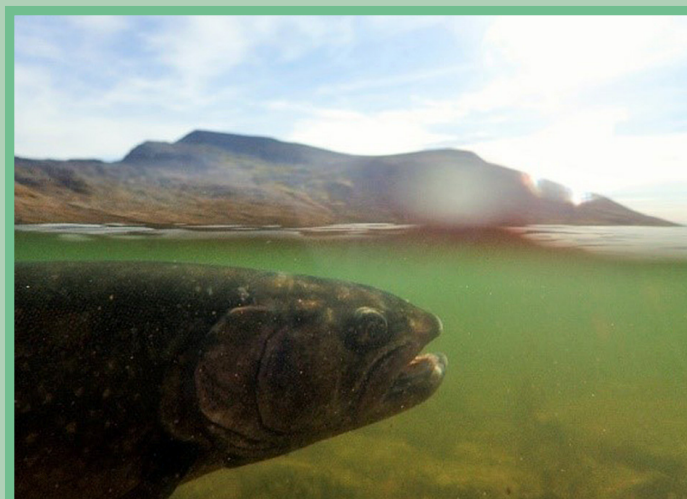


Magali Lucia, Geir W. Gabrielsen, Dorte Herzke, Guttorm Christensen

Screening of UV chemicals, bisphenols and siloxanes in the Arctic





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1. Preface

The purpose of this project is to investigate the incidence of UV chemicals, bisphenols and fluorinated siloxanes in biological samples from the Arctic. The goal was therefore to identify concentrations of substances in top predators of the Arctic.

To that end, eggs from kittiwakes (*Rissa tridactyla*) and glaucous gulls (*Larus hyperboreus*), as well as Arctic char (*Salvelinus alpinus*) were sampled in Svalbard.

This project involved the collaboration between the Norwegian Polar Institute (NPI), Akvaplan-Niva and the Norwegian Institute for Air Research (NILU). Financial support was provided by the Norwegian Environment Agency (Miljødirektoratet).

2. Summary

The Arctic Region is currently undergoing dramatic environmental changes. It is predicted that climate change will result in profound modifications of the Arctic marine food web and contaminant pathways in this region through temperature increase, more low-pressure activity, increased river discharges and runoff from glaciers, as well as accelerated rate of sea-ice melting. New anthropogenic chemicals in commercial use constantly appear in the Arctic ecosystem. Amongst them, organic ultra-violet (UV) filters, siloxanes and bisphenols are of high interest due to their high production volumes, likelihood of long-range transport and potential to bioaccumulate in biota. This screening study aimed therefore to assess the occurrence of those compounds in three species in Svalbard, the glaucous gull, kittiwake and Arctic char. Contaminants were measured in eggs of seabirds (n=10) and muscle of fishes (n=10).

The most quantitatively abundant compounds found in seabird eggs were D5, BPA and BPB. Glaucous gulls displayed overall higher concentrations than kittiwakes for these compounds. For the Arctic char, EHMC, BPG, BPP, and BPZ were found at the highest concentrations. In parallel, EHMC and BPZ were found to be linked to the diet of Arctic chars.

This screening study demonstrated the low levels encountered for siloxanes, UV compounds and bisphenols. Nevertheless, some compounds found at higher levels require further investigation and monitoring.

3. Background

The human impact on Arctic ecosystems is complex. Natural and anthropogenic factors interact to affect ecosystems at different levels. Two major anthropogenic drivers identified in the Arctic are climate change and pollutants. The Arctic is subjected to pollution from both local and remote sources, acting as a sink for chemicals that are produced and released in industrialized parts of the world and transported northwards by sea, air and water masses, making it a global problem.

Persistent organic pollutants (POPs) of anthropogenic origin have been found in Arctic species for decades. These 'legacy' POPs have declined in the Arctic following bans and restrictions on uses and emissions in different parts of the world. However, new anthropogenic chemicals in commercial use constantly appear in the Arctic ecosystem. Amongst them, organic ultra-violet (UV) filters, siloxanes and bisphenols were investigated in 2013 and consequently found in the Norwegian environment. Indeed, those compounds were identified in the effluent water from treatment plants and organisms living in lakes and fjords. It is therefore of interest to check if these compounds are found in the Arctic ecosystem.

3.1. Organic UV filters

Organic UV filters absorbing UV light are considered as emerging contaminants due to their high production volume and increasing use, not only in sunscreen but also in many daily use products, such as cosmetics, plastics, and varnish. UV compounds can be loosely divided into two categories: UV filters used in personal care products to protect hair and cutaneous membranes from sun damage, and UV stabilizers used in technical products such as paints and plastics to protect polymers and pigments against photodegradation, and to prevent discoloring. The first reported environmental occurrence of an organic UV filter was over 30 years ago in the Baltic Sea (Ehrhardt et al. 1982) Since then, various studies have reported their presence in aquatic environmental samples (Tarazona et al. 2010; Grabicova et al. 2013). UV filters and stabilizers used in personal care products enter the aquatic environment indirectly via sewage effluent discharges and directly from skin surfaces into receiving waters. Residues have indeed been detected in a variety of places going from tap water, wastewaters and treated sewage sludge (Gago-Ferrero et al. 2011), to continental and coastal waters (Fent et al. 2010a; Tovar-Sánchez et al. 2013). The hydrophobic properties of some UV filters also indicate that they show the potential to bioaccumulate in biota such as fishes, aquatic birds and aquatic macroinvertebrates, and in urine and blood samples from humans as well (Buser et al. 2006; Calafat et al. 2008; Fent et al. 2010b; Zhang et al. 2013).

