Chemosphere 211 (2018) 1193–1202

Contents lists available at ScienceDirect

Chemosphere

journal homepage: www.elsevier.com/locate/chemosphere

New brominated flame retardants and dechlorane plus in the Arctic: Local sources and bioaccumulation potential in marine benthos

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HIGHLIGHTS

- First investigation of nBFRs and dechlorane plus in Arctic waters and amphipods.
- Potential for bioaccumulation of nBFRs and DDC-COs in amphipods in the local environment.
- Show the importance of thorough elucidation of local sources in Arctic.
- Local contaminant sources need to be distinguished from long-range transport sources.

GRAPHICAL ABSTRACT

ARTICLE INFO

Article history:
Received 28 March 2018
Received in revised form
25 July 2018
Accepted 26 July 2018
Available online 30 July 2018

Handling Editor: Myrto Petreas

Keywords:
Passive sampling
PBDE
Arctic
Benthic amphipods
Atmospheric
Water

ABSTRACT

The aim of the present study was to investigate the presence and bioaccumulation of new flame retardants (nBFRs), polybrominated diphenyl ethers (PBDEs) and dechlorane plus (DDC-CO) in the marine environment close to an Arctic community. Passive sampling of air and water and grab sampling of sediment and amphipods was used to obtain samples to study long-range transport versus local contributions for regulated and emerging flame retardants in Longyearbyen, Svalbard. BDE-47 and -99, α- and β-tetrabromoethylcyclohexane (DBE-DBCH), *syn*- and *anti*-dechlorane plus (DDC-CO) were detected in all investigated matrices and the DDC-COss at higher concentrations in the air than reported from other remote Arctic areas. Water concentrations of ΣDDC-COSs were low (3 pg/L) and comparable to recent Arctic studies. ΣnBFR was 37 pg/L in the water samples while ΣPBDE was 3 pg/L. In biota, ΣDDC-COSs dominated (218 pg/g ww) followed by ΣnBFR (95 pg/g ww) and ΣPBDEs (45 pg/g ww). When compared with other areas and their relative distribution patterns, contributions from local sources of the analysed compounds cannot be ruled out. This should be taken into account when assessing long-range transport of nBFRs and DDC-COs to the Arctic. High concentrations of PBDEs in the sediment indicate that they might originate from a small, local source, while the results for some of the more volatile compounds such as hexabromobenzene (HBBz) suggest long-range transport to be more

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https://doi.org/10.1016/j.chemosphere.2018.07.158

important than local sources. We recommend that local sources of flame retardants in remote areas receive more attention in the future.

1. Introduction

Brominated flame retardants (BFRs) have been extensively used during the last decades (AMAP, 2017). The polybrominated diphenyl ethers (PBDEs) have been shown to bioaccumulate, be toxic and undergo long-range transport in the environment (de Wit et al., 2006, 2010). As a consequence, three commercial mixtures (c-penta-, c-octa-, and c-deca-BDE) and the congener groups included (tetra-, penta-, hexa-, hepta- and deca-BDE) have been subject to regulatory measures and added to the list of persistent organic pollutant (POPs) under the Stockholm Convention and the Aarhus protocol of the Convention on Long-range Transboundary Air Pollution (CLRTAP) (UNECE, 2018). Parties of the conventions must take measures to eliminate the production and use of the PBDEs (Stockholm Convention, 2015). However, PBDEs are still present in the environment, and still pose a possible environmental threat. After regulation of the PBDEs, the industry has introduced alternative BFRs as replacements. These new BFRs (nBFRs) comprise a wide range of individual compounds and the list is continuously growing. Examples of nBFRs are listed in the supplementary information (Table A1, A3) while structure and properties are covered by Bergman et al. (2012). Several chlorinated flame retardants such as *syn*- and *anti*- dechlorane plus (*syn*-DDC-CO and *anti*-DDC-CO) are also produced. Analytical methods are under development and as a result, halogenated FRs are being analysed and detected in the environment at sites all over the world, including the Arctic (AMAP, 2017).

Among the regulatory criteria for the Stockholm convention (persistent, bioaccumulative and toxic), the "persistent" criteria is considered to be fulfilled if a compound reaches the Arctic. The target compounds of the present study have been found in air, glacier and biota from high trophic levels within the Arctic (AMAP, 2017; de Wit et al., 2010; Hermanson et al., 2010; Verreault et al., 2007; Vorkamp et al., 2015; Vorkamp and Rigét, 2014). Examples of nBFRs found in biota at higher trophic levels are 2,3-dibromopropyl- 2,4,6-tribromophenyl-ether (TBP-DPBE, former DPTE) in hooded seals *(Cystophora cristata)* from the Barents Sea and decabromodiphenylethane (DBDPE) in various biota from Svalbard: Brünnich's guillemot *(Uria lomvia)* eggs, polar cod *(Boreogadus saida)* and polar bear *(Ursus maritimus)* plasma (de Wit et al., 2010; Harju et al., 2013; Sagerup et al., 2010; von der Recke and Vetter, 2007). de Wit et al. (2010) concluded that 1,2-bis(2,4,6-tribromophenoxy)ethane (BTBPE) can undergo long-range transport and that it bioaccumulates in the Arctic food web. Vorkamp and Rigét (2014) concluded that DDC-COs can undergo biomagnification while 2-ethylhexyl-2,3,4,5-tetrabromobenzoate (EH-TBB) and BTBPE might biomagnify. There are several other nBFRs available on the market and little is known about the bioaccumulation potential, toxicity and persistency of these compounds. These features need to be investigated for the nBFRs in order to provide a sound management of these compounds, especially with regards to the vulnerable Arctic environment (AMAP, 2017). There is to date a lack of information regarding uptake and concentrations at low and medium trophic levels in the marine Arctic food web, although some information for higher trophic levels and air exist from Greenland (AMAP, 2017; Vorkamp et al., 2015; Vorkamp and Rigét, 2014). Furthermore, the exposure and uptake links between abiotic matrices (e.g. water, sediment, air) and biota are not well understood, and studies are needed to fully understand the fate and environmental behavior of nBFRs.

