

Applicability of
organic temperature
proxies around
Iceland

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Constraints on the applicability of the organic temperature proxies $U_{37}^{K'}$, TEX_{86} and LDI in the subpolar region around Iceland

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Abstract

Subpolar regions are key areas to study natural climate variability due to their high sensitivity to rapid environmental changes, particularly through sea surface temperature (SST) variations. Here, we have tested three independent organic temperature proxies (U_{37}^K , TEX_{86} and LDI) on their potential applicability for SST reconstruction in the subpolar region around Iceland. U_{37}^K , TEX_{86} and TEX_{86}^L temperature estimates from suspended particulate matter result in a substantial discrepancy with instrumental data, while long chain alkyl diols were below detection limit in most of the stations. In the northern Iceland basin sedimenting particles revealed a seasonality in lipid fluxes similar to that in the bulk flux and the net primary production, i.e. highest flux of alkenones and GDGTs were measured during late spring-summer, and late summer for long chain alkyl diols. The flux-weighted average temperature estimates had a significant negative (ca. 2.3 °C for U_{37}^K) and positive (up to 5 °C for TEX_{86}) offset with satellite-derived SSTs and the temperature derived from the underlying surface sediment. In contrast, sedimentary U_{37}^K estimates temperatures around Iceland correlate well with summer mean temperatures, while TEX_{86} derived temperatures correspond well with either annual or winter mean 0–200 m temperatures, suggesting a subsurface temperature signal. Anomalous LDI-SST values in surface sediments, and low mass flux of 1,13- and 1,15-diols compared to 1,14-diols, suggest that *Proboscia* diatom is the major source of long chain alkyl diols in this area rather than eustigmatophyte algae, and therefore the LDI cannot be applied in this region.

1 Introduction

Several organic proxies, based on different lipids, have been developed for estimating sea surface temperatures (SST) (Brassell et al., 1986; Schouten et al., 2002; Rampen

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et al., 2012). One of the first organic temperature proxy developed was the $U_{37}^{K'}$ index (Prahl and Wakeham, 1987), which is based on the relative abundances of C_{37} di- and tri-unsaturated ketones. Culture and core tops studies demonstrated that haptophyte algae adjust the degree of alkenone unsaturation in response to their growth temperature, and the $U_{37}^{K'}$ index is strongly related with average annual mean SST (Prahl and Wakeham, 1987; Müller et al., 1998). However, the $U_{37}^{K'}$ index may be affected by variation in nutrient concentrations, light limitation, and diagenesis (e.g. Hoefs et al., 1998; Gong and Hollander, 1999; Prahl et al., 2003; Rontani et al., 2013).

Another organic temperature proxy commonly used in the last decade is the TEX_{86} (Schouten et al., 2002, 2013) based on a ratio of glycerol dialkyl glycerol tetraethers (GDGTs) with a varying number of cyclopentane moieties in the membrane lipids of marine Thaumarchaeota (Sinninghe Damsté et al., 2002). The TEX_{86} is strongly correlated with satellite-derived annual mean SST in global core-top datasets (Kim et al., 2008, 2010; Ho et al., 2014). However, marine Thaumarchaeota occur throughout the whole water column (e.g., Karner et al., 2001), and thus the TEX_{86} is often reflecting the water temperature of subsurface water masses (e.g., Huguet et al., 2007). The TEX_{86} -SST calibrations by Kim et al. (2010) distinguish between low temperature ($< 15^\circ C$, TEX_{86}^L) and high temperature ($> 15^\circ C$, TEX_{86}^H) regions, which takes into account an increase influence of the crenarchaeol regio-isomer in the high temperature region. Furthermore, a subsequent re-calibration of TEX_{86}^L with depth-integrated annual mean temperatures from 0 to 200 m water depth was established following evidence of abundant subsurface Thaumarchaeota during winter in Antarctic regions (Kim et al., 2012b). The TEX_{86} seems to be less affected by diagenesis than the $U_{37}^{K'}$ index (Schouten et al., 2004; Kim et al., 2009b), but it can be biased by contributions of soil-derived isoprenoid GDGTs, which can be assessed by the BIT (Branched and Isoprenoids Tetraether) index (Hopmans et al., 2004), when BIT values are $> \sim 0.3$ (Weijers et al., 2006).

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Recently, Rampen et al. (2012) proposed the long chain diol index (LDI), based on the fractional abundances of long chain C_{30} 1,15-alkyl diol relative to those of C_{28} 1,13-, C_{30} 1,13- and C_{30} 1,15-alkyl diols (hereafter referred as diols) showing a strong correlation with annual mean SST in globally distributed surface sediments. The LDI proxy seems to be independent of salinity and nutrient variations but the effect of degradation or nutrient limitation is not yet known. C_{28} and C_{30} 1,13-diols and C_{30} and C_{32} 1,15-diols have been reported in eustigmatophyte algae (Volkman et al., 1992, 1999; Gelin et al., 1997; Méjanelle et al., 2003) but since these algae are not widely distributed in open ocean settings and their diol distributions differ from those found in the natural environment, there are still uncertainties about the biological source of long chain 1,13- and 1,15-diols in marine sediments (Versteegh et al., 1997, 2000; Rampen et al., 2012). Besides this set of diols, 1,14-diols have also been identified in both marine sediments and *Proboscia* diatoms cultures, which are thought to be their source (Sinninghe Damsté et al., 2003; Rampen et al., 2007) as well as the marine alga *Apedinella radians* (Rampen et al., 2011).

SST reconstructions derived from the various organic molecular proxies can differ as proxies may reflect temperatures of different seasons, different habitat depths and/or sensitivity to environmental changes. Importantly, the use of organic proxies at high latitude regions is often problematic. Previous studies have raised doubts about the applicability of alkenone paleothermometry at high latitudes, due to the nonlinearity of the relationship of U_{37}^K index with SST at low temperatures ($< 6^\circ\text{C}$) and the high, erratic, abundance of the $C_{37:4}$ alkenone (e.g., Sikes and Volkman, 1993; Rosell-Melé et al., 1994; Rosell-Melé, 1998; Rosell-Melé and Comes, 1999; Conte et al., 2006). Concerning the TEX_{86} , studies in subpolar regions have observed significant deviations in reconstructed SST, even with the TEX_{86}^L calibration, (Ho et al., 2014 and references therein) as well as a substantial scatter in the correlation (Kim et al., 2010). The LDI has been applied thus far in the mid-latitudes of the Northern (Rampen et al., 2012; Lopes dos Santos et al., 2013; Rodrigo-Gámiz et al., 2014b) and Southern Hemispheres (Smith et al., 2013) but not in high latitude regions, although surface

sediments from high latitudes were included in the surface sediment calibration (cf. Rampen et al., 2012).

To test and constrain the application of the different organic temperature proxies at high latitudes, we have collected suspended particulate matter, sedimenting particles, and marine surface sediments from several stations distributed around Iceland. This region is of particular interest for climate studies because it is in the transition zone between polar and temperate climate regimes, thereby subjected to large variations in hydrographic conditions (Ólafsson, 1999). Thus, this high latitude region presents an ideal setting for testing and applying organic temperature proxies, including the novel LDI, in cold regions.