Although studies on the toxicological impact of UV filters are scarce, their benefits and impacts have come under debate due to suspicion of their endocrine disruptive effects (Krause et al. 2012). Recent studies in mammals and fishes have demonstrated that estrogen, as well as other hormonal targets are affected by UV filters (Ma et al. 2003; Suzuki et al. 2005; Seidlová-Wuttke et al. 2006; Axelstad et al. 2011). However, there is still a lack of knowledge about occurrences of these compounds in the Arctic environment and biota. This study therefore aimed at assessing the presence of nine organic UV filters in Arctic biota (Table 1).

Table 1. UV compounds and siloxanes selected for screening. Names, acronyms and CAS numbers are indicated.

Type	Compound	Acronym	CAS
Benzophenone	Benzophenone-3	BP3	131-57-7
Cinnamate	Ethylhexylmethoxycinnamate	EHMC	5466-77-3
	Octocrylene	OC	6197-30-4
Benzotriazoles	2-(2H-benzotriazol-2-yl)-4,6-bis(1,1-dimethylethyl)phenol	UV-320	3846-71-7
	2-(5-chloro-2H-benzotriazol-2-yl)-6-(1,1-dimethylethyl)-4-methylphenol	UV-326	3896-11-5
	2-(5-Chloro-2H-benzotriazol-2-yl)-4,6-bis(2-methyl-2-propanyl)phenol	UV-327	3864-99-1
	2-(2H-Benzotriazol-2-yl)-4,6-bis(2-methyl-2-butanyl)phenol	UV-328	25973-55-1
	2-(2H-Benzotriazol-2-yl)-4-(2,4,4-trimethyl-2-pentanyl)phenol	UV-329	3147-75-9
Aminobenzoic acid derivative	2-ethylhexyl-4-dimethylaminobenzoate	ODPABA	21245-02-3
Siloxanes	Octamethylcyclotetrasiloxane	D4	556-67-2
	Decamethylcyclopentasiloxane	D5	541-02-6
	Dodecamethylcyclohexasiloxane	D6	540-97-6
	Tris(trimethylsiloxy)phenylsilane	M3T(ph)	2116-84-9

Among them, five orthohydroxy benzotriazole UV stabilizers were analyzed (UV-320, UV-326, UV-327, UV-328, UV-329). Benzotriazoles are heterocyclic compounds with a hydroxyphenyl group attached to the benzotriazole structure. This class of UV stabilizers has a broad range of physico-chemical properties enabling them to absorb or scatter UV light as well as reflect it, making them very useful for UV protection. Benzotriazoles were developed to absorb the full spectrum of light from 280 nm to 400 nm, the ozone layer being efficient at removing radiation below 280 nm (Crawford, 1999).

Benzophenone-3 (BP3), 2-ethylhexyl-4-dimethylaminobenzoate (ODPABA), Ethylhexylmethoxycinnamate (EHMC) and octocrylene (OC) were also determined. Benzophenones have a high stability in UV light and absorb UV light in the UVA and UVB range. As an example, BP3 has been shown to interact with hormone receptors in fathead minnow (*Pimephales promelas*; Kunz and Fent 2006). ODPABA also absorbs UV light but only in the UVB range. Similarly, OC absorbs light in the UVB range, but also short wavelength UVA light. Finally, EHMC is the most commonly used UV filter in sun lotions and is used in over 90% of those available in Europe. This compound is lipophilic. It has been shown to bio-accumulate in biota and along the trophic chain (Fent et al. 2010a).

3.2. Bisphenols

Bisphenols are currently under consideration in international chemicals regulation. Bisphenol A (BPA) in particular exhibits endocrine-disrupting effects that raise concern about its suitability in some consumer products and food containers. Indeed, BPA is a high production volume synthetic monomer used in the production of polycarbonate plastics, epoxy resin linings of canned foods and beverage containers, dental sealants, and thermal receipt paper. The BPA production is expected to exceed 5.4 million tons this year. The United States Environmental Protection Agency (US EPA) reported that over 1 million pounds of BPA are released into the environment each year (Erler and Novak 2010). BPA has consequently been banned from being used in baby bottles by Health Canada, Denmark, and the European Union. It has also been banned by the US Food and Drug Administration (US FDA) in the coating of infant formula packaging.

Bisphenols are synthesized by the condensation of a ketone (such as acetone, hence the suffix A in the name) with two equivalents of phenol. The restrictions for the use of BPA by the polymer industry triggered its replacement with bisphenol S (BPS) in thermal paper and other products. Bisphenol F (BPF) and bisphenol B (BPB) can replace BPA in the production of epoxy resin and polycarbonate. They have been detected in canned foods and soft drinks. In addition to these analogs, bisphenol AF (BPAF) has broad application in the manufacture of phenolic resins or fluoroelastomers. Annual production is assumed to be in the range of 5 to 300 tons in the USA (Yang et al. 2014).

Unfortunately, those new bisphenol compounds could have similar deleterious effects as BPA. Recent studies have indeed demonstrated possible estrogenic activity similar to that of BPA (Rosenmai et al. 2014). Therefore this study aimed at assessing the occurrence of these compounds in Arctic biota (Table 2).