BFRs are used in a large suite of materials such as textiles, plastic and electronic equipment. In 2013, 280 tonnes of BFRs were used in Norway alone (Norwegian Environment Agency, 2017). With increased human activity and larger, modern settlements in the Arctic, the use of flame retarded goods will most likely increase in these settlements. Hence, even if some of the nBFRs might not undergo long-range transport, they could still be present in the Arctic due to local sources. It is important to understand the impact of local sources on the nearby environment as well as knowing the concentrations and distribution patterns in the environment to disentangle local sources from long-range transport. There are examples where pollution at Arctic sites mainly comes from local sources and not from long-range transport, such as PFAS from airports in remote parts of Canada, PCB from abandoned settlements and siloxanes from local sewage outlets in Svalbard (Pedersen et al., 2011; Stock et al., 2007; Warner et al., 2010).

As pointed out in the new AMAP assessment on contaminants of emerging concern in the Arctic, there is a need for more research and information on the distribution, sources and pathways of these chemicals (AMAP, 2017). The present study aims to fill some of these knowledge gaps and investigate whether an Arctic community with about 2000 inhabitants impacts the concentrations of new and regulated flame retardants in local air and its marine surroundings. This study is also a pilot study on the accumulation of BFRs and DDC-COs in marine, relatively long-lived amphipods in comparison to their environmental surroundings.

2. Materials and methods

2.1. Sample collection

Four matrices were sampled in this study: sediment, biota (littoral amphipods), water and air. The BFRs in water and air were sampled using passive samplers while biota and sediment were sampled directly (grab sampling). A total of 24 samples were collected within Longyearbyen during 2015–2017 (78°13′N, 15°38′E, Table 1, Fig. 1) All equipment used for sampling was pre-cleaned with acetone and *n*-hexane to minimise contamination by the sampling equipment. The handling of samples indoors was kept at a minimum to minimise the risk of contamination. All samples were wrapped in aluminium foil and frozen (−20 °C) directly after sampling and kept frozen until analysis.

2.1.1. Air and water

Air (freely hanging ~1 m above surface) and water (surface water, deployed at the bottom at ~1 m depth) samples were collected at station 1 using passive samplers based on silicone rubbers (SR) from Altesil (Altec, UK, sheet thickness 500 μm). Before deployment in the field, the SRs were pre-cleaned with methanol and spiked with a suite of 14 performance reference compounds (PRCs; including perdeuterated biphenyl and 13 PCB congeners that do not occur in technical mixtures (PCB-1, -2, -3, -10, -14, -21, -30, -50, -55, -78, -104, -145 and -204)) according to the

Table 1
Overview of collected samples for flame retardants analyses, stations, number of samples and time period for sampling campaigns. Locations of station numbers are shown in Fig. 1.

Sample type	Number of samples	Time period for sampling	Station number within Longyearbyen
Water	3; 1 in 2015, 2 in 2016	March–May 2015, Feb–May 2016	1
Air	3; 1 in 2015, 2 in 2016	March–May 2015, Feb–May 2016	1
Sediment	10[a]; 8 in 2016, 1 in 2017	Jan 2016, March 2017	1 (n = 8), 3 (n = 1)
Amphipods	9 (pooled individuals)	July–Aug 2016	1 (n = 3), 2 (n = 1), 3 (n = 3), 4 (n = 2)

[a] One bulk sample (station 1) of 2016 samples is also included in the total number of analyses but is not included in the station column.

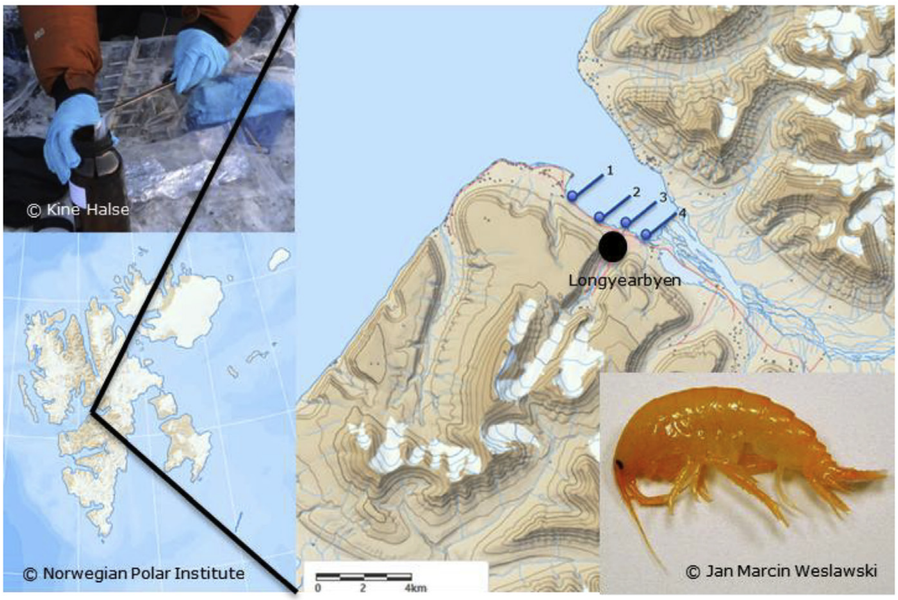

Fig. 1. Map over Svalbard, the settlement of Longyearbyen and sampling sites. The water, air and sediments were sampled at the sampling station 1 and one additional sediment sample was taken from station 3. The amphipods (e.g. *Gammarus setosus*, lower right corner) were sampled from stations 1–4 along the beaches in Longyearbyen. Deployment of the silicone rubbers (SRs) for passive air and water sampling is shown in the upper left corner.

procedure described by Smedes and Booij (2012). The PRCs were partially released from the sampler during exposure and could therefore be used for evaluation sampling rates in the individual samplers. The SRs were deployed for three months in 2015 and 2016 at the small boat harbour in the vicinity of Longyearbyen, Svalbard (Fig. 1, Table 1) Due to waves and currents, the SRs deployed in water (2016) were pushed into shallow water and were exposed to air during parts of the low tide at the time they were collected. After retrieving the SRs, any visible particles were wiped off with clean tissues. One field blank SR for each campaign was exposed to air when the SRs were deployed and when collected. In addition, two un-exposed SRs were kept in the freezer at all time as a reference for the initial PRC concentrations. The average daily temperatures in the air during the sampling period varied between −15 °C and +6 °C (The Norwegian Meteorological Institute, 2017). The surface water temperature of Adventfjorden was in the range of −0.5 °C to +4 °C and is ice-free all year round nowadays (Norwegian Institute for Water Research, 2017).