2 Material and methods

2.1 Oceanographic setting

The oceanographic configuration around Iceland is predominantly characterized by the interplay of two water masses, i.e., warm and saltier Atlantic water vs. cold Arctic or subpolar waters. From the South flows the Irminger Current (IC) – a branch of the warm and salty Atlantic Current, which moves northwards along the West Iceland coast and continues along the North Iceland coast, extending down to several hundred meters (Hopkins, 1991) (Fig. 1). Flowing southward from the North to the West Iceland, the East Greenland Current (EGC) transports cold and low-salinity polar waters. A branch of the EGC, the East Iceland Current (EIC), turns eastward and flows southward along the East coast of Iceland (Hopkins, 1991). The EGC carries icebergs and sea ice formed in the Arctic Ocean and in East Greenland fjords for most of the year (Sigtryggsson, 1972). The wide transitional zone between the polar waters and the Atlantic waters in the Denmark Strait is defined as the Polar Front. The position of this front is known to vary on annual, interannual and longer time scales (Malmberg and Jónsson 1997; Sigtryggsson, 1972). During episodes of extensive sea ice, the

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contribution of polar waters from the EGC to EIC is relatively large and responsible for carrying sea ice, icebergs and cold, low-salinity waters to the northwest coast of Iceland (Sigtryggsson, 1972).

2.2 Sample collection

5 Sample material was collected around Iceland during Long Chain Diol Cruises in summer of 2011 and 2012 on board of the R/V *Pelagia* (Fig. 1, Table 1). During July 2011, suspended particulate matter (SPM) from ca. 5 m water depth and surface sediments were collected at different stations (St) around Iceland (Table 1), and a sediment trap was deployed at 1850 m water depth in St 1, located in the northern
10 part of the Iceland Basin, to recover sinking particulate matter (Fig. 1). During July 2012, SPM was collected at 20 and 50 m water depth in a transect from the site of the sediment trap deployment to Reykjavik (Fig. 1, Table 1) during which the sediment trap was recovered.

SPM was obtained by filtering over 142 mm diameter glass-fiber filters (GFFs) with
15 a pore diameter of 0.7 μm using a McLane Research Laboratories WTS 6-1-142LV in situ pump installed on a CTD rosette frame. The CTD measured the vertical distribution of temperature, salinity, turbidity, oxygen and fluorescence. SPM filters were frozen directly after filtration and stored at -20°C until analysis.

A McLane Parflux 78H-21 sediment trap was set to collect sinking material each
20 17.5 days in a 21-cup automated sampling carousel covering one complete annual cycle (Table 2). Prior to mooring the sediment trap, the sample cups were filled with a mercuric chloride-poisoned and borax-buffered solution of seawater collected from the deployment depth (1 g L^{-1} of HgCl_2 ; $\text{pH} \sim 8.5$). After recovery the sediment trap, collecting cups were stored in the dark at 4°C .

25 Surface sediment samples were taken using a multicorer, sliced in 1 cm intervals and frozen onboard.

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2.3 Extraction and lipid fractionation

In the laboratory, any larger “swimmers” were removed from the sediment trap collecting cups prior to subdividing into two volumetrically split aliquots using a Folsom wet-splitter (Sell and Evans, 1982) with a precision of > 95 %. One half was stored in the dark at 4 °C, and the second half was used for lipid analysis. The trap material was centrifuged at 3000 rpm for 15 min, followed by pipetting the water layer and washed with bidistilled H₂O (3×) to remove HgCl₂ and borax solution. The sediment trap samples (*n* = 21) and surface sediment samples (*n* = 10) were freeze-dried, homogenized in agate mortar and extracted after addition of extracted diatomaceous earth in an Accelerated Solvent Extractor 350 (ASE 350, DIONEX) using a solvent mixture of 9 : 1 (v : v) dichloromethane (DCM) to methanol (MeOH) at 100 °C and 7.6 × 10⁶ Pa. The solvent from all the extracts was reduced by TurboVap LV Caliper, dried over Na₂SO₄ and concentrated under a stream of N₂.

SPM filters (*n* = 14) were freeze-dried and half of each filter was saponified according to de Leeuw et al. (1983), by refluxing for 1 h with 1 M KOH in MeOH (96 %). After cooling, the solvent was acidified with 2 N HCl in MeOH (1 : 1, v : v) to pH 2, transferred to a separatory funnel containing bidistilled H₂O and extracted using H₂O : MeOH (1 : 1, v : v, 1×), MeOH (1×) and DCM (3×). The DCM layer was separated from the H₂O : MeOH layer and rotary evaporated to near dryness. The obtained extracts and residual filters were acid hydrolyzed (3 h reflux with 2 N HCl : MeOH, 1 : 1, v : v) and neutralized with 1 M KOH in MeOH (96 %). Residuals were extracted in a separatory funnel as described previously while for extracts, 3 mL bidistilled H₂O was added and lipids were extracted using DCM (4×). All extracts were combined, dried under N₂, eluted in DCM over a pipette column containing Na₂SO₄ and dried under a stream of N₂.

Three internal standards were added to the total lipid extracts (TLEs) of sedimenting particles and SPM transect, i.e. 10-nonadecanone (C₁₉ ketone) for alkenones, C₂₂ 7,16-diol for long chain diols and the C₄₆ glycerol trialkyl glycerol tetraether (GTGT) for GDGTs (Huguet et al., 2006b). For some surface sediments and SPM around

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Iceland only the C₂₂ 7,16-diol standard was added to the TLEs. Activated copper and DCM were added to some of the TLEs from the sediment trap material to remove elemental sulphur. After stirring overnight with a small stirring bar, TLEs were filtered over a pipette column containing Na₂SO₄ and dried under a stream of N₂.

Extracts of SPM, descending particles and surface sediments were separated into apolar, ketone (containing alkenones) and polar fractions (containing GDGTs and long chain diols) by column chromatography using a Pasteur pipette filled with Al₂O₃ (activated for 2 h at 150 °C) using 9 : 1 (v : v) hexane:DCM, 1 : 1 (v : v) hexane:DCM, and 1 : 1 (v : v) DCM : MeOH as the eluents, respectively.

2.3.1 Alkenone analysis

The ketone fraction was dried under N₂ and re-dissolved in an appropriate volume (20–100 µL) of hexane. Analysis of the di- (C_{37:2}) and tri-unsaturated (C_{37:3}) alkenones was performed on an Hewlett Packard 6890 Gas Chromatograph (GC) using a 50 m CP Sil-5 column (0.32 mm diameter, film thickness of 0.12 µm), equipped with flame ionization detector and helium as the carrier gas. The temperature of the oven was initially 70 °C and increased with a rate of 20 °C min⁻¹ to 200 °C and subsequently with a rate of 3 °C per min to 320 °C, at which it was held for 25 min. Alkenone relative abundances were determined by integration of relevant peak areas.

The U₃₇^{K'} index (Eq. 1) was used to estimate SSTs according to the equation by Prahl et al. (1988):

$$U_{37}^{K'} = [C_{37:2}] / ([C_{37:2}] + [C_{37:3}]) \quad (1)$$

U₃₇^{K'} values were converted to SSTs using the global core top calibration of Müller et al. (1998):

$$U_{37}^{K'} = 0.033 \cdot SST + 0.044 \quad (2)$$

Five samples were run in duplicate resulting in a SD of 0.02 or better, equivalent to 0.8 °C.

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2.3.2 GDGT analysis

Polar fractions of the extracts, containing the GDGTs, were dried under a stream of N₂, redissolved by sonication (5 min) in 200 μL hexane/propanol (99 : 1, v : v), and filtered through 0.45 μm polytetrafluoroethylene (PTFE) filters. GDGTs were analyzed by high-pressure liquid chromatography–mass spectrometry (HPLC/MS) following the method described by Schouten et al. (2007). Samples were analyzed on an Agilent 1100 series LC/MSD SL. A Prevail Cyano column (150 mm × 2.1 mm, 3 mm) was used with hexane : propanol (99 : 1, v : v) as an eluent. After the first 5 min, the eluent increased by a linear gradient up to 1.8% isopropanol (vol) over the next 45 min at a flow rate of 0.2 mL min⁻¹. Scanning was performed in single ion monitoring (SIM) mode. Identification and quantification of the GDGTs isomers and C₄₆ GTGT standard was achieved by integrating the peak areas of relevant peaks in *m/z* 1302, 1300, 1298, 1296, 1292, 1050, 1036, 1022 and 744 mass chromatograms.