3.3. Siloxanes

Within environmental research, several studies have identified potential new Arctic contaminants through mathematical modelling of the likelihood of long-range transport and bioaccumulation, taking into account the compounds' physical-chemical properties and production volumes (Vorkamp and Rigét 2014). A list of 610 prioritized compounds was previously established (Howard and Muir 2010). Of the combined list, 62% of the compounds were halogenated (62%) and 7.9% were siloxanes, i.e. known to be environmentally stable. Siloxanes are high production volume chemicals used in a wide range of consumer and industrial products, (e.g. personal care products, medical devices). The abbreviation "siloxanes" actually refers to organosilicon compounds including silicon, oxygen and alkanes (Alaee et al. 2013). They are further divided into groups of methylsiloxanes, polydimethylsiloxanes and polyether-methylsiloxanes. Three cyclic volatile methylsiloxanes, octamethylcyclotetrasiloxane (D4 CAS No. 556-67-2), decamethylcyclopentasiloxane (D5 CAS No. 541-02-6), and dodecamethylcyclohexasiloxane (D6 CAS No. 540-97-6), have been found to accumulate in biota (Warner et al. 2010; Kierkegaard et al. 2011; Kierkegaard et al. 2013). They however bioaccumulate to large varying degrees depending on the chemical and organism studied. The European Community Regulation on Registration, Evaluation, Authorization and Restriction of chemicals (REACH) classified D4 and D5 as very bioaccumulative, in contrast to D6. Furthermore, fluorinated siloxanes copolymers have been also used to improve siloxane property.

Data from the Arctic are fundamental to assess environmental levels of those compounds. The aim of this study was therefore to assess the levels encountered in eggs of two seabird species breeding in Svalbard, the black-legged kittiwake (*Rissa tridactyla*) and glaucous gull (*Larus hyperboreus*), as well as in the Arctic char (*Salvelinus alpinus*) from this area (Table 1).

Table 2. Bisphenols selected for screening. Names, acronyms and CAS numbers are indicated.

Compound	Acronym	CAS
Bisphenol A or 4,4'-(1-methylethylidene)bisphenol	BPA	80-05-7
Tetrabromobisphenol A or 2,2',6,6'-tetrabromo-4,4'-isopropylidenediphenol	TBBPA	79-94-7
4,4-bisphenol F or 4,4'-methylenebisphenol	4,4'-BPF	620-92-8
2,2-bisphenol F or 2,2'-methylenebisphenol	2,2'-BPF	2467-02-9
2,4-bisphenol F or 2,4'-methylenebisphenol	2,4'-BPF	2467-03-0
Bisphenol AF or 4,4'-[2,2,2-trifluoro-1-(trifluoromethyl)ethylidene]bisphenol or Hexafluorobisphenol A	BPAF	1478-61-1
Bisphenol BP or 4,4'-(diphenylmethylene)bisphenol	BPBP	1844-01-5
Bisphenol S or 4,4'-sulfonylbisphenol	BPS	80-09-1
4-nonylphenol	NOPHE4	104-40-5
4-octylphenol	OCphe4	1806-26-4
4-tert-octylphenol	TOCPHE4	140-66-9
Bisphenol B or 2,2-Bis(4-hydroxyphenyl)butane	BPB	77-40-7
Bisphenol Z or 1,1-Bis(4-hydroxyphenyl)-cyclohexane	BPZ	843-55-0
Bisphenol AP or 1,1-Bis(4-hydroxyphenyl)-1-phenyl-ethane	BPAP	1571-75-1
Bisphenol E or 1,1-Bis(4-hydroxyphenyl)ethane	BPE	2081-08-5
Bisphenol FL or 9,9-Bis(4-hydroxyphenyl)fluorene	BPFL	3236-71-3
Bisphenol P or 1,4-Bis(2-(4-hydroxyphenyl)-2-propyl)benzene	BPP	2167-51-3
Bisphenol M or 1,3-Bis(2-(4-hydroxyphenyl)-2-propyl)benzene	BPM	13595-25-0
Bisphenol G or 2,2-Bis(4-hydroxy-3-isopropyl-phenyl)propane	BPG	127-54-8
Bisphenol TMC or 1,1-Bis(4-hydroxyphenyl)-3,3,5-trimethylcyclohexane	BPTMC	129188-99-4

1. Materials and methods

1.1. Species and sampling

A total of 10 eggs of black-legged kittiwakes ($n=5$) and glaucous gulls ($n=5$) were collected randomly in 2013 and 2014, respectively. Sampling took place in June of both years in Kongsfjorden, Svalbard. Kongsfjorden is an inlet on the west coast of Spitsbergen, an island of the Svalbard Archipelago. The inlet is 26 km long and ranges in width from 6 to 14 km. Two

glaciers, Kronebreen and Kongsvegen, head the fjord. Eggs were more precisely sampled in Observasjonsholmen, Kapp Guisnez and Krykkjefjellet (Figure 1).

The black-legged kittiwakes is the most numerous species of gulls in the world and the most oceanic in its habits. It has a circumpolar distribution, breeding in the Arctic and boreal zone, throughout much of the Northern hemisphere. In Svalbard, it is a common breeding species. Individuals feed mainly on invertebrates and small fish, including capelin, polar cod, amphipods and euphausiids (Mehlum and Gabrielsen 1993). In contrast, the glaucous gull is a generalist and opportunistic predator that feeds on a variety of fish, molluscs, echinoderms, crustaceans, eggs, chicks and adults of other seabird species, insects, carrion, refuse and offal (Mehlum and Gabrielsen 1993; Anker-Nilssen et al. 2000; Strøm 2006). This species has a circumpolar, high-Arctic breeding distribution. It is one of the largest gulls breeding in the Arctic. Morphometric measures were recorded (Table 3). The eggs collected were wrapped in aluminium foil and stored frozen until laboratory analyses.

