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Occurrence of emerging brominated flame retardants and organophosphate esters in marine wildlife from the Norwegian Arctic[☆]

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ABSTRACT

To understand the exposure and potential sources of emerging brominated flame retardants (EBFR) and organophosphate esters (OPEs) in marine wildlife from the Norwegian Arctic, we investigated concentrations of EBFRs in 157 tissue samples from nine species of marine vertebrates and OPEs in 34 samples from three whale species. The samples, collected from a wide range of species with contrasting areal use and diets, included blubber of blue whales, fin whales, humpback whales, white whales, killer whales, walruses and ringed seals and adipose tissue and plasma from polar bears, as well as adipose tissue from glaucous gulls. Tris(2-ethylhexyl) phosphate (TEHP) and tris(2-chloroisopropyl) phosphate (TCIPP) ranged from <0.61 to 164 and < 0.8–41 ng/g lipid weight, respectively, in blue whales and fin whales. All other EBRFs and OPEs were below the detection limit or detected only at low concentration. In addition to the baseline information on the occurrence of EBFRs and OPEs in marine wildlife from the Arctic, we provide an in-depth discussion regarding potential sources of the detected compounds. This information is important for future monitoring and management of EBFRs and OPEs.

1. Introduction

Flame retardant (FR) chemicals are widely used to decrease flammability of many consumer products, like furniture, textiles and electronics (Alaee, 2003). A large range of new FRs have been introduced to replace polybrominated diphenyl ethers (PBDEs), hexabromocyclododecane (HBCDD) and hexabromobiphenyl (HBB), which have been gradually phased out since early 2000s (Abbasi et al., 2019; Li and Wania, 2018). PBDEs, HBCDD and HBB leaked into the environment, where they persisted, and were found to travel over long distances, bioaccumulate in and pose health threats to wildlife and humans (http://chm.pops.int/). Consequently, tetra, penta and octaBDE, and HBB were listed under Annex A (i.e., eliminate the production and use) of the Stockholm Convention on Persistent Organic Pollutants (POPs) in 2009, followed by HBCDD in 2013 and decaBDE in 2017. Despite of the global actions to regulate use of PBDEs, HBCDD and HBB, the global use of these FRs increased ~40% from 2008 to 2017 (Wang et al., 2020c). Over two million tons of flame retardants were used globally in 2017, of which a third were emerging brominated FRs (EBFRs) and organophosphate esters (OPEs) (UN Environment, 2019). OPEs, which are also used as plasticizers in addition to being flame retardants, are not chemically bonded to the product that they are meant to protect, and readily leak out of treated products, decreasing their fire safety properties over time (van der Veen and de Boer, 2012).

EBFRs and OPEs have been described as "regrettable substitutions" for PBDEs due to their ubiquitous presence in the environment and their toxic properties (Blum et al., 2019; Zuiderveen et al., 2020). Like PBDEs, they are present worldwide, even in remote Arctic regions, which

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indicates both their persistence and capacity for long-range transport (Fu et al., 2021; Hermanson et al., 2010; Moller et al., 2012; Salamova et al., 2014). Atmospheric concentrations of OPEs are globally at least an order of magnitude higher than PBDE concentrations, whereas concentrations of EBFRs are generally lower than PBDE concentrations (Rauert et al., 2018). However, EBFRs have been shown to bioaccumulate in marine predators, though to a lesser extent than legacy POPs, while OPEs seem to be rapidly metabolized and show low bioaccumulation potential in marine predators (de Wit et al., 2020; Garcia-Garin et al., 2020; Greaves et al., 2016; Strobel et al., 2018b). ***In vitro*** studies on cell lines and *in vivo* experimental studies on invertebrates, fish, birds and rodents have demonstrated that EBFRs have the potential to cause endocrine disruption, genotoxicity and behavioral modification (Xiong et al., 2019). Likewise, experimental and human epidemiological studies suggest that OPEs also elicit neurotoxicity, cardiotoxicity, hepatotoxicity and endocrine disruption as well as reproductive and developmental toxicity (Blum et al., 2019; Yan et al., 2021). Current exposure levels for both halogenated and non-halogenated OPEs are of concern for human and animal health (Blum et al., 2019), whereas available data suggests that current environmental concentrations of EBFRs are lower than toxicity thresholds (Xiong et al., 2019). However, this assessment could be challenged given the limited amount of scientific data available to assess toxicity.

To our knowledge, EBFRs are currently not regulated, although their production and use in Europe and USA is monitored to some extent (Zuiderveen et al., 2020). Only a few regulations have been established for OPEs, but regulatory authorities have started to recognize the risk they pose to animal and human health and the need to gather data for further assessment of risk (Blum et al., 2019). Among regulated OPEs, for example, tris(2-chloroethyl)phosphate (TCEP) is listed as a chemical of high concern in Europe (European Chemicals Agency, 2022a), and is included in the Toxic Substances Control Act in the US (U.S. Environmental Protection Agency, 2014). Also, regulatory efforts are ongoing in Europe and North America for tris(1,3-dichloroisopropyl) phosphate (TDCIPP), a high production volume neurotoxic and a potential carcinogenic chemical that is widely found in the environment and humans (Wang et al., 2020a).

Studies on chemicals of emerging concern in Arctic wildlife are highly relevant for management as they may indicate the potential for persistence, long-range transport, bioaccumulation and biomagnification of chemicals in food webs. These properties fulfill three out of four requirements for a chemical being classified and regulated as a persistent organic pollutant. However, EBFRs and OPEs have been studied only in a few Arctic species and there is limited knowledge on these chemicals in Arctic wildlife populations (reviewed by Marteinson et al., 2021; Vorkamp et al., 2019).