The TEX₈₆ and TEX₈₆^L index were calculated following Kim et al. (2010):

$$\text{TEX}_{86} = \frac{[\text{GDGT 2}] + [\text{GDGT 3}] + [\text{cren}']}{[\text{GDGT 1}] + [\text{GDGT 2}] + [\text{GDGT 3}] + [\text{cren}']} \quad (3)$$

$$\text{TEX}_{86}^{\text{L}} = \frac{[\text{GDGT 2}]}{[\text{GDGT 1}] + [\text{GDGT 2}] + [\text{GDGT 3}]} \quad (4)$$

where numbers correspond to isoprenoid GDGTs from marine Thaumarchaeota with 1, 2 or 3 cyclopentane moieties, and cren' corresponds to crenarchaeol regioisomer, which has the antiparallel configuration of crenarchaeol (Sinninghe Damsté et al., 2002).

As present-day mean annual SST around Iceland is lower than 10 °C, TEX₈₆ and TEX₈₆^L values were converted to SSTs using calibrations (Eqs. 5 and 6) proposed by

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Kim et al. (2010):

$$\text{SST} = 81.5 \cdot \text{TEX}_{86} - 26.6 \quad (5)$$

$$\text{SST} = 67.5 \cdot \log(\text{TEX}_{86}^L) + 46.9 \quad (6)$$

Calibration errors are 5.2 and 4 °C, respectively, due to the large scatter in the polar regions (Kim et al., 2010).

Furthermore, the Kim et al. (2012b) TEX_{86}^L temperature calibration with 0–200 m water depth was also used:

$$T(0 - 200 \text{ m}) = 50.8 \cdot \log(\text{TEX}_{86}^L) + 36.1 \quad (7)$$

The Branched and Isoprenoid Tetraether (BIT) index, a measure for soil vs. marine organic matter input in marine sediments, was calculated according to Hopmans et al. (2004):

$$\text{BIT} = \frac{[\text{GDGT-I}] + [\text{GDGT-II}] + [\text{GDGT-III}]}{[\text{Crenarchaeol}] + [\text{GDGT-I}] + [\text{GDGT-II}] + [\text{GDGT-III}]} \quad (8)$$

where roman numerals correspond to the major branched GDGTs (see Hopmans et al., 2004).

A total of 17 samples were run in duplicate for TEX_{86} and the BIT, showing a mean SD 0.09, equivalent to 1.0 °C or better for TEX_{86}^L , and a SD of 0.002 or better for the BIT index.

2.3.3 Long chain diol analysis

After GDGT analysis, polar fractions were silylated by adding 15 μL N_2O bis(trimethylsilyl)trifluoroacetamide (BSTFA) and pyridine and heating in an oven at 60 °C for 20 min. Long chain diol distributions were analyzed using a Thermo Finnigan Trace Gas Chromatograph (GC) Ultra coupled to Thermo Finnigan DSQ

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0.26 and 0.45 (corresponding to 6.5 to 12.5 °C) during summer 2012 (Fig. 2b, open green diamonds). GDGTs were detected in all samples and the TEX_{86} values ranged between 0.49 and 0.55 (corresponding to 13.1 to 17.9 °C) around Iceland (Fig. 2a, dark blue circles) and between 0.34 and 0.50 (corresponding to 1.0 to 14.4 °C) along the transect of 2012 (Fig. 2b, dark blue circles) based on the linear calibration proposed by Kim et al. (2010). TEX_{86}^L varied between 0.28 and 0.32 (corresponding to 9.4 to 13.7 °C) around Iceland (Fig. 2a, open blue circles) and between 0.22 and 0.29 (corresponding to 2.5 to 10.6 °C) along the transect (Fig. 2b, open blue circles). Long chain alkyl diols were not detected in SPM around Iceland collected during summer 2011 (Fig. 2a, b), while C_{28} and C_{30} 1,13- and 1,14-diols, and C_{30} and C_{32} 1,15-diols were only detected in SPM collected at St A, B, F and G during the transect in the summer of 2012. LDI values varied between 0.08 and 0.49 (Fig. 2b, open brown squares) or -0.4 to 12 °C when converted into temperature.

3.2 Descending particulate matter

Bulk sediment fluxes varied between 4 and 165 $\text{mg m}^{-2} \text{ day}^{-1}$ (Fig. 3b), with high fluxes in July, September and October 2011, and from May to July 2012. C_{37} alkenone fluxes varied between 0.02 and 130 $\mu\text{g m}^{-2} \text{ day}^{-1}$, peaking during spring-summer seasons, i.e. in July 2011 and during May and June 2012 (Table 2; Fig. 3c). Fluxes of the GDGTs used for the calculation of the TEX_{86} index ranged between 53 and 2300 $\mu\text{g m}^{-2} \text{ day}^{-1}$ with highest values from May to June 2012 (Table 2; Fig. 3d). GDGT-0 and crenarchaeol fluxes followed the same pattern as those of the GDGTs used in the TEX_{86} , ranging from minimum values of ca. 170 $\mu\text{g m}^{-2} \text{ day}^{-1}$ to maximum of 15 and 11 $\text{mg m}^{-2} \text{ day}^{-1}$, respectively (Fig. 3d). BIT values were always below < 0.01. Fluxes of long chain 1,13- and 1,15-diols used in the LDI were low compared to alkenones and GDGT fluxes and varied between 1.5 and 170 $\text{ng m}^{-2} \text{ day}^{-1}$, with highest values during July, September and October 2011, and from May to June 2012 (Table 2; Fig. 3e). Generally, the flux of the C_{30} 1,15-diol was always low (up to 2 $\text{ng m}^{-2} \text{ day}^{-1}$) or even below detection

4 Discussion

4.1 $U_{37}^{K'}$

Long chain alkenones are produced by several haptophyte algal species thriving in the photic zone (Volkman et al., 1980, 1995; Marlowe et al., 1984), and are, therefore, thought to reflect SST. Although previous studies of cold polar waters ($< 4^{\circ}\text{C}$) of the North Atlantic have shown relatively high abundances of $C_{37:4}$ (e.g., Sicre et al., 2002), C_{37} alkenones in SPM, descending particles and surface sediments around Iceland comprised only $C_{37:3}$ and $C_{37:2}$ and no $C_{37:4}$ was detected. Comparison of $U_{37}^{K'}$ -derived temperatures with in-situ temperatures showed generally lower $U_{37}^{K'}$ -derived temperatures of up to 3.4°C for SPM around Iceland (Fig. 2a) and up to 6.6°C for the SPM transect (Fig. 2b). A reduced temperature differences (up to 2.6°C) was observed when we compared $U_{37}^{K'}$ -derived SST with summer temperatures at 50 m water depth (Fig. 2b, purple crosses; derived from the World Ocean Atlas (WOA) 09 database; Locarnini et al., 2010) at which most of the SPM was recovered from the transect. Possibly, the alkenones collected in the SPM were not representing recently produced material but alkenones synthesized over several months. Since SPM was collected in July, the warmest month of the year, the $U_{37}^{K'}$ would reflect lower temperatures if the signal also reflected material synthesized in the preceding colder months.