A total of 10 Arctic charrs was also sampled in two lakes in Svalbard, the 14th of August 2015 at Erlingvatnet on Spitsbergen ($n=5$), and the 24th of July at Ellasjøen on Bjørnøya ($n=5$). Individuals weighed between 434 and 1222 g and were between 39 and 53 cm long (Table 4). The char is a cold-water fish from the Salmonidae family, native to alpine lakes and arctic and subarctic coastal waters. It has a circumpolar distribution. The char diet varies with the seasons. During late spring and summer, individuals mainly feed on insects found on the water's surface, salmon eggs, snails and other smaller crustaceans found on the bottom of the lake. They can also feed on smaller fish that can go up to a third of the char's size. During the autumn and winter months, the char diet is mainly composed with zooplankton and freshwater shrimps that are suspended in the lake, as well as occasionally smaller fishes. Fishes were frozen until further analyses.

Both lakes (Ellasjøen og Erlingvatnet) are very close to seabird colonies. Ellasjøen, the deepest lake on Bjørnøya with a maximum depth of 34 m, is situated in the southern, mountainous part of the island (Figure 1). The lake has a surface area of 0.72 km² and an estimated retention time of 1.5 years. The catchment area is estimated at 6.1 km² (Evenset et al 2004). Ellasjøen is more exposed to seabirds since several thousand seabirds every day wash themselves in the lake. Indeed, a large breeding colony of little auk (*Alle alle*) is present in the southern part of the catchment area of Ellasjøen. In addition, , approximately 300 000-500 000 seabirds, encompassing kittiwakes and glaucous gulls, use the lake as a resting area (Klemetsen et al.1985; Theisen, 1997). Birds seem to impact the lake as indicated by the growth of green algae along its shoreline. Erlingvatnet, however, is close to much smaller seabird colonies at Nilspynten in Lilliehöökfjorden (Krossfjorden). This colony has only approximately 1000-1500 pairs of seabirds.



Figure 1. Locations of the sampling sites on Spitsbergen and Bjørnøya, Svalbard, Norway.

Table 3. Kittiwakes (Kit) and glaucous gull (GG) length and width (cm). Individuals were sampled in Krykkjefjellet, Observasjonsholmen and Kapp Guisseez on Spitsbergen, Svalbard.

Sample	Location	Length (cm)	Width (cm)
Kit1	Krykkjefjellet	57.7	41.6
Kit2	Observasjonsholmen	56.1	41.0
Kit3	Observasjonsholmen	60.0	42.0
Kit4	Observasjonsholmen	55.8	41.1
Kit5	Observasjonsholmen	57.2	41.6
GG1	Kapp Guisseez	78.4	54.6
GG2	Kapp Guisseez	81.8	54.2
GG3	Kapp Guisseez	77.5	55.3
GG4	Kapp Guisseez	78.7	53.7
GG5	Kapp Guisseez	75.0	52.9

Table 4. Arctic char weight (g) and length (cm). Individuals were sampled in Ellasjøen, Bjørnøya, or Erlingvatnet, Spitsbergen.

Sample	Location	Weight (g)	Length (cm)
Char1	Ellasjøen	434	40
Char2	Ellasjøen	1222	53
Char3	Ellasjøen	441	39
Char4	Ellasjøen	618	43
Char5	Ellasjøen	1074	53
Char6	Erlingvatnet	731	44
Char7	Erlingvatnet	711	45
Char8	Erlingvatnet	1015	48
Char9	Erlingvatnet	680	46
Char10	Erlingvatnet	940	48

1.2. Analysis of siloxanes

Siloxanes (D4: octamethylcyclotetrasiloxane, D5: decamethylcyclopentasiloxane, D6: dodecamethylcyclohexasiloxane, M3T(Ph): tris(trimethylsiloxy)phenylsilane) were analysed by NILU.

- *Extraction*

Eggs and fish muscle samples were extracted with a biphasic cold solvent extraction adapted from a sediment-method by Sparham et al. (2011), extracting 1 g of tissue with 1 mL acetonitrile and 3 mL *n*-hexane. Hexane-extracts were analysed for cVMS on GCMS (Warner et al. 2014) using a concurrent solvent recondensation large volume injection (CSR-LVI) technique (Biedermann et al. 2004; Companioni-Damas et al. 2012).

- *Analysis*

Analysis of siloxanes was performed using gas chromatography with mass spectrometric detection (GC-MS).

NILU has extensive experience with analysis of siloxanes. The greatest risk in the analysis is background contamination, as these chemicals (Decamethylcyclopentasiloxane 'D5') are applied in e.g. skin care products. Using a state-of-the-art cleanroom, NILU may perform trace analysis of these compounds in matrices from pristine environments, such as the Arctic (Krogseth et al. 2013; Warner et al. 2013).

- *Limits of Detection*

Limits of detection (Method detection limits, MDLs) and quantification (limits of quantification, LoQ) were used to evaluate the detection of analytes. The method used to calculate the MDL has been previously reported (Warner et al. 2013). LoQ was calculated as nine times the signal/noise ratio of the GC-MS instrument.

- *Quality assurance and accreditation*

NILU's laboratories are accredited by Norwegian Accreditation for ISO/IEC 17025. NILU is not accredited for the analysis of siloxanes. However, to the extent possible, documentation, preparation, analysis and calculations were performed in accordance with accredited methods. NILU has previously participated in a laboratory intercalibration of siloxanes (McGoldrick et al. 2011) and has also worked closely with the industry.

Samples were analysed in groups with at least one additive standard sample and a blank control. The data from these were used to calculate the uncertainty for each sample group. To ensure repeatability, a random sample from each matrix was selected for duplicate analysis.