2.1.2. Sediment

Surface sediments (n = 8, upper 3 cm of sediment, Table 1) were sampled by Van Veen-grab from Adventfjorden, in the vicinity of the small boat harbour (station 1, Fig. 1) in January 2016. One additional sediment sample was collected from station 3 (Fig. 1) in March 2017. The wet sediment was covered with aluminum foil and dried at 30 °C in a drying oven for 3–4 days before sieving (0.5 mm mesh sieve). A bulk sample consisting of a composite mixture of all replicates from station 1 was prepared from dry sediment for additional analyses at a different laboratory.

2.1.3. Biota (amphipods)

Amphipods (n = 9 pooled samples) were collected in the littoral zone of the Adventdalen estuary and from Adventfjorden littoral zone (station 1–4, Fig. 1, Table 1) in July–August 2016. The amphipods hide under stones and can be accessed by turning stones during low tide. Individuals were picked from the sediment surface or from the underside of the turned stones. For practical reasons the amphipods were not allowed empty their gut contents before freezing. The amphipods collected were *Gammarus* spp., especially *G. setosus* (Ambrose and Leinaas, 1990; Weslawski, 1994). It is unlikely that this spatial distribution impacted concentrations or relative distribution to any extent that would be larger than natural variability and the analytical variance. Hence, they were treated as one group for comparison with the sediment, water and air samples.

2.2. Analysis

All samples were analysed for a suite of eight PBDEs, 22 nBFRs, *anti-* and *syn-*DDC-CO, and Dechlorane Plus Mono Adduct (DPMA) at RECETOX, Masaryk university, CZ except for total organic carbon (TOC) in all sediment samples, one sediment sample from 2017 and a bulk sediment sample of the 2016 samples that were analysed at NIVA (Oslo, Norway) instead-. The reason was to compare results between the laboratories. CAS number, log K_{ow}, log K_{oa}, internal standards (IS) and *m/z* for quantification and qualification masses are listed in Tables A1-A3. The passive SR samplers were also analysed for 18 organophosphorous flame retardants (OPFRs). The passive samplers were analysed for OPFRs as well. However, due to little information on K_{pw} in cold temperatures for SRs, these data are not discussed within the paper, but are presented in Table A8 together with the analytical method. All halogenated standards were purchased from Wellington Laboratories, Guelph, Canada (except BDE-118, which was purchased from AccuStandard, New Haven, CT). All glassware was heated at 150 °C for two hours, and all equipment used was cleaned with acetone and *n*-hexane right before use.

TOC was analysed at NIVA (accredited laboratory, ISO 17025). Briefly, the homogenised sediment samples were mixed with hydrochloric acid, flushed with air to remove the inorganic and volatile carbons. Afterwards, the samples were burned and the amount of CO_2 (proportional to the amount of TOC) was measured with infra-red light.

2.2.1. Passive samplers for air and water

All passive samplers were spiked with ^{13}C labelled internal standards (IS) for the BFRs and DDC-COs (Table A2) and three non-labelled PCBs (PCB 4, 29 and 185) that do not occur in technical mixtures before extraction. As recovery and method control, five non-exposed passive samplers were spiked with the target BFRs and DDC-COs and further treated in the same way as the real samples. Two solvent blank samples were also processed. The samples were Soxhlet extracted for eight hours in methanol and thereafter evaporated by Kuderna-Danish apparatus to ~2 mL before solvent exchange to *n*-hexane and further evaporated to ~2 mL.

2.2.2. Sediment

The sediment samples from station 1 (n = 8, 10 g each) were spiked with ^{13}C-labelled IS for PBDEs, nBFRs and DDC-COs (Table A2) before Soxhlet extraction for eight hours in DCM followed by evaporation by Kuderna-Danish to ~2 mL and solvent exchange to *n*-hexane. One composite bulk sample of sediment (station 1, 2016 samples) and one sediment sample from spring 2017 (station 3) were analysed at NIVA for PBDEs as a comparison between the two laboratories. The NIVA method differs slightly from RECETOX' method and is described in the appendix. All reported PBDEs were analysed at both laboratories except for BDE-49, -196, -209 which were analysed only at NIVA (bulk sample and the sediment sample from station 3) and BDE-66 and -85 which were analysed at RECETOX only.

2.2.3. Biota

The amphipods were thawed, and water and sediment particles were removed before homogenisation with a kitchen blender. Each sample (n = 9) contained 3–8 g of amphipods. ^{13}C-labelled IS of PBDEs, nBFRs and DDC-COs (Table A2) were added to each sample before the homogenates were freeze dried for 50 h. Dried samples were Soxhlet extracted for eight hours in dichloromethane (DCM) and thereafter evaporated using Kuderna-Danish apparatus to ~2 mL, solvent exchanged to *n*-hexane and further evaporated to ~2 mL.

2.2.4. Clean-up of all sample matrices

All samples were further cleaned up using silica columns and GPC (for biota and sediment only). Details are provided in the appendix.

2.2.5. GC-MS analyses

2.2.5.1. PBDEs, nBFRs and DDC-COs. Analyses of PBDE, DDC-COs and nBFRs and PRCs were performed using a gas chromatograph connected to a mass spectrometer (GC-MS). Details are provided in the appendix.