Interestingly, $U_{37}^{K'}$ -derived SSTs of sedimenting particles also show major discrepancies compared to satellite SSTs, i.e. somewhat higher $U_{37}^{K'}$ -derived SSTs were observed from January to mid-May (around $2\text{--}3^{\circ}\text{C}$) and lower temperatures from mid-May to July (up to 4.9°C) (Fig. 4, green line and open diamonds) at the time of the highest alkenone flux (Fig. 3c). The underestimation of temperatures by $U_{37}^{K'}$ in sedimenting particles in July is consistent with the discrepancy between $U_{37}^{K'}$ -derived and in-situ temperature observed for SPM. Considering the calibration error of 1.5°C (Müller et al., 1998), these differences are significant. A compilation of previous

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to the flux-weighted mean. This may explain the mismatch between surface sediment signal and flux-weighted mean signal.

To test the effect of seasonality and diagenetic alkenone alteration around Iceland, we compared $U_{37}^{K'}$ -SST signal from all surface sediments with annual mean and seasonal SSTs (derived from the WOA09 database; Locarnini et al., 2010) (Fig. 6a, note color code as in Fig. 1). $U_{37}^{K'}$ -derived SSTs show a good linear correlation with annual mean SSTs, although absolute temperature values are higher at each station (from 1.0 to 4.0 °C) (Fig. 6a), generally higher than the calibration error, i.e. 1.5 °C (Müller et al., 1998), and with the highest deviations for the most northern stations. When we compared $U_{37}^{K'}$ -derived temperature values with SST from different seasons, the best fit is obtained with the summer mean SSTs (Fig. 6b). This is in agreement with peak alkenone fluxes recorded in our sediment trap during late spring-early summer. Thus, the sedimentary signal of $U_{37}^{K'}$ -SST seems to reflect the maximum production season of alkenones, i.e. the blooming period of the haptophyte algae, in accordance with C_{37} alkenones flux being the highest during late spring and early summer.

4.2 TEX_{86}

GDGTs in the marine environment are likely biosynthesized by Thaumarchaeota (Sinninghe Damsté et al., 2002) that are omnipresent in the global ocean, including the polar regions (e.g., Hoefs et al., 1997; DeLong et al., 1998; Schouten et al., 2000). TEX_{86}^L was developed for polar oceans in order to improve the correlation between TEX_{86} and SST (Kim et al., 2010), but a recent study has shown that TEX_{86} is still suitable as well (Ho et al., 2014). The TEX_{86}^L -SST estimates in the SPM around Iceland showed a highly variable relationship with in situ temperature, with up to 6 °C higher values around Iceland during 2011 (Fig. 2a), while for the SMP 2012 transect the difference with summer temperatures at 50 m water depth from WOA09 was up to 7 °C lower (Fig. 2b). Even higher offsets, both positive and negative, up to 11 °C were obtained with TEX_{86} -SST estimates (Fig. 2a, b). A reason for the poor correspondence

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of TEX₈₆-derived temperatures with in-situ and satellite temperatures could be a depth habitat effect, since Thaumarchaeota can thrive deeper in the marine water column (Karner et al., 2001; Herndl et al., 2005), although they tend to have their highest cell numbers at depths < 200 m (e.g. Karner et al., 2001). We also applied a specific TEX₈₆ SPM calibration proposed by Schouten et al. (2013), but temperatures still did show significant offsets with in situ temperatures (data not shown).

Part of this mismatch may be due to the fact that Thaumarchaeota are smaller in cell size than the 0.7 µm pore diameter of the SPM filters (Könneke et al., 2005) and thus may not be quantitatively captured on the GFFs (Ingalls et al., 2012), possibly impacting the TEX₈₆ values. However, other studies have shown comparable TEX₈₆ values obtained with both 0.7 and 0.2 µm pore diameter filters (e.g., Herfort et al., 2007) or good correspondence with depth (Schouten et al., 2012) or seasonal (Pitcher et al., 2011) profiles of isoprenoidal GDGT concentration and Thaumarchaeotal rRNA gene abundance. Thus, the filter size is unlikely to have affected TEX₈₆ values. Another issue may be that we analyzed core (non-intact) GDGT-based lipids derived from dead cells, not from living organisms, that may thus represent a fossil signal from other areas or represent an integrated annual temperature signal. Lipp and Hinrichs (2009) showed notable differences in derived temperatures using core- vs. intact polar lipids-GDGTs. Usually, IPL-GDGTs show warmer derived temperatures than core-GDGTs, as was observed in SPM from the Arabian Sea (Schouten et al., 2012), which may reduce the differences in derived-SST in the SPM transect. However, this does not fully explain the dissimilarities observed in the SPM around Iceland. Therefore, there is not a clear explanation for the offsets obtained in estimated temperatures in SPM.

Regarding sedimenting particles during the year cycle 2011–2012, TEX₈₆^L-derived SSTs (Fig. 4, blue line and open circles) were all much higher (up to ca. 9 °C) than satellite SST. Reduced differences (up to ca. 5 °C higher than satellite SSTs) were obtained when estimated temperature values using the TEX₈₆^L 0–200 m calibration (Kim et al., 2012b) were compared with 0–200 m integrated temperature using the WOA09 dataset (Fig. 4, red line and open circles). Interestingly, differences in temperature

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decreased significantly when TEX_{86} -derived temperature and satellite derived SST are compared (Fig. 4; dark blue line and filled circles), particularly during times of low GDGT flux (Fig. 3d). During times of higher GDGT fluxes, TEX_{86} -SST were lower by up to 4.6 °C, which may suggest that the temperature signal is not derived just from surface waters. We observed notable differences in estimated temperatures when we used different calibrations, obtaining better results in the sedimenting particles with the TEX_{86} calibration (Kim et al., 2010). Similar findings were observed by Ho et al. (2014) who applied TEX_{86}^L and TEX_{86} in different polar and subpolar regions, as the Pacific sector of the Southern Ocean and the Subarctic Front in the North Pacific. Lateral transport of GDGTs is not likely to have an effect on TEX_{86} temperatures since isoprenoid GDGTs are less susceptible to long distance advection than alkenones (Mollenhauer et al., 2008; Shah et al., 2008; Kim et al., 2009a). Short-term degradation has also shown to have no significant impact on the TEX_{86} (Schouten et al., 2004; Kim et al., 2009b). Terrigenous GDGTs are also unlikely to be the reason of the offset in estimated temperatures around Iceland, based on values of the BIT index (< 0.01) (Weijers et al., 2006). Comparison of the flux weighted mean TEX_{86} value with surface sediment at St 1 shows similar values, independent of calibration used, i.e. 8.5 and 9.2 °C using TEX_{86} -SST, 14.5 and 14.0 °C using TEX_{86}^L -SST, 11.7 and 11.4 °C for TEX_{86}^L 0–200 m, suggesting no alteration of the GDGT signal during transport to the sea floor (Table 3).

GDGT fluxes were high during July, September and October 2011, followed by May and June 2012 (Fig. 3d), showing similar patterns as bulk sediment flux and primary production (Fig. 3a, b). This GDGT flux pattern is not entirely consistent with the ecology of marine Thaumarchaeota, the abundance of which is often negatively correlated with that of phytoplankton, since they are chemolithoautotrophs using ammonia derived from breakdown of organic matter (e.g., Massana et al., 1997; Herndl et al., 2005; Könneke et al., 2005; Wuchter et al., 2006). However, the same GDGT flux pattern was also observed in the Arabian Sea (Wuchter et al., 2006) and Santa Barbara basin, off the coast of southern California (Huguet et al., 2007), i.e. high at

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times of high primary productivity. This was explained as a more efficient transport of thaumarchaeotal cells, and thus GDGTs, to depth by packaging activity of zooplankton thriving after a phytoplankton bloom. This could be also the case in the northern Iceland basin. The fact that highest fluxes are observed in late summer suggest that TEX_{86} here also represents summer temperatures, as was previously noted Ho et al. (2014) in surface sediments from the Arctic, northern Pacific and southern Ocean.