1.3. Analysis of UV compounds and biocides

The organic UV-filters benzophenone-3 (BP3), ethylhexylmethoxycinnamate (EHMC), octocrylene (OC), 2-(3,5-di-*t*-butyl-2-hydroxyphenyl)benzotriazole (UV-320), 2-(5-chloro-2H-benzotriazol-2-yl)-4,6-bis(2-methyl-2-propanyl)phenol (UV-327), 2-(2H-benzotriazol-2-yl)-4,6-bis(2-methyl-2-butanyl)phenol (UV-328), and 2-(2H-benzotriazol-2-yl)-4-(2,4,4-trimethyl-2-pentanyl)phenol (UV-329) were analyzed at NIVA (Norwegian Institute for Water Research) in Arctic char muscle and seabird eggs.

Chrysene-d₁₂ and benzophenone-d₁₀ was used as internal standards. Muscles and eggs were extracted with iso-hexane/isopropanol (50/50) by ultrasonication for 1 hour. Samples were centrifuged and the solvent decanted. This extraction was repeated and the extracts combined. The iso-hexane fraction was isolated by the addition of 0.5% NaCl and the evaporated to approximately 1 ml before solvent exchange to cyclohexane. Different clean-up methods were used for each matrix in response to differing interferences.

1.4. Analysis of bisphenols

Bisphenols (Table 2) were analysed by NILU.

- *Extraction*

Prior to extraction, the samples were added a mixture of isotope labelled phenols for quantification purposes.

Egg samples were extracted using ultrasonic assisted liquid extraction, cleaned on a Florisil column and with dSPE (C18). Remaining interferences were removed with SPE.

Biological samples were extracted with acetonitrile and water. Separation of the organic fraction including analytes with induced by the addition of salts. Fat was removed by liquid-liquid extraction with hexane and remaining interferences were removed with SPE.

- *Analysis*

All samples were analysed with the use the Agilent 1290 UHPLC coupled to Agilent 6550 HR-QTOF equipped with Agilent Dual Jet Stream electrospray source operating in a negative mode.

Separation of analytes was achieved with the use of Accucore Polar Premium column (2.6µm, 250 x 2.1 mm) with a gradient of water and methanol used as a mobile phase.

The data was processed with Mass Hunter B07.

- *Limits of Detection*

The limits of detection (LoD) and quantification (LoQ) were calculated for each sample, using the accepted standard method; three times the signal/noise ratio (z/n) and 9 times z/n, respectively.

- *Quality assurance and accreditation*

NILU's laboratories are accredited by Norwegian Accreditation for ISO/IEC 17025. NILU is not accredited for the analysis of bisphenols, but as far as possible, the documentation, sample preparation, analysis and calculation procedures were conducted according to the accredited methods.

1.5. Nitrogen and carbon stable isotope analysis

Stable isotopes were determined in eggs and fish muscles. Analyses were performed at the Institute for Energy Technology (IFE), Kjeller, Norway. Cleaned feathers of ivory gulls were chopped using surgical scissors and blood samples were accurately weighed (± 0.001 mg) to a range between 0.1 and 0.4 mg. All samples were placed in tin capsules for carbon and nitrogen

stable isotope analysis and analysed using an elemental analyser coupled to an isotope ratio mass spectrometer. The results are reported in δ unit notation (expressed in per mil relative to standards: Vienna Pee Dee Belemnite for $\delta^{13}\text{C}$ and N_2 in air for $\delta^{15}\text{N}$).

1.6. Data analysis

As normality and homogeneity of variance were not achieved despite $\log_{10}(x+1)$ transformation (Cochran C test), non-parametric analysis of variance (Mann-Whitney U-test) was applied to assess differences in stable isotope values for each parameter (species, location). Moreover, the Spearman test was applied to study correlations between parameters (contaminant concentrations, isotopic values).

2. Results and discussion

2.1. Siloxanes

Cyclic volatile methylsiloxanes are considered as emerging contaminants of concern because of their predicted persistence and bioaccumulative characteristics (Howard and Muir 2010). Studies have also demonstrated the biomagnifying potential of D5 and D6 compounds in two Norwegian lakes (Borgå et al. 2013). In contrast, D4 was below the LoQ in the majority of samples and had lower biomagnification than for D5 and D6. Large concentration variations have been observed between studies in the Arctic and in other parts of the world (Borgå et al. 2013; Kierkegaard et al. 2013; Sanchis et al. 2016). Siloxanes results of this study are presented for each individual in Table 5.

Overall, results are all below the LoQ for D4, D5, D6 and M3T(ph). Only egg samples from glaucous gulls displayed D5 concentrations above the quantification limit. Concentrations were indeed ranged between 3.06 and 40.1 ng g^{-1} . More data are needed to confirm those results and the possible threat for glaucous gulls. In contrast, the overall low concentrations observed in arctic char from the remote Arctic are consistent with other observations for this species in other remote lakes such as Femunden in Norway which receive no anthropogenic (e.g., wastewater) inputs (Kierkegaard et al. 2010; Borgå et al. 2013).

2.2. UV filters

Concentrations of UV filters are presented in Table 5. All the UV compounds were below the LoQ in the three species studied. Only EHMC displayed detectable concentrations in the Arctic char. Concentrations ranged between 5 and 39 ng g^{-1} wet weight (ww). EHMC is one of the most commonly used UV filter present in a large number of personal care product. It is used not only to protect human skin from UV radiation but also as a UV absorber to prevent light-induced product degradation in many personal care products such as lipsticks and lip balms, makeup, perfumes, facial creams, aftershaves, hand creams, face powders and hairsprays (Manová et al. 2013). Though knowledge of its health effects is limited, this compound has been associated with a potential disruption of the hormonal system and genotoxicity (Klammer et al. 2007; Carbone et al. 2010; Nečasová et al. 2016). EHMC is included in the European Union's database of possible endocrine disruptors. Because of its high production volume (>1000 tons per year in the EU; Nečasová et al. 2016) and frequent use, this compound is found in high concentrations in water (Tsui et al. 2014). In the present study, EHMC concentrations appear to be lower than in fish

from other areas (Gago-Ferrero et al. 2015; Langford et al. 2015). However, due to its detection in this remote area of the Arctic, further investigation is needed.