2.3. Quality assurance and control

Two blanks made of solvent pre-extracted cotton wool were processed together with the sediment and biota samples. The field blanks and reference SRs were processed together with the air and water SRs. 2-ethylhexyl-2,3,4,5-tetrabromobenzoate (EH-TBB) and bis(2-ethylhexyl)tetrabromophthalate (BEH-TEBP) were not reported for the biota and sediment samples due to their partial destruction during sample clean-up with silica gel with sulfuric acid. A standard reference material (SRM 1944 from National Institute of Standards and Technology (NIST)) was analysed for PBDEs in sediment at NIVA and the median deviation from the reference value for PBDEs were 12%. The limit of detection (LOD) was defined as 3 × S/N (signal to noise-ratio) when a compound was not detected in the blank, otherwise the average blank concentration + 3 × standard deviation in the blanks (and reference SRs for air and water) were used. LOQ for the matrix water was calculated by inserting the amount of a target compound detected in the field blank to the formula for calculation of water concentrations (equation (2)). Detection frequency is listed in Table A4 and the LODs are listed in Table A5. The field blank concentrations are listed in Table A6. The sample results were not blank subtracted. Samples with concentrations < LOD was not included in statistical analyses and all values are presented based on concentrations > LOD together with a note on the frequency of detection.

2.4. Calculations of atmospheric and aqueous concentrations of BFRs

The performance reference compounds (PRCs) indicated the degree of equilibrium reached by the passive sampler for compounds with a range of partition coefficients; K_{pa} (SR/air) or K_{pw} (SR/water). The rate that PRCs are leaking out from the passive sampler is known by laboratory experiments and this rate can be related to accumulation of (similar) compounds into the passive sampler. The concentrations left in the SRs after exposure indicated that the samplers were far from equilibrium with the sampled matrix for most compounds. Hence, the sampling was assumed to be fully integrative for all targeted compounds. In this situation, concentration of compounds in the air can be calculated as in equation (1).

$$C_a = \frac{N_{SR}}{R_S t} \tag{1}$$

Where N_{SR} is the concentration of the analyte in the sampler (pg/sampler) at the exposure time, t is time of exposure (in days), R_s is the sampling rate of the compound (m^3/day) where generic values from Okeme et al. (2016) were used.

Aqueous concentrations, C_w, of individual compounds were calculated from the mass absorbed by the SRs, N_{SR}, the degree of equilibrium *DEQ* (equation (3)) that the compound attained during sampler exposure, the mass of sampler, m_{SR}, and their sampler-water partition coefficients $K_{SR,w}$ as described in Booij et al. (2007). This is shown in equation (2).

$$C_w = \frac{N_{SR}}{K_{SR,w} m_{SR} DEQ} \tag{2}$$

$$DEQ = \left(1 - \exp\left(-\frac{R_{S,SR} t}{K_{SR,w} m_{SR}}\right)\right) \tag{3}$$

The sampling rate $R_{S,SR}$ of individual compounds by SR passive samplers was modeled as a function of molar mass M by the water boundary layer (WBL) controlled uptake model with an exposure-specific parameter B: $R_{S,SR} = B \times M^{-0.47}$ (Rusina et al., 2010). The parameter B was estimated from the dissipation of PRCs from samplers during exposure using nonlinear least squares method (Booij and Smedes, 2010). This method considers the fraction of individual PRCs that is retained in sampler after exposure as a continuous function of their sampler-water partition coefficient $K_{SR,w}$.

2.5. Bioaccumulation calculations

The bioaccumulation factor (BAF; equation (4)) was estimated using the average concentrations measured in the passive water samplers (C_{water}; dissolved water concentrations) and amphipods (C_{biota}; lipid weight normalised concentrations). The lipid content in the amphipods were assumed to be 10% based on species and locality (Nygård et al., 2010; Szaniawska and Wolowicz, 1985). To take the seasonal lipid variability into account, estimations with lower (5%) and higher (15%) lipid content were included. For biota-sediment accumulation (BSAF, equation (5)), the average TOC-normalised sediment concentrations ($C_{sediment}$) were used.

$$Log\ BAF = \mathrm{Log}\left(\frac{C_{biota}}{C_{water}}\right) \tag{4}$$

$$Log\ BSAF = Log\left(\frac{C_{biota}}{C_{sediment}}\right) \tag{5}$$

3. Results and discussion

All samples were collected within the same fjord. Any contribution from long-range transport should therefore impact all samples equally, although the compound distribution and uptake patterns will differ due to the different properties of the matrices. Concentration data are presented in Table 2.

3.1. Distribution patterns in the Longyearbyen samples

3.1.1. Air

Seven halogenated FRs were detected in more than 50% of the passive air samples; BEH-TEBP, EH-TBB, α- and β-DBE-DBCH, PBT, *syn*- and *anti*-DDC-CO. In addition, PBEB were detected in one of the samples (Table 2) The PBDE congeners BDE-47 and -99 were detected in all samples -while BDE-100 was detected in two of the air samples. This is consistent with results from outdoor calibration studies of another type of passive air sampler (polyurethane foam; PUF) (Bohlin et al., 2014). As a comparison on the relative contribution of all halogenated FRs analysed, BEH-TEBP was the predominating compound (55%, 1867 pg/m^3) followed by *syn-/anti*-DDC-CO (20% together, 356 and 349 pg/m^3, respectively, Fig. 2, Table 2). The samples where PBEB (<LOD-0.02 pg/m^3) and PBT (<LOD in one sample, 0.03–0.06 pg/m^3 in the other) were >LOD showed comparable (PBEB) but higher (PBT) concentrations than previously reported from passive sampling at other Arctic sites. The range of PBEB concentrations at other Arctic sites were <LOD-0.11 pg/m^3 in Barrow and St. Laurence Island (Alaska, USA), Stórhöfði (Iceland) and Ny-Ålesund (Svalbard, Norway) while the PBT concentrations were <LOD-0.005 pg/m^3 at the same stations except Iceland where they PBT was not analysed (AMAP, 2017).