TEX_{86}^- and TEX_{86}^L -derived SST in the surface sediments distributed around Iceland, correlate with WOA09 annual mean SSTs (Fig. 6c, e), although they show a substantial offset of 1 to 4.7 °C. A similar fit is obtained with winter mean temperatures (Fig. 6d, f), whereas that with summer mean temperatures is substantially reduced (data not shown). This latter observation contradicts the observation that highest GDGT fluxes are recorded for late summer (Fig. 3d). In addition, this is in contrast with observations by Ho et al. (2014), who showed anomalously high SST estimates in surface sediments from the Arctic, northern Pacific and southern Ocean obtaining the best correlation of TEX_{86}^L -SSTs with summer SSTs.

To test if TEX_{86} around Iceland is mainly reflecting subsurface temperature waters as suggested for some other regions (e.g., Huguet et al., 2007; Lopes dos Santos et al., 2010; Kim et al., 2012a, b), we compared TEX_{86}^L 0–200 m temperature estimates (Kim et al., 2012b) with the temperature of the first 200 m water column based on WOA09. We obtained a better correspondence with both annual and winter mean temperatures (Fig. 6g, h), with differences ranging from 0.5 to 3.5 °C. This suggests that TEX_{86} -derived signal in the surface sediments around Iceland may reflect subsurface (i.e. 0–200 m) temperatures.

4.3 LDI

Long chain alkyl diols, either the 1,13- and 1,15-diols involved in the LDI or the 1,14-diols produced by *Proboscia* diatoms (Sinninghe Damsté et al., 2003) and *Apedinella radians* (Rampen et al., 2011), were below detection limit in SPM sampled around Iceland in summer 2011. In the SPM sampled during the 2012 transect, small amounts

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of long chain 1,15-, 1,14- and 1,13-diols were detected. This suggests that July is not the highest productivity period of any long chain diol producers around Iceland. Long chain 1,13- and 1,15-diol mass fluxes were relatively high during July, September and October 2011, and from May to July 2012 (Fig. 3e), suggesting that diol producers were present although not at the time and depths of where the SPM was sampled. This suggests a patchy distribution of diol producers. Interestingly, the fluxes of 1,13- and 1,15-diols were always much lower than C_{28} and C_{30} saturated and mono-unsaturated 1,14-diol fluxes, as well as three order of magnitude lower than those of alkenones and GDGTs. This indicates that they are not very abundant lipids in this environment, and therefore their producers are also not very abundant. The presence of C_{28} and C_{30} saturated and mono-unsaturated 1,14-diol fluxes suggest that *Proboscia* diatoms have bloomed in late summer-autumn and late spring-summer. This is supported by the identification of C_{27} and C_{29} mid-chain hydroxyl methyl alkanates in the sedimenting particles (data not shown), which are also biomarker lipids from *Proboscia* diatoms (Sinninghe Damsté et al., 2003). Indeed, it has been observed that diatom distribution and composition around Iceland is strongly influenced by different environmental variables and particularly by summer sea surface temperature (Jiang et al., 2001). Interestingly, trace amounts of both C_{28} and C_{30} 1,13-diols have been identified in *Proboscia* species (Rampen et al., 2007), and the relatively high abundance of saturated and mono-saturated 1,14-diols could suggest that *Proboscia* may also be partially source the 1,13- and 1,15-diols in this area in addition to eustigmatophyte algae.

Where LDI values could be calculated for the SPM, temperatures were substantially lower than satellite SSTs (Fig. 2b), exceeding the calibration error of 2°C (Rampen et al., 2012). For the sediment trap, LDI could also not always be calculated due to the non-quantifiable amount of C_{30} 1,15-diol. For cases where it was possible, the temperature values were, like the SPM, much lower than satellite temperatures (Fig. 4, brown line and open squares; Table 3). The eukaryotic phytoplankton responsible for the production of 1,13- and 1,15-diols are likely eustigmatophyte algae, autotrophs

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living in the upper photic zone (Volkman et al., 1992). The best correlation of LDI is with surface temperatures of late summer-early autumn (cf. Rampen et al., 2012). Thus, a contribution from colder deep water is unlikely to explain the low temperatures observed with the LDI. A similar observation is made for the LDI-derived SSTs in the surface sediments around Iceland which are always lower than annual mean SST, even when compared with the coldest season, i.e. winter mean SST (Fig. 6i, j). Furthermore, there is no correlation of LDI values with SST. Also in the surface sediments, low relative abundances of 1,13- and 1,15-diols relative to 1,14-diols were observed. The mismatch of LDI values with temperature as well as the low abundances of long chain 1,13- and 1,15-alkyl diols reinforce the hypothesis that *Proboscia* diatoms seem to be a partial source of 1,13- and 1,15-diols in the Iceland region. This suggests that the LDI may not be applicable in the region around Iceland. Furthermore, we advise that if distributions of long chain alkyl diol are dominated by 1,14-diols the LDI should generally not be applied.

5 Conclusions

The application of three independent organic temperature proxies at high latitudes was studied in the region around Iceland. $U_{37}^{K'}$ -derived SSTs based on SPM and the annual weighted alkenones flux are in general lower than annual mean SST. In contrast, a better correspondence of $U_{37}^{K'}$ -derived SSTs in the surface sediments around Iceland is observed with summer mean SST, which seems in agreement with the seasonal alkenones fluxes observed during late spring-early summer. The mismatch between both flux-weighted mean and surface sediment signals seems to be due to the seasonality effect of alkenones around Iceland or the diagenetic alkenone alteration during the sedimentation process. High fluxes of the isoprenoidal GDGTs used in the TEX_{86} proxy were also observed during warmer months with major shifts in TEX_{86} estimated temperatures. This flux pattern can be explained as a preferential transport of GDGTs to depth by packaging activity of zooplankton thriving after

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a phytoplankton bloom. In surface sediments TEX_{86} -derived temperatures showed a good correspondence either with annual or winter mean satellite temperatures of the first 200 m water depth, thus tentatively suggesting that the TEX_{86} signal is primarily derived from subsurface waters (0–200 m). In contrast, the LDI around Iceland did not show any substantial relationship with SST. The similarity in trends of all saturated long chain alkyl diols and the high abundances of 1,14-diols over 1,13- and 1,15-diols may suggest that *Proboscia* diatom is a partial source of 1,13- and 1,15-diols in the Iceland region, limiting the LDI application in this area. Thus in general, we advise for future anomalous LDI values in other areas, that when 1,14-diols are dominant in the long chain alkyl diol distributions, the index should not be confidently applied.

Author contributions. J. S. Sinninghe Damsté, S. Schouten and S. W. Rampen designed the research cruises. S. W. Rampen, M. Baas and H. de Haas were on board the research cruises. M. Rodrigo-Gámiz performed all the experimental laboratory work. M. Rodrigo-Gámiz prepared the manuscript with contributions from all co-authors.

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Table 1. Location, depth and other information of each material collected at different stations around Iceland.