2.3. Bisphenols

Bisphenol concentrations were reported in Table 6. Most of the bisphenol measured were below quantification limits for all species, especially TBBPA, 4,4'-BPF, 2,2'-BPF, BPAF, BPBP, NOPHE4, TOCPHE4, BPAP, BPE, BPFL, BPM, BPTMC, and 2,4'-BPF. Nevertheless, depending on the species, several bisphenol reached detectable levels. BPA concentrations were the highest in seabird eggs and were comprised between <1 and 8.8 ng g⁻¹ ww. In those samples, detectable concentrations were also recorded for BPS, OCPHE4, and BPB.

In the Arctic char, OCPHE4, BPB, BPZ, BPAP, BPP, and BPG reached detectable concentrations. BPG was the bisphenol found at the highest concentrations in this species. The type of bisphenol bioaccumulated appeared to be species-dependent when comparing the Arctic char to the seabird species. Overall concentrations reached appeared to be lower than in fishes from other ecosystems such as the Oslofjord and Lake Mjøsa in Norway (Thomas et al. 2014).

In the present study, the geographical location seems to have very little influence on the bioaccumulation of bisphenols. One exception can however be made for BPZ in the Arctic char. Individuals collected at Erlingvatnet displayed higher concentrations than fishes from Ellasjøen. Bisphenols, especially BPA, tend to have a low volatility and are rapidly degraded in the environment, leading to reduced long-range transport capacity (Cousins et al. 2002). However, plastic burning in urban regions is a significant source of atmospheric BPA (Fu and Kawamura 2010). The concentration of BPA show however a declining trend from the continent to the Arctic. Even if significantly lower, this emission in the atmosphere could partly explain BPA detection in seabird eggs. Overall, these results indicate that bisphenol concentrations are still below other ecosystems, but are nonetheless present in the Arctic and should be looked at in the future.

2.4. Relationship between compound accumulation and the diet

Carbon and nitrogen stable isotopes were analysed in the three species studied (Figure 2). Isotopic values in eggs of glaucous gulls and kittiwakes were in the same range, whereas those of Arctic char collected at Erlingvatnet (Spitsbergen) and Ellasjøen (Bjørnøya) showed significant discrepancies ($\delta^{13}\text{C}$: $p = 0.01$; $\delta^{15}\text{N}$: $p = 0.01$, Mann-Whitney test).

For the Arctic char, carbon values were negatively correlated with EHMC concentrations ($r = -0.72$; Spearman test, $p < 0.05$, Figure 3). Individuals collected at Erlingvatnet and with higher $\delta^{13}\text{C}$ values had a tendency of lower EHMC concentrations. This result demonstrates that higher EHMC concentrations are linked to lower carbon values. This could potentially highlight a terrestrial origin for this compound. More samples are therefore needed in the future to confirm this result. BPZ was correlated with both $\delta^{13}\text{C}$ ($r = 0.64$) and $\delta^{15}\text{N}$ ($r = -0.72$). No correlations were found between nitrogen values and contaminant concentrations. Finally, no correlations were found between contaminant concentrations and isotopic value for seabirds.

Table 5. Concentrations (ng g⁻¹ wet weight) of siloxanes (D4, D5, D6, M3T(ph)) and other compounds encompassing BP3, EHMC, OC, UV compounds (UV-320, -326, -327, -328, -329), ODPABA analysed in kittiwake (Kit, *n* =5) and glaucous gull (GG, *n*=5) egg homogenate samples, as well as Arctic char muscles (*n*=10). Sampling locations are indicated for each sample.

Sample	Location	BP3	EHMC	OC	UV-					ODPABA	D4	D5	D6	M3T(ph)
					320	326	327	328	329					
Char1		<3	31.7	<2	<1	<3	<1	<1	<2	<3	<3.11	<2.2	<10.4	<0.15
Char2		<2	30.7	<2	<1	<3	<1	<1	<1	<5	<3.11	<2.2	<10.4	<0.15
Char3	Ellasjøen	<5	11.6	<2	<1	<3	<1	<1	<1	<5	<3.11	<2.2	<10.4	<0.15
Char4		<3	39.3	<3	<1	<3	<1	<1	<1	<6	<3.11	<2.2	<10.4	<0.15
Char5		<3	8.0	<3	<1	<3	<1	<1	<1	<5	<3.11	<2.2	<10.4	<0.15
Char6		<3	12.4	<2	<1	<3	<1	<1	<1	<5	<3.11	<2.2	<10.4	<0.15
Char7		<3	8.9	<2	<1	<3	<1	<1	<1	<5	<3.11	<2.2	<10.4	<0.15
Char8	Erlingvatnet	<3	11.2	<2	<1	<3	<1	<1	<1	<5	<3.11	<2.2	<10.4	<0.15
Char9		<3	5.0	<2	<1	<3	<1	<1	<1	<5	<3.11	<2.2	<10.4	<0.15
Char10		<5	13.7	<4	<1	<3	<2	<2	<1	<5	<3.11	<2.2	<10.4	<0.15
Kit1	Krykkjefjellet	*	<20	<17	<2	<3	<1	<1	<2	<5	<2.78	<2.38	<8.01	<0.23
Kit2		*	<20	<17	<2	<3	<2	<2	<2	<10	<2.78	<2.38	<8.01	<0.23
Kit3		*	<20	<17	<2	<3	<1	<1	<2	<5	<2.78	<2.38	<8.01	<0.23
Kit4	Observasjonsholmen	*	<20	<17	<2	<3	<1	<1	<2	<5	<2.78	<2.38	<8.01	<0.23
Kit5		*	<20	<17	<2	<3	<1	<1	<2	<5	<2.78	<2.38	<8.01	<0.23
GG1		*	<20	<17	<2	<3	<1	<1	<2	<5	5.77	3.37*	<8.01	<0.23
GG2		*	<20	<17	<2	<3	<1	<1	<2	<5	4.19*	11.6	<8.01	<0.23
GG3	Kapp Guisseez	*	<20	<17	<2	<3	<2	<2	<2	<5	3.54*	3.84*	<8.01	<0.23
GG4		*	<20	<17	<2	<3	<2	<2	<2	<5	<2.78	3.06*	<8.01	<0.23
GG5		*	<20	<17	<2	<3	<2	<2	<2	<5	<2.78	40.1	<8.01	<0.23