The Barents Sea and the Norwegian Sea are highly productive ecosystems that host a large number and diversity of mammalian and avian predators (Sakshaug et al., 2009; Skjoldal, 2004; Yaragina and Dolgov, 2009). Studying contaminants of emerging concern in marine predators with contrasting space use and feeding strategies may give us insight about partitioning and bioaccumulation of these compounds within ecosystems. Marine mammals and avian predators also vary in their abilities to metabolize xenobiotic compounds. Polar bears (*Ursus maritimus*) generally have high biotransformation abilities while whales and birds have a limited number of genes involved in detoxification (Hecker et al., 2019; Kim et al., 2016; Tian et al., 2019; Zhao et al., 2015), which affects their ability to transform pollutants into excretable metabolites.

The aim of this study was to understand the exposure and potential sources of EBFRs and OPEs in marine biota from the Norwegian Arctic. The presence and accumulation of EBFRs and OPEs was investigated in a wide range of marine wildlife with contrasting areal use and diets (Table 1). EBFRs were analysed in blubber/adipose tissue of blue whales (*Balaenoptera musculus*), fin whales (*Balaenoptera physalus*), humpback whales (*Megaptera novaeangliae*), killer whales (*Orcinus orca*), white whales (*Delphinapterus leucas*), ringed seals (*Pusa hispida*), walruses

Table 1
Biological information on the animals sampled from Svalbard and northern Norway. Age and lipid content are given as median and range.

Species	n	Year	Sex (F/M)	Age (group or years)	Tissue	Tissue lipid content (%)	Area used by the study population	Main diet
Blue whale	15	2014–2017	4/11	Adult	Blubber	49.4 (24.3, 62.7)	Barents Sea, North Atlantic[b]	Krill[c]
Fin whale	12	2014–2017	8/4	Adult	Blubber	44.9 (26.1, 57.8)	Barents Sea, North Atlantic[d]	Krill, fish[c]
Humpback whale	5	2018	4/1	Adult	Blubber	44.3 (39.7, 69.9)	Barents Sea to West Indies[e]	Fish[c]
White whale	13	2014–2016	8/4/?	Adult	Blubber	78.4 (33.5, 98.0)	Barents Sea[f]	Fish[f]
Killer whale	24	2018	5/19	Adult/Juvenile (n = 22/2)	Blubber	31.2 (8.05, 46.7)	Northern Norway/Barents Sea[g]	Fish, seals[h]
Walrus	18	2014	0/18	Adult	Blubber	30.4 (9.8, 52.6)	Barents Sea[i]	Benthic bivalves[j]
Ringed seal	10	2014	3/7	17 (3, 26)	Blubber	90.3 (81.4, 93.8)	Barents Sea[k]	Fish[l]
Polar bear	6	2012	0/6	15 (7, 19)	Adipose tissue	40.9 (21.8, 55.5)	Barents Sea[m]	Seals[n]
Polar bear	65	2014–2017	65/0	12 (4, 24)	Plasma	1.25 (0.53, 1.63)	Barents Sea[m]	Seals[n]
Glaucous gull	5	2011	0/5	Adult	Adipose tissue	86.5 (52.5, 98.6)	Barents Sea, Iceland, northern Norway[o]	Plankton, fish, seabirds, eggs[p]

[a] 1 unknown.
[b] (Silva et al., 2019).
[c] (Gavrilchuk et al., 2014).
[d] (Lydersen et al., 2020).
[e] (Smith et al., 1999; Vacquié-Garcia et al., 2018).
[f] (Vacquié-Garcia et al., 2018).
[g] (Dietz et al., 2020; Vogel et al., 2021).
[h] (Bories et al., 2021; Jourdain et al., 2020; Vogel et al., 2021).
[i] (Lydersen et al., 2008; Wiig et al., 2009).
[j] (Gjertz and Wiig, 2009; Scotter et al., 2019).
[k] (Hamilton et al., 2015; Hamilton et al., 2016).
[l] (Bengtsson et al., 2020).
[m] (Blanchet et al., 2020; Mauritzen et al., 2001).
[n] (Derocher et al., 2002; Iversen et al., 2013).
[o] http://seatrack.seapop.no/map/.
[p] (Wold et al., 2011).

(*Odobenus rosmarus*), polar bears and glaucous gulls (*Larus hyperboreus*). Additionally, OPEs were analysed in blubber of blue whales, fin whales, and white whales.

2. Material and methods

2.1. Field sampling

The tissue samples were collected in or around the Svalbard Archipelago (76.5–80.7 N, 9.3–23.5 E) and in coastal waters off northern Norway (70 N, 21 E) (Fig. 1, Table 1).

Twelve fin whales and 15 blue whales were biopsied during May to September 2014–2017 (fin whales: 2014: n = 3, 2015: n = 2, 2016:n = 1, 2017: n = 6; blue whales 2014: n = 2, 2015: n = 4, 2016: n = 5, 2017: n = 4) off the west and north coast of the Svalbard Archipelago (Fig. 1). Five humpback whales and 24 killer whales were sampled in Kvænangen Fjord, northern Norway, in December 2017 and January 2018 (Fig. 1). A custom made 10 cm long steel biopsy dart was attached to a crossbow