Station	Latitude	Longitude	Depth (m b.s.l.)	Long (cm)	Flow meter (l)	Cross cut (l)
Sediment trap						
1	N 61° 59.757'	W 16° 00.191'	Trap top failed Trap bottom 1850			
SPM around Iceland Jul 2011						
1	N 62° 0.008'	W 16° 0.016'	5		no data	
7	N 61° 29.917'	W 24° 10.333'	5		26	
8	N 64° 17.583'	W 24° 8.811'	5		25	
10	N 66° 40.647'	W 24° 10.794'	5		130	
13	N 67° 30.098'	W 15° 4.109'	5		164	
16	N 63° 59.132'	W 12° 12.472'	6		10	
SPM transect Jul 2012						
A	N 61° 59.757'	W 16° 00.191'	20		36	34.1
A	N 61° 59.757'	W 16° 00.191'	50		229	200
B	N 62° 14.963'	W 16° 51.877'	50		199	200
C	N 62° 29.635'	W 17° 50.287'	50		183	175.5
D	N 62° 44.568'	W 18° 48.771'	50		216	200
E	N 62° 58.981'	W 19° 47.270'	50		220	200
F	N 63° 12.696'	W 20° 44.820'	50		219	200
G	N 63° 27.319'	W 21° 44.951'	50		219	200
Multicore						
1	N 62° 0.019'	W 15° 59.951'	2255	18		
3	N 63° 21.972'	W 16° 37.696'	240	5		
5	N 63° 34.996'	W 22° 8.624'	188	15		
6	N 63° 14.294'	W 22° 33.685'	315	15		
7	N 61° 29.913'	W 24° 10.335'	1628	28		
8	N 64° 17.591'	W 24° 8.825'	260	33		
10	N 66° 40.647'	W 24° 10.770'	241	30		
11	N 66° 37.999'	W 20° 50.006'	367	10		
13	N 67° 30.098'	W 15° 4.153'	884	10		
14	N 66° 18.186'	W 13° 58.369'	262	no data		

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Table 2. Sampling intervals in the sediment trap with the average flux of lipids and derived organic proxies.

Start sampling (mm/dd/yy)	Sampling interval (days)	$C_{37:2} + C_{37:3}$ Av. flux ($\mu\text{g m}^{-2}\text{day}^{-1}$)	U_{37}^K	GDGTs-TEX ₈₆ Av. flux ($\mu\text{g m}^{-2}\text{day}^{-1}$)	TEX ₈₆	TEX _{86}^L}	1,14 long chain diols Av. flux ($\text{ng m}^{-2}\text{day}^{-1}$)	1,13+1,15 long chain diols Av. flux ($\text{ng m}^{-2}\text{day}^{-1}$)	LDI
1 (07/15/11)	16.5	115	0.36	645	0.43	0.32	498	160	–
2 (08/01/11)	17.5	0.91	0.35	52.5	0.41	0.33	32	10.7	0.07
3 (08/19/11)	17.5	0.32	0.41	54.5	0.41	0.32	13.6	4.8	–
4 (09/05/11)	17.5	1.03	0.42	546	0.41	0.31	546	120	0.01
5 (09/23/11)	17.5	–	–	609	0.43	0.33	767	147	–
6 (10/10/11)	17.5	1.04	0.37	384	0.42	0.32	452	96	–
7 (10/27/11)	17.5	0.10	0.40	94	0.43	0.34	23	6.3	–
8 (11/14/11)	17.5	0.11	0.40	132	0.43	0.35	33.2	7.4	–
9 (12/01/11)	17.5	0.32	0.36	384	0.43	0.35	86	28.5	0.07
10 (12/19/11)	17.5	0.27	0.34	222	0.44	0.36	31.2	11	0.09
11 (01/05/12)	17.5	0.18	0.38	274	0.44	0.37	57	16.2	–
12 (01/23/12)	17.5	0.20	0.37	297	0.44	0.36	46	12	–
13 (02/09/12)	17.5	0.21	0.36	343.4	0.44	0.37	40.5	19.2	0.05
14 (02/26/12)	17.5	0.12	0.42	159	0.44	0.36	33	9	–
15 (03/15/12)	17.5	0.02	–	83	0.43	0.36	41.4	35	–
16 (04/01/12)	17.5	0.08	0.35	156	0.44	0.37	6.8	2.3	–
17 (04/19/12)	17.5	0.04	0.35	64	0.44	0.37	3.8	1.5	0.10
18 (05/06/12)	17.5	53.5	0.26	687	0.44	0.31	536	165	0.01
19 (05/24/12)	17.5	134	0.22	2335	0.44	0.32	407	155	–
20 (06/10/12)	17.5	74	0.27	1971	0.43	0.32	185	106	–
21 (06/27/12)	17.5	2.6	0.29	381.1	0.44	0.33	33	9.8	0.05

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Table 3. Proxy derived temperatures at Station 1 at the northern part of Iceland basin from the total-weighted flux of sedimenting particles, surface sediments and satellite data from AVHRR (NOAA) and WOA09 (Locarnini et al., 2010).

Derived-temperature (°C)	U ₃₇ ^{K'}	TEX ₈₆	TEX ₈₆ ^L	TEX ₈₆ ^L 0–200 m	LDI	Satellite
Total-weighted flux of sedimenting particles	7.1	8.5	14.5	11.7	–2.7	
Annual mean of sedimenting particles	9.4	8.5	15.3	12.3	–1.2	
Surface sediment	10.7	9.2	14	11.4	–1.3	
SPM Jul 2011 5 m	10.8	13.4	11.1	–	–	
SMP Jul 2012 20 m	12.4	14.4	8.8	–	3.4	
AVHRR (NOAA) Jul 2011 5 m						11.3
AVHRR (NOAA) Jul 2012 20 m						12.9
WOA09 annual mean 0 m						9.4
WOA09 annual mean 0–200 m						8.7

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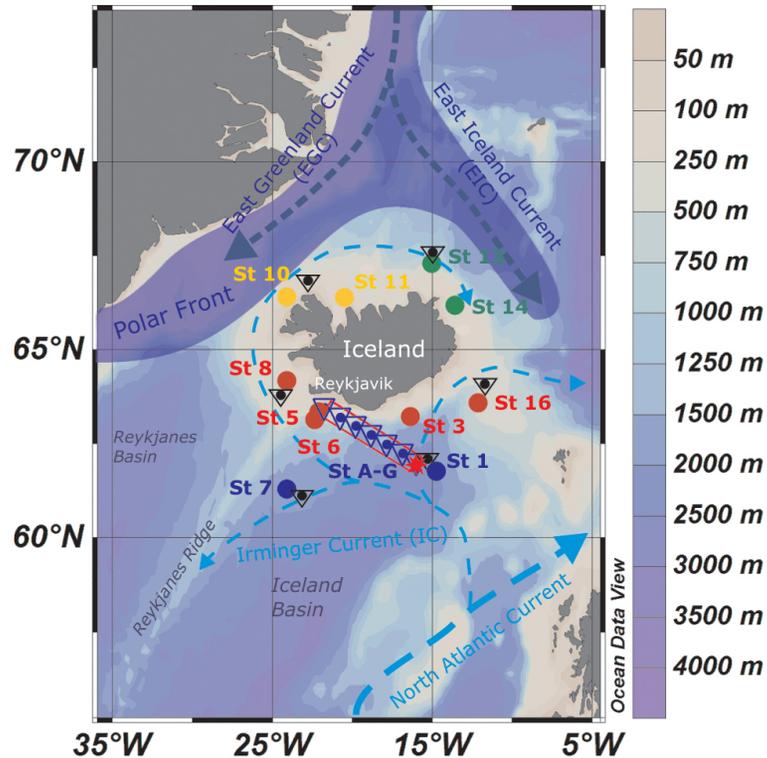


Figure 1. Bathymetric map of the study area with the location of the different sampling stations around Iceland. Filled circles indicates surface sediment stations with different color according with their area of location, i.e. yellow and green circles for the northern stations, red circles for the shallowest stations in the south of Iceland and blue circles for the deepest and southern stations. Black and blue inverse triangles with filled circles indicate surface particulate matter stations around Iceland during July 2011 and transect from Iceland Basin to Reykjavik during July 2012, respectively, and the red star indicates the location of sediment trap deployment. Dashed blue lines show the theoretical circulation of the different water masses.