* denotes interferences

Table 6. Concentrations (ng g⁻¹ wet weight) of bisphenols analysed in kittiwake (Kit, *n* =5) and glaucous gull (GG, *n*=5) egg homogenate samples, as well as Arctic char muscles (*n*=10). Sampling locations are indicated for each sample.

Sample	Location	BPA	TBBPA	4,4'-BPF	2,2'-BPF	BPAF	BPBP	BPS	NOPHE4	OCPE4	TOCPHE4
Char1		1.0	<1	<1	<1	<1.1	<3	<0.3	<1	2.8	<1.5
Char2		<2	<2	<1	<1	<1.1	<3	<0.3	<1	1.3	<1.5
Char3	Ellasjøen	<2	<2	<1	<1	<1.1	<3	<0.3	<1	1.4	<1.5
Char4		<2	<2	<1	<1	<1.1	<3	<0.3	82	2.3	<1.5
Char5		<2	<2	<1	<1	<1.1	<3	<0.3	<1	8.6	<1.5
Char6		<2	<2	<1	<1	<1.1	<3	<0.3	<1	5.9	<1.5
Char7		<2	<2	<1	<1	<1.1	<3	<0.3	<1	<2	<1.5
Char8	Erlingvatnet	<2	<2	<1	<1	<1.1	<3	1.3	<1	<2	<1.5
Char9		<2	<2	<1	<1	<1.1	<3	<0.3	<1	<2	<1.5
Char10		<2	<2	<1	<1	<1.1	<3	0.7	<1	<2	<1.5
Kit1	Krykkjefjellet	<1	1.0	<1	1.3	<1.1	<3	0.4	4.1	2.1	<1.5
Kit2		8.0	<1	7.4	<1	3.1	<3	1.0	<1	<2	<1.5
Kit3	Observasjonsholmen	<1	<1	<1	1.3	<1.1	<3	<0.3	<1	15.5	<1.5
Kit4		8.8	<1	7.5	<1	<1.1	<3	1.1	<1	<2	<1.5
Kit5		3.7	<1	<1	1.3	<1.1	<3	0.5	<1	4.8	<1.5
GG1		8.2	<1	<1	<1	3.1	<3	1.1	<1	<2	<1.5
GG2		7.0	<1	<1	<1	2.0	<3	1.0	<1	19	<1.5
GG3	Kapp Guissez	6.3	<1	6.0	<1	<1.1	<3	0.5	<1	6.0	<1.5
GG4		7.1	<1	<1	<1	<1.1	<3	0.9	<1	3.8	<1.5
GG5		2.0	<1	2.0	<1	<1.1	<3	<0.3	<1	<2	<1.5

Table 6. Concentrations (ng g⁻¹ wet weight) of bisphenols analysed in kittiwake (Kit, *n* =5) and glaucous gull (GG, *n*=5) egg homogenate samples, as well as Arctic char muscles (*n*=10). Sampling locations are indicated for each sample.

Sample	Location	BPB	BPZ	BPAP	BPE	BPFL	BPP	BPM	BPG	BPTMC	2,4'-BPF
Char1		<3	<3	<1.5	<1.5	<2	<2	<3	<2	<2	<1.5
Char2		8.8	23	1.5	<1.5	<2	2.4	<3	17	<2	<1.5
Char3	Ellasjøen	6.0	<3	<1.5	<1.5	<2	10	<3	84	<2	<1.5
Char4		9.5	<3	4.8	<1.5	<2	7.8	<3	94	<2	<1.5
Char5		8.3	78	5.1	12	<2	19	<3	61	<2	<1.5
Char6		<3	142	<1.5	<1.5	<2	20	<3	29	<2	<1.5
Char7		6.9	96	<1.5	<1.5	<2	<2	<3	35	6.3	<1.5
Char8	Erlingvatnet	<3	32	2.0	<1.5	<2	14	<3	49	<2	<1.5
Char9		<3	114	8.9	<1.5	<2	<2	<3	42	<2	<1.5
Char10		14	98	5.2	<1.5	<2	33	<3	73	4.3	<1.5
Kit1	Krykkjefjellet	<3	<3	<1.5	<1.5	<2	<2	<3	15	<2	<1.5
Kit2		2.0	<3	<1.5	<1.5	1.3	<2	<3	<2	<2	0
Kit3	Observasjonsholmen	<3	<3	<1.5	<1.5	<2	<2	<3	<2	3.2	5.4
Kit4		11	<3	<1.5	<1.5	<2	<2	<3	<2	<2	<1.5
Kit5		<3	6.3	<1.5	<1.5	<2	<2	<3	<2	3.5	<1.5
GG1		6.0	<3	<1.5	<1.5	2.4	<2	<3	<2	<2	<1.5
GG2		2.0	<3	<1.5	<1.5	<2	<2	<3	<2	3.0	<1.5
GG3	Kapp Guissez	8.0	<3	<1.5	<1.5	<2	<2	<3	<2	<2	<1.5
GG4		11	<3	<1.5	<1.5	<2	<2	<3	<2	5.0	<1.5
GG5		<3	<3	<1.5	<1.5	<2	<2	<3	<2	<2	<1.5