3.1.2. Water

The total concentration of halogenated FRs in the passive water samples were 43 pg/L on average. As a comparison on the relative contribution of all halogenated FRs analysed, α-TBCO (48% in 2015 and 76% in 2016) was the predominating compound in the water samples, followed by BEH-TEBP (14–23%). The PBDEs contributed to 15% (campaign in 2015) and 5% (campaign in 2016) of all BFRs/DDC-COss analysed (Fig. 2). The average ΣPBDE concentrations (2.9 pg/L) were slightly higher in the water around Longyearbyen compared to East Greenland Sea, which could be related to the samplers being located close to a village in the present study (Möller et al., 2011). *Syn*- and *anti*-DDC-CO contributed to 13% in 2015 but decreased to 3% contribution in 2016 of the FRs analysed. This was a consequence of slightly lower DDC-CO-concentrations in combination with increased concentrations of α-TBCO in the 2016 water samples. The water samples in 2016 got partially exposed to air due to wind and weather that moved the samplers, but the concentrations measured in 2016 are in the very same range as the results from 2015, and hence, this has probably not affected the concentrations more than the analytical uncertainty.

The individual sediment samples presented here were analysed for all compounds while the bulk sample of these samples was only analysed for PBDEs. The sediment sample from 2017 is shown here but was discussed separately due to the much lower concentrations (all PBDEs were <LOD) found there.

3.1.3. Amphipods

Syn- and *anti*-DDC-CO dominated in the amphipods and contributed on average 61% of the total concentration of the analysed compounds that were above LOD in the samples (Fig. 2). The PBDE concentrations were below LOD in one of the samples from station 1 and in all three samples from station 3, where also the PBDE concentration in sediment was <LOD. For the other samples, BDE-47, -99 and -100 were the predominant congeners and they contributed 5–26% to the sum of the compounds above LOD analysed in the amphipods (Fig. 2). The amphipods were analysed without cleaning the gut and hence, the samples may reflect the contaminant distribution within the sediment from the respective sampling spots. For concentrations and detailed information about the samples, see Table 2 and A4. To our knowledge, nBFRs have not been analysed in Arctic benthic fauna and PBDEs have not been analysed in Arctic littoral amphipods before.

3.1.4. Sediment

PBDEs dominated extensively among the analysed compounds analysed in the sediment samples, followed by *syn*- and *anti*-DDC-CO (Fig. 2). The predominant BDE congeners were BDE-47 and -99, with concentrations (bulk sample) of 1910 and 1840 pg/g dw, respectively. The next highest concentration was found for BDE-196 (1730 pg/g dw in the bulk sample; Table 2). Concentrations in the individual samples from station 1 was in the same concentration range (Table 2).

3.2. Potential for bioaccumulation

The lighter nBFRs with a predicted potential for bioaccumulation within the Arctic (HBBz, PBEB and PBT) were all detected in the amphipods although PBT was the only one of these

Table 2
Average concentrations > LOD measured in all samples. Compounds/samples without numbers were <LOD.

	Air (pg/m³)				Water (pg/L)				Sediment (pg/g dw)				Sediment (ng/g dw)		Amphipods (pg/g ww)			
	Average >LOD	Min	Max	Std dev	Average >LOQ	Min	Max	Std dev	Average (individual samples) >LOD	Min	Max	Std dev	Composite sample station 1, n=1	station 3. n = 1	Average >LOD	Min	Max	Std dev
PBBz	<LOD				1.5	<LOD	1.9	NA	<LOD				NA	NA	3.8	<LOD	5.6	1.3
BTBPE	<LOD					<LOD			<LOD				NA	NA	0.5	<LOD	0.5	0.1
HBBz	<LOD					<LOD			<LOD				NA	NA	3.5	<LOD	6.7	1.6
PBEB	16	<LOD	16			<LOD			2.5	1.2	4.0	1.3	NA	NA	1.1	<LOD	3.5	1.1
pTBX	<LOD					<LOD			0.8	<LOD	0.8		NA	NA	<LOD			
PBT	45	29	61		0.02	<LOD	0.02	NA	<LOD				NA	NA	7.3	<LOD	14	3.2
α-TBCO	<LOD				30	13	55	18	<LOD				NA	NA	<LOD			
BEH-TEBP	1867	459	2588	996	7.1	4.6	10	2.3	NA				NA	NA	NA			
EH-TBB	86	10	164	63		<LOD			NA				NA	NA	NA			
TBP-AE	<LOD					<LOD			<LOD				NA	NA	0.1	<LOD	0.2	0.1
TBP-DBPE	<LOD					<LOD			<LOD				NA	NA	5.5	<LOD	6.8	1.3
syn-DDC-CO	356	65	668	246	1.1	0.3	2.1	0.6	6.2	3.9	8.6	1.6	NA	NA	82	3.8	241	65
anti-DDC-CO	349	72	531	199	1.1	0.5	1.6	0.5	9.6	5.9	17	3.5	NA	NA	130	4.3	329	93
DPMA	<LOD					<LOD			2.1	2.0	2.2	0.1	NA	NA	4.9	<LOD	5.7	0.9
α-DBE-DBCH	48	29	65	15	0.3	0.2	0.5	0.1	1.1	<LOD	1.7	0.5	NA	NA	0.4	0.1	1.4	0.4
β-DBE-DBCH	31	17	46	12		<LOD			1.3	<LOD			NA	NA	0.3	<LOD	0.9	0.2
													Sediment Bulk st. 1	Sediment St. 3				
PBDE28	<LOD					<LOD			34	15	62	15	<LOD	<LOD	<LOD			
PBDE47	234	74	318	113	1.6	0.9	2.5	0.7	4529	2070	6460	1522	1910	<LOD	38.9	<LOD	70	16
PBDE66	<LOD					<LOD			117	34.3	271	81	BDE-49; <LOD	BDE-49; <LOD	<LOD			
PBDE85	<LOD					<LOD			106	<LOD			BDE-126; <LOD	BDE-126; <LOD	<LOD			
PBDE99	295	88	439	150	1.3	0.7	1.7	0.4	5555	2750	11200	2699	1840	<LOD	30	<LOD	47	12
PBDE100	96	<LOD	106			<LOD			1028	404	2120	535	330	<LOD	14	<LOD	14	1
PBDE153	<LOD					<LOD			357	<LOD			<LOD	<LOD	<LOD			
PBDE154	<LOD					<LOD			332	114	930	254	120	<LOD	<LOD			
BDE-183	NA				NA				NA				<LOD	<LOD	NA			
BDE-196	NA				NA				NA				1730	<LOD	NA			
BDE-209	NA				NA				NA				<LOD	<LOD	NA			
ΣPBDEs	593	162	835	306	2.9	1.6	4.1	1.0	11969	5520	21000	5115	5930	<LOD	35	<LOD	132	41