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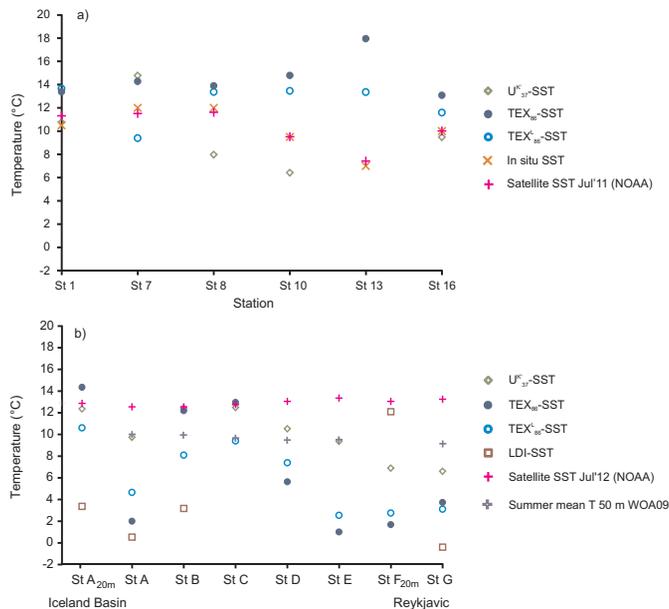


Figure 2. U_{37}^K -, TEX_{86}^- -, TEX_{86}^L - and LDI-derived temperatures from SPM obtained at the indicated sampling stations. **(a)** SPM collected at ca. 5 m water depth during July 2011 at various stations around Iceland; **(b)** SPM collected at 50 m water depth except where noted collected on a transect from the site of the sediment trap deployment to Reykjavik during July 2012. Open green diamonds indicate U_{37}^K -derived temperatures; filled dark blue circles indicate TEX_{86}^- -derived temperatures using calibration by Kim et al. (2010); open blue circles indicate TEX_{86}^L -derived temperatures using calibration by Kim et al. (2010); open brown squares indicate LDI-derived temperatures when the abundance of long chain diols were detectable. Orange symbols indicate in situ SST measured at the CTD, pink symbols indicate satellite SST from NOAA remote sensing records at the time period of collection of the samples, i.e. July 2011 and July 2012, and purple symbols indicate summer mean temperatures at 50 m water depth from WOA09 database (Locarnini et al., 2010).

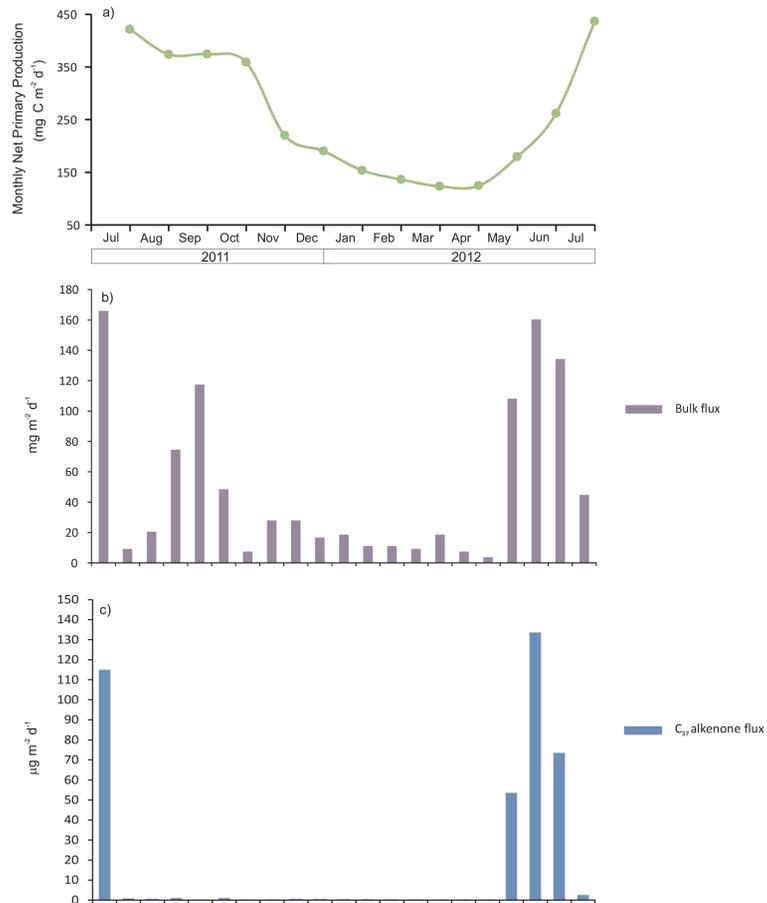


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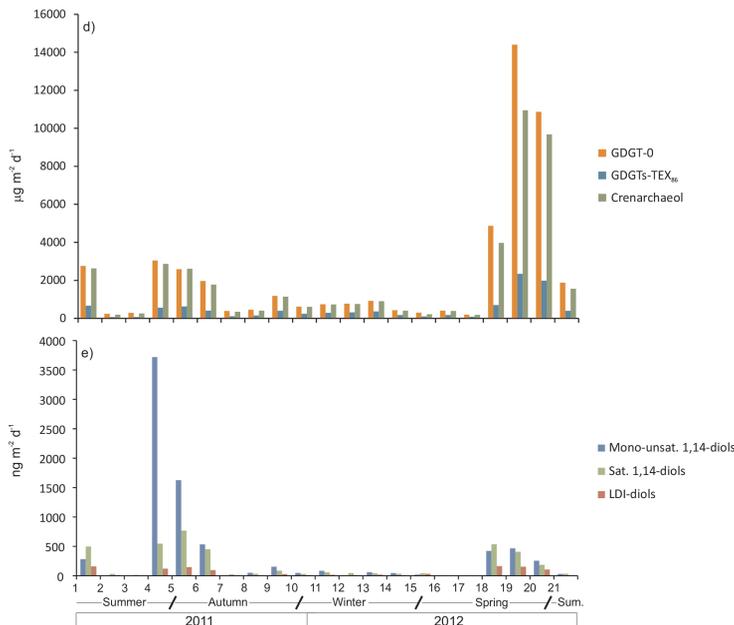


Figure 3. (a) Variations in the net primary productivity from July 2011 to July 2012 derived from Ocean Color Web (Behrenfeld and Falkowski, 1997a). Bar plots of (b) annual variation of total bulk flux, and fluxes of (c) C_{37} alkenones, (d) isoprenoid GDGTs, and (e) long chain diols as determined from the sediment trap data. Numbers refer to sampling intervals specified in Table 2.

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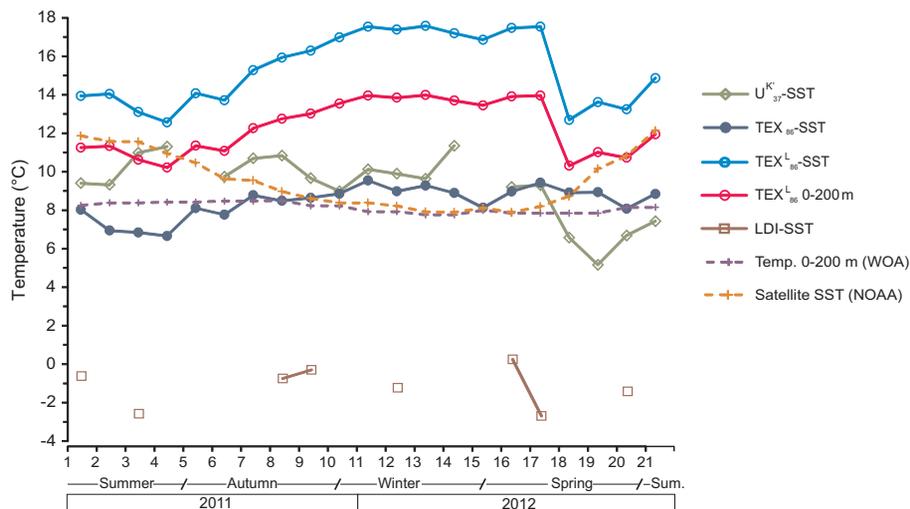


Figure 4. Changes in $U_{37}^{K'}$ - (green line and open diamonds), TEX_{86}^{SST} - (dark blue line and filled circles), TEX_{86}^{L} - (light blue line and open circles), TEX_{86}^{L} 0–200 m (light red line and open circles), and LDI- (brown line and open squares) derived temperatures in descending particles over one complete annual cycle, from July 2011 to July 2012, as measured by the sediment trap experiment. Numbers refer to sampling intervals specified in Table 2, and data points represent the center of collection intervals. Satellite SSTs from AVHRR (NOAA) at St 1 during the sampling period are indicated with a dashed orange line and satellite-derived mean temperatures integrated from 0 to 200 m from WOA09 are indicated with a dashed purple line.