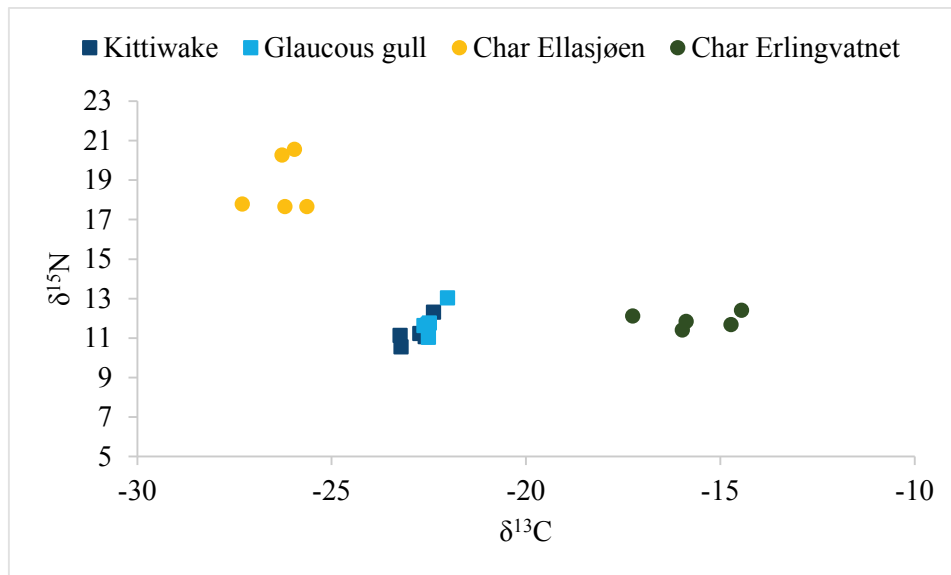


Figure 2. Carbon ($\delta^{13}\text{C}$) and nitrogen ($\delta^{15}\text{N}$) isotopic values (‰) in eggs of glaucous gulls and kittiwakes from Spitsbergen, as well as muscles of Arctic chars from Erlingvatnet on Spitsbergen and Ellasjøen on Bjørnøya.

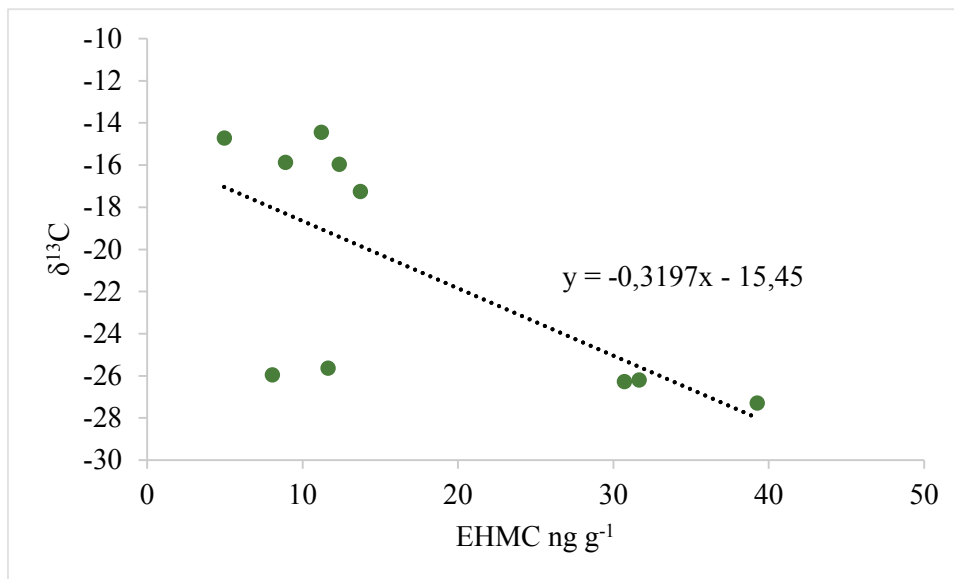


Figure 3. $\delta^{13}\text{C}$ values versus EHMC concentrations (ng g⁻¹ wet wt) in Arctic chars. The equation of the trend curve was indicated.

3. Conclusion

The most quantitatively abundant compounds found in seabird eggs were D5, BPA and BPB. Glaucous gulls displayed overall higher concentrations than kittiwakes for these compounds. For the Arctic char, EHMC, BPG, BPP, and BPZ were found at the highest concentrations. In parallel, EHMC and BPZ were found to be linked to the diet of Arctic chars.

This screening study demonstrated the low levels encountered for siloxanes, UV compounds and bisphenols. Nevertheless, some compounds found at higher levels require further investigation and monitoring.

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