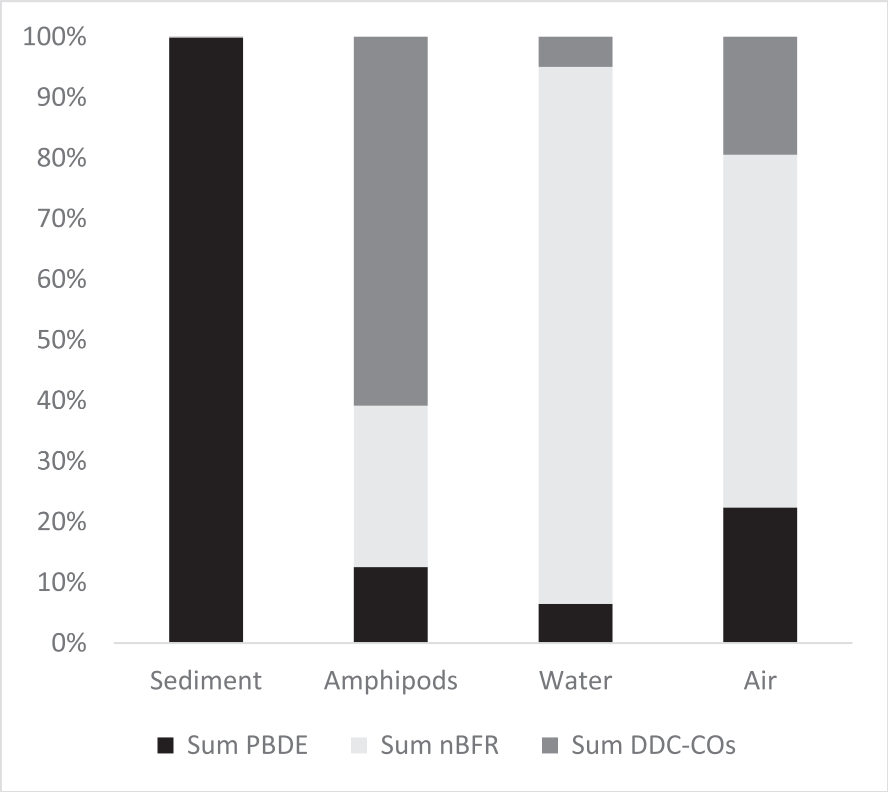

Fig. 2. Relative distribution of ΣPBDE, ΣnBFR and ΣDDC-COs in all samples.

compounds > LOD in the dissolved water phase. PBT showed the highest log BAF value among the compounds analysed in the present study (9.6). For comparison, log BAF was estimated to be 8.3 for BDE-47 and 8.2 for BDE-99 in the present study. This indicates that the uptake/degradation of BDE-47 and -99 at low-medium trophic levels are similar and not impacted to any large extent by e.g. biological degradation. Due to the molecular structure and physical-chemical properties of PBEB and HBBz, there is reason to believe that their BAFs would be cause for concern with regards to bioaccumulation (Harju et al., 2009).

Although *syn*- and *anti*-DDC-CO are larger molecules than BDE-47 and -99, they showed high log BAFs in the present study; 8.9 and 9.1, respectively. There are a few studies of these compounds in the Arctic with different results regarding detected/non-detected concentrations in biota and it is difficult to draw any conclusions on the bioavailability, bioaccumulation and biomagnification, as concluded elsewhere (AMAP, 2017; Vorkamp et al., 2015) The knowledge gaps on uptake and transformation processes need to be further investigated before we can conclude on the bioaccumulation potential of DDC-COs.

Bioaccumulation calculations are sensitive to the sampling season since lipid content of the organisms impact the calculations, and lipid content of Arctic and high-latitude organisms can vary considerably throughout the year (Carlsson et al., 2016; Nygård et al., 2010; Varpe, 2017). Seasonality, including lipid dynamics also impacts bioaccumulation in pelagic food webs (Hallanger et al., 2011). This impact of seasonality was smaller in a benthic food web in Kongsfjorden compared to the pelagic food web, and this feature may also be valid for Adventfjorden (Evenset et al., 2016). The log BSAF and log BAF values in the present study (Table 3) should be evaluated with age, lipid dynamics and seasonality in mind, especially since the transport pathways of nBFRs and DDC-COs are much less known compared to legacy POPs such as PCBs. Log BAF increased with increasing K_{ow} and compounds with similar K_{ow} showed similar log BAF values (Fig. A1).

Table 3
Log BAF and log BSAF calculated from concentration data in amphipods, passive water samplers and sediment in the present study[a]. The table also illustrates the differences in BSAF values between an average of the individual sediment samples and the results from the bulk sample analysis.