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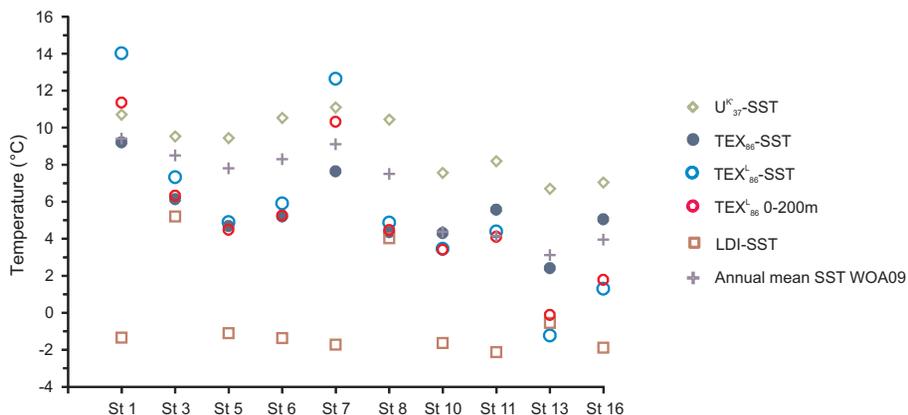


Figure 5. U₃₇^{K'}- (open green diamonds), TEX₈₆- (dark blue circles), TEX₈₆^L- (open light blue circles), TEX₈₆^L 0–200 m (open red circles) and LDI- (open brown squares) derived temperatures in surface sediments from stations around Iceland. Annual mean SSTs at each station derived from the WOA09 database are indicated as purple crosses.

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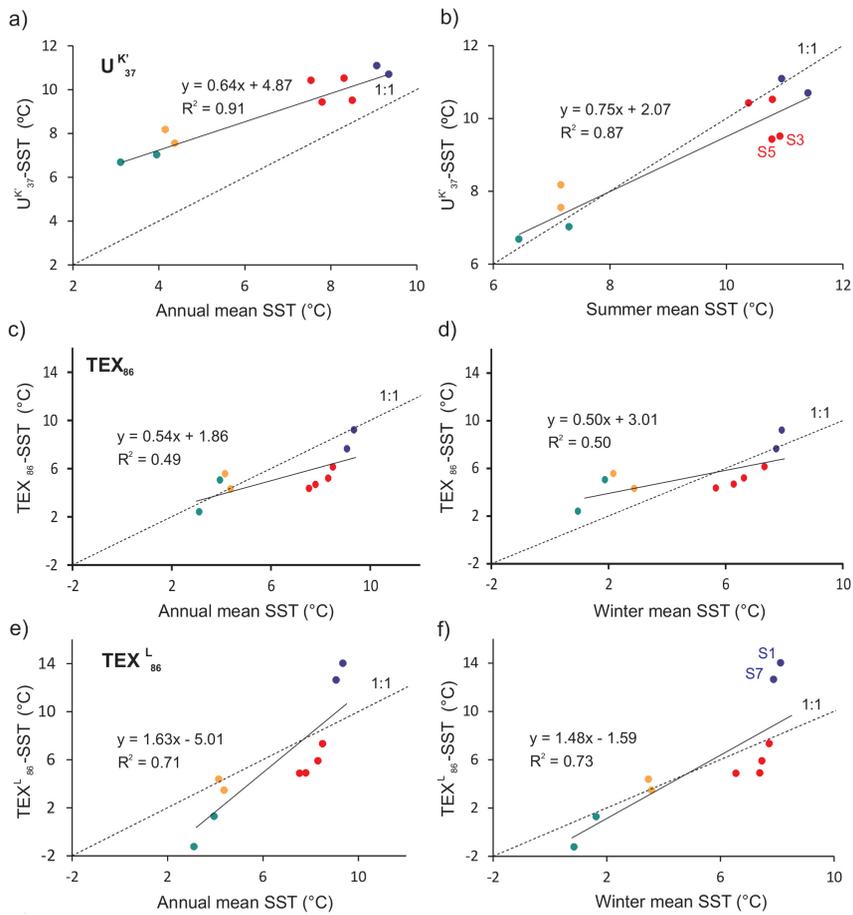


Figure 6.

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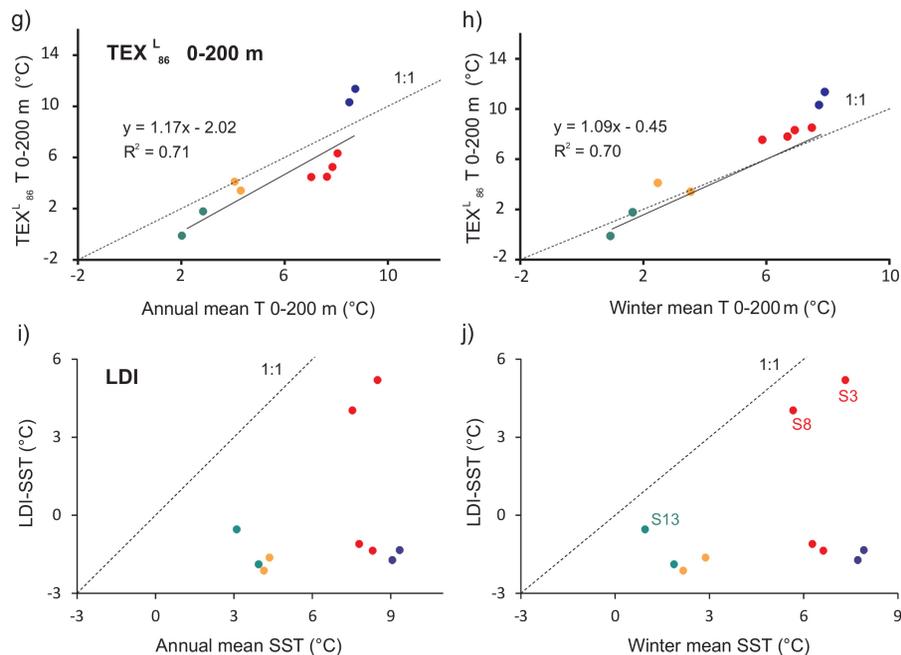


Figure 6. Cross-plots of surface sediment proxy-derived temperatures (U_{37}^K , TEX_{86}^L , TEX_{86}^L , TEX_{86}^L 0–200 m and LDI) with annual and seasonal mean temperature (only showed the best seasonal correlation) from the WOA09 database. Regression lines are represented as black lines, and diagonal black dashed lines show 1 : 1 correlation. Different color codes indicate different station locations according to Fig. 1.