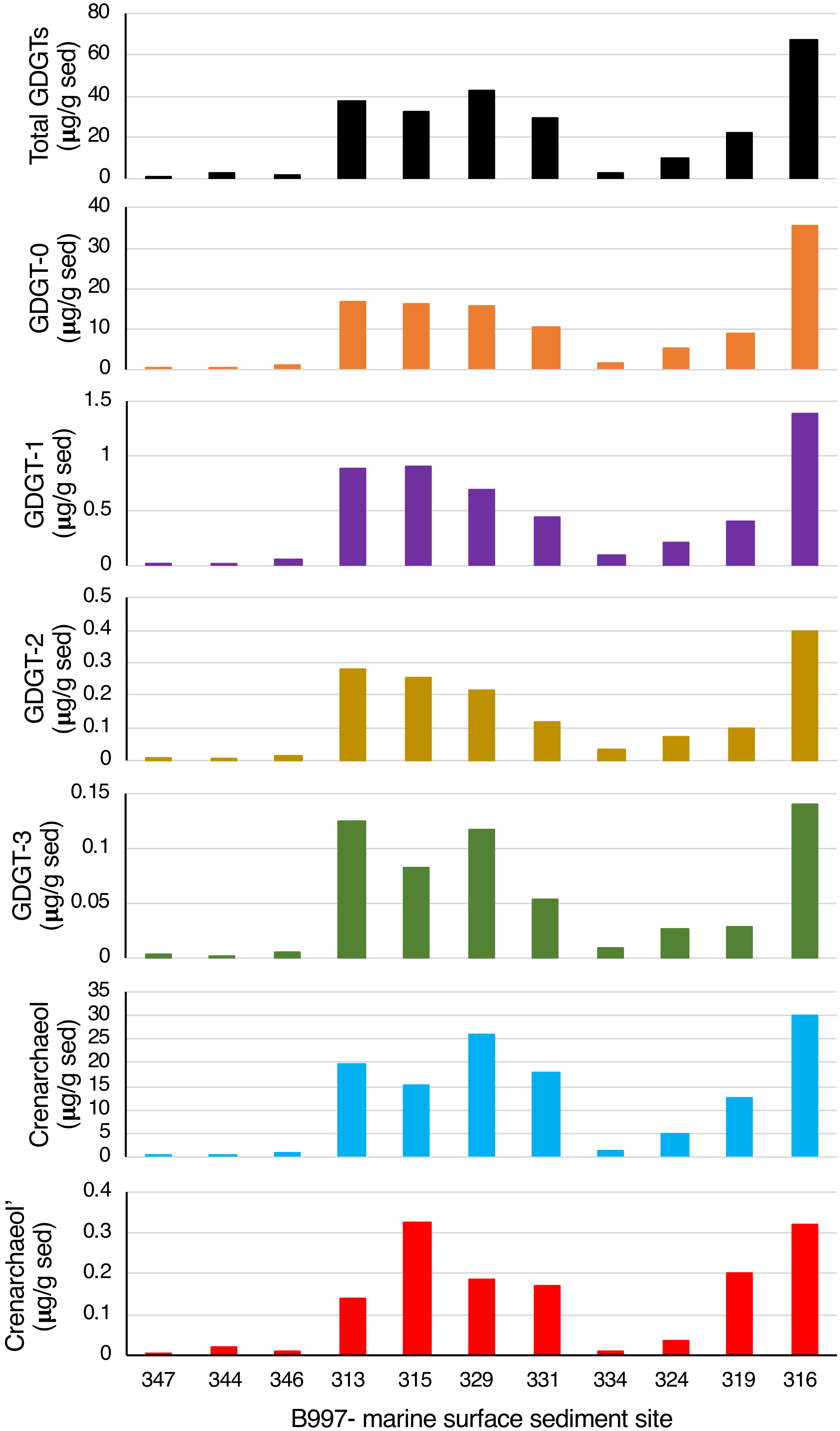


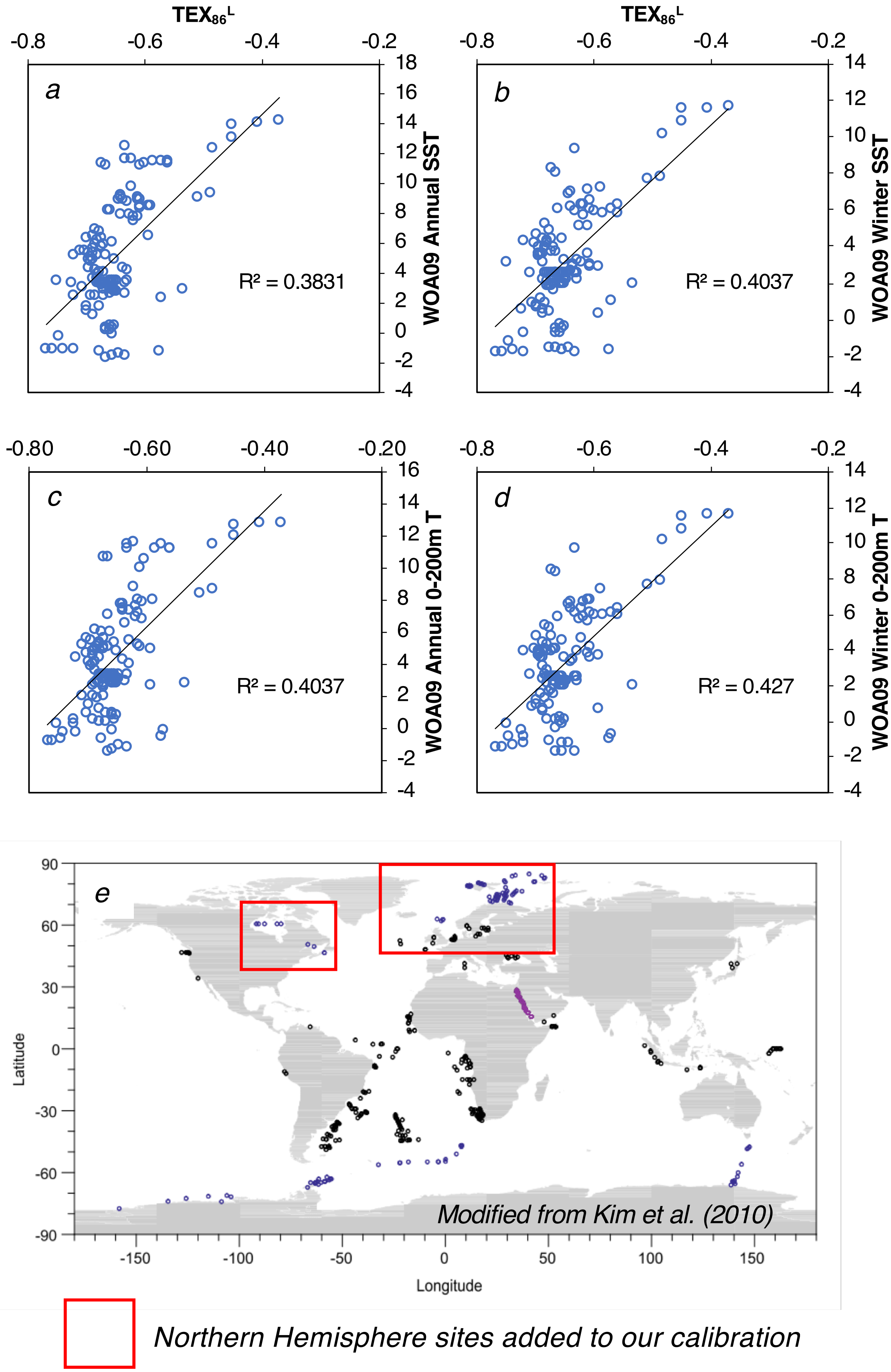
Fig. 6: Comparison of select B997-316 GGC marine climate proxies to other well-dated, high-resolution Icelandic NIS marine climate records. A) B997-316 GGC IP_{25} concentrations (this study), B) Triene Z concentrations (this study), C) MD99-2275 alkenone-inferred SST (Sicre et al., 2011), D) B997-316 GGC GDGT-inferred subsurface temperatures, with values below the record mean highlighted in blue (this study), and E) schlerochronological ΔR record, where increases in ΔR_{shell} values reflect the incursion of older, Arctic waters, and a weaker AMOC (Wanamaker et al., 2012). Vertical yellow bars highlight the period of interpreted thick sea ice, and then the delayed associated insulation/warming of the subsurface. Dashed blue lines bound the inferred periods of LIA-like conditions for the surface (A-C) and subsurface (D) proxies.



Supplemental Fig S1: GDGT concentrations in B997-316 GGC marine sediment samples.



Supplemental Fig S2: GDGT concentrations in B997 marine surface sediment samples. Sample labels are abbreviated (i.e., 347 = B997-347) and ordered geographically from the southwestern-most (347, left) to the northeastern-most (316, right).



Supplemental Fig S3: Using samples selected from the global calibration of Kim et al. (2010) we tested whether we could improve the local calibration by extending the range of samples, and thus, the environmental gradient. A) Annual SST, B) Winter SST, C) Annual 0-200 m T, and D) 0-200m T. Panel E highlights the northern hemisphere samples included from the the global calibration of Kim et al. (2010).