% lipids in amphipods:	Log BAF			Log BSAF[a]			Log Kow
	15%	10%	5%	15%	10%	5%	
PBBz	7,2	7,4	7,7				5,4
PBEB				0,8	1,0	1,3	6,8
PBT	9,4	9,6	9,9				
α-DBE-DBCH	7,0	7,1	7,4	0,7	0,9	1,2	5,5
β-DBE-DBCH				0,6	0,8	1,1	5,5
syn-DDC-CO	8,7	8,9	9,2	2,3	2,5	2,8	9,3
anti-DDC-CO	8,9	9,1	9,4	2,3	2,5	2,8	9,3
DPMA				1,6	1,7	2,0	8,0
BDE-47	8,2	8,4	8,7	−0,9	−0,7	−0,4	6,8
BDE-99	8,2	8,4	8,7	−1,1	−0,9	−0,6	7,4
BDE-100				−0,7	−0,5	−0,2	7,1
Composite sediment sample							
BDE-47				−0,5	−0,3	−0,03	6,8
BDE-99				−0,6	−0,4	−0,1	7,4
BDE-100				−0,2	−0,01	0,3	7,1

Only compounds > LOD in sediment/water and amphipods were included.

[a] The TOC content in the sediment samples was 2.3% and the total carbon (TC) was 5.0%.

3.3. Potential local sources

3.3.1. Polybrominated diphenyl ethers (PBDEs)

The concentrations of BDE-47 and -99 in air at Longyearbyen, obtained from the passive air samplers in this study, were two to three orders of magnitude higher than those measured at Zeppelin Observatory, using active high-volume air samplers, during the same time period (March–May 2015, Table A7) (Aas and Bohlin-Nizzetto, 2017). These results suggest that PBDEs in Adventfjorden harbour area might come from local sources and not solely from long-range transport (Ruus et al., 2014). Nevertheless, the PBDEs were not the predominant FRs in the air, water and biota samples, but they did predominate the sediment samples. This feature could be due to the longer historical usage of PBDEs, the affinity of PBDEs for sediment particles, and a high content of coal particles in the sediment. The coal is present due to the geology of the area, but mostly as a consequence of the coal storage along the shoreline close to the sampling area (few 100 m away). However, several of the nBFRs are similar enough in their structure to PBDEs that they also would be associated with the coal particles, but the concentration of $\sum$nBFRs in the sediment was around a factor 200 lower compared to the $\sum$PBDEs in the bulk sediment sample.

The concentrations of the PBDEs in the sediment samples from the present study are two orders of magnitude higher than those at remote areas in Bering Sea, Chukchi Sea and the Canada Basin where the BDE-47 and -99 ranged between $<$ LOD-75.6 pg/g dw (Cai et al., 2012). $\sum$PBDE in lake sediments from earlier studies (sampled 2004–06) on Svalbard were 2–470 times lower than the present study (Table 2), ranging from 25.5 pg/g dw in Kongressvatnet, close to Barentsburg in Grønnfjorden and up to 2383 pg/g dw in Åsövatnet on the north-west part of Spitsbergen (Christensen and Evenset, 2008). Åsövatnet is impacted by bird guano input. However, bird guano is not a likely explanation for explaining the high concentrations in the present study. Instead the close distance between sampling site and a harbour is more likely to be of importance. There is reason to believe that the higher concentrations measured in the present sediment samples are caused by local sources, most likely more influenced by harbour activities and other activities happening on land close to the sampling area. The local sewage outlet was not a source as effluents are released into a different water mass than the one sampled.

Compared to PBDE concentrations in harbour sediment from the 75 000 people city Tromsø in Northern Norway (ΣPBDE: 130 pg/g dw), the sediment sample concentrations from this study were one order of magnitude higher (Fjeld et al., 2004). The concentration and relative distribution pattern of PBDEs in the sediment samples from Longyearbyen were similar to the distribution pattern of those PBDE congeners around Lillehammer (28 000 inhabitants), Lake Mjøsa, where there have been point sources (Fjeld et al., 2004). Only congeners analysed at both sites were considered in this comparison (BDE-28, -47, -99, -100, -153, -154 and -183) and the total PBDE concentrations (excluding BDE-209) in Lake Mjøsa sediments were 14 050–16 480 pg/g dw. BDE-99 contributed to 47% in Longyearbyen sediment and 49–51% in the Lillehammer sediment while BDE-47 had a slightly higher relative contribution in Longyearbyen sediment of 38% compared to 26–28% in Lillehammer (Fjeld et al., 2004). The higher relative contribution of BDE-47 in Longyearbyen might be caused by long-range transport, but the concentration in the present study are too high to be explained only by long-range transport. A recent study of BDE-209 in sediments in Kongsfjorden, Svalbard showed elevated concentrations outside the Ny-Ålesund harbour there compared to stations further out in the fjord (230 and 100 pg/g dw, respectively) (Ma et al., 2015). Ny-Ålesund also receives several cruise ships during the summer time, which could contribute to elevated concentrations of BFRs around harbours on Svalbard. However, earlier studies (Evenset et al., 2009) as well as the one sample from a local beach in the present study did not show high concentrations within Adventfjorden. The concentrations differences between the samples from the local harbour in the present study, and the samples further out in Adventfjorden, are either a result of a very local "hot-spot" sampled in the present study, or dilution/high sedimentation rates in the fjord that causes low PBDE concentrations there. There is reason to believe that the PBDE in the sediment samples in the present study came from a local "hot spot", although it might be difficult to identify the source or to re-localise due to sedimentation in the fjord and harbour area. This is further supported by the results from station 3 (PBDEs $<$ LOD). Taken together, the results in the present study means that it cannot be out-ruled that some of the PBDE in the environment comes from local sources.

3.3.2. Hexabromobenzene (HBBz)

HBBz was detected in two of the sediment and three of the amphipod samples although at low concentrations (Table 2 and A4). It was not detected in the water nor the air samples even though recent studies in the same area showed the presence of HBBz (median 0.12 pg/m^3)in the atmospheric particle phase (Salamova et al., 2014). Samples from Ny-Ålesund (gas phase; <LOD-0.67 pg/m^3) were in the same concentration range (AMAP, 2017; Lee et al., 2016). However, the LODs in that study were lower than in the present study. HBBz was not detected in previous studies from Svalbard in liver in biota at medium-high trophic levels such as in kittiwakes, Arctic foxes and ringed seals (Sagerup et al., 2010). The overall few samples -including amphipods- where HBBz was detected in the present study, in combination with similar concentrations at remote areas on Svalbard suggests that the source might not be of local origin. HBBz is a fairly small and volatile compound that can undergo long-range transport (AMAP, 2017; de Wit et al., 2010) Nevertheless, diffuse run-off from local sources cannot be ruled out due to the findings close to the settlement in the present study.

3.3.3. Syn- and anti-dechlorane plus

Syn- and *anti*-DDC-CO were detected in all matrices in the study. The average concentrations in the sediment samples (6.2 and 9.6 pg/g dw of *syn*-DDC-CO and *anti*-DDC-CO, respectively) were lower than sediments from the Great Lakes (total DDC-CO concentration ranging between 330 and 26000 pg/g dw) but similar to sediment from Kongsfjorden at Svalbard (1.4 and 4.5 pg/g dw of *syn*-DDC-CO and *anti*-DDC-CO, respectively) (Ma et al., 2015; Shen et al., 2010; Sverko et al., 2011).

The relationship between *syn*- and *anti*-DDC-CO can be expressed as f_{anti}, which is the fraction of *anti*- compared to the sum of *syn*- and *anti*-DDC-CO. The technical DDC-CO mixture has a f_{anti} of 0.75. The f_{anti}-DDC-CO in air (0.5) and water (0.5) in the present study could indicate that these samples are closer to a source than earlier samples from East Greenland Sea (f_{anti} of 0.33), which is also supported by the higher dissolved concentrations reported in the present study (Table A7) (Möller et al., 2010). The *syn*-DDC-CO isomer is suggested to be more persistent to e.g. photodegradation in air compared to the *anti*-DDC-CO, although it might be affected by biodegradation in the sediment and water (Möller et al., 2010; Sverko et al., 2011). Hence, a lowered fraction of *anti*-DDC-CO compared to the technical mixture can indicate long-range transported of DDC-COs, which is the case in both Möller et al. (2010) and in the present study. Hence, local sources of DDC-COs cannot be excluded as explanation for the presence of DDC-COs in the present study. Both the sediment and amphipod samples in the present study had an average f_{anti} of 0.6 which indicates that there is little selective biotransformation of DDC-COs in the amphipods though, and that sediment poses a larger exposure to the amphipods than water with regards to uptake. Another recent study from

Kongsfjorden showed higher concentrations in the sediment; average of 270 and 73 pg/g dw of *syn*-DDC-CO and *anti*-DDC-CO, respectively (Na et al., 2015). Ma et al. (2015) suggest that the detection of DDC-CO in sediment from Kongsfjorden can be caused by transport by water currents from the Fram Strait. However, the highest concentrations measured in Kongsfjorden coincided with the ship route. Thus, contamination from ships could be a plausible explanation for detection of DDC-CO in sediment from Ny-Ålesund as well as from the Longyearbyen sediment samples. The general lack of data, analytical uncertainty between laboratories on these relatively little analysed compounds and lack of TOC data should also be taken into account when data are being compared.

The similar fraction measured in both air and water could indicate that there is an interaction between these phases where any degradation in one of the phases quickly reaches the other phase, or, the slightly lower f_{anti} ratio in air could also be interpreted as a result of photodegradation in air and a slow delivery of *syn*-DDC-CO from air to water. However, there is very little research on isomer selective photo- and biodegradation of DDC-COs, and other, e.g. microorganisms in the water column might degrade one or both of the DDC-COs. Stereoselective degradation is an important process for the fate of chiral environmental pollutants (Carlsson, 2013; Kallenborn and Hühnerfuss, 2001) and this process may be of high interest for further elucidation of the environmental fate of the chiral *syn*- and *anti*-DDC-CO.

3.3.4. Pentabromotoluene (PBT) and pentabromoethylbenzene (PBEB)

PBT and PBEB are considered as single/multiple hoppers in the atmosphere, meaning that they have the potential to undergo long-range transport Nevertheless, a local contribution from Longyearbyen cannot be excluded due to higher atmospheric concentrations in Longyearbyen (PBEB: <LOD-16 pg/m^3, PBT: <LOD-61 pg/m^3) compared to e.g. the East Greenland Sea (PBT; 0.001–0.02 pg/m^3) (Möller et al., 2011). This conclusion is somewhat hampered by the usage of passive air samplers that have an uncertain uptake/accumulation of less volatile compounds.

4. Conclusions

Passive sampling with silicone rubbers is a practical sampling technique for gaseous air and dissolved water fractions of PBDEs, nBFRs and DDC-COs in cold and remote climate where electricity driven sampling is not always feasible. The sampling campaign represents several months and thus shows the concentrations over time instead of a snapshot as with active sampling. In addition to the PBDEs, there is also a bioaccumulation potential for PBT, PBBz, *syn*- and *anti*-DDC-CO, and maybe also for α-DBE-DBCH, BEH-TEBP, HBBz and PBEB, although more data is needed before firm conclusions can be drawn. Many of the nBFRs were present in the sediment and also in the amphipods although not always above the detection limits in the water, which indicates that sediment is an important exposure pathway. Nevertheless, there is very little information available on nBFRs in water and in animals at low to medium trophic levels, and the present study is the first to report nBFRs and DDC-COs in Arctic benthic fauna. This paper contributes to a better understanding of bioaccumulation potentials of nBFRs and DDC-COs in the Arctic benthic ecosystem.

Acknowledgement

The study was funded by the Svalbard Environmental Fund grant number (15/79). We are also grateful for additional support from AMAP. We acknowledge Emma Johansson-Karlsson for assistance in the field and Katarina Bjarnar Løkken, Simon Vojta and Ondrej Audy for assistance with the analyses.

Appendix A. Supplementary data

Supplementary data related to this article can be found at https://doi.org/10.1016/j.chemosphere.2018.07.158.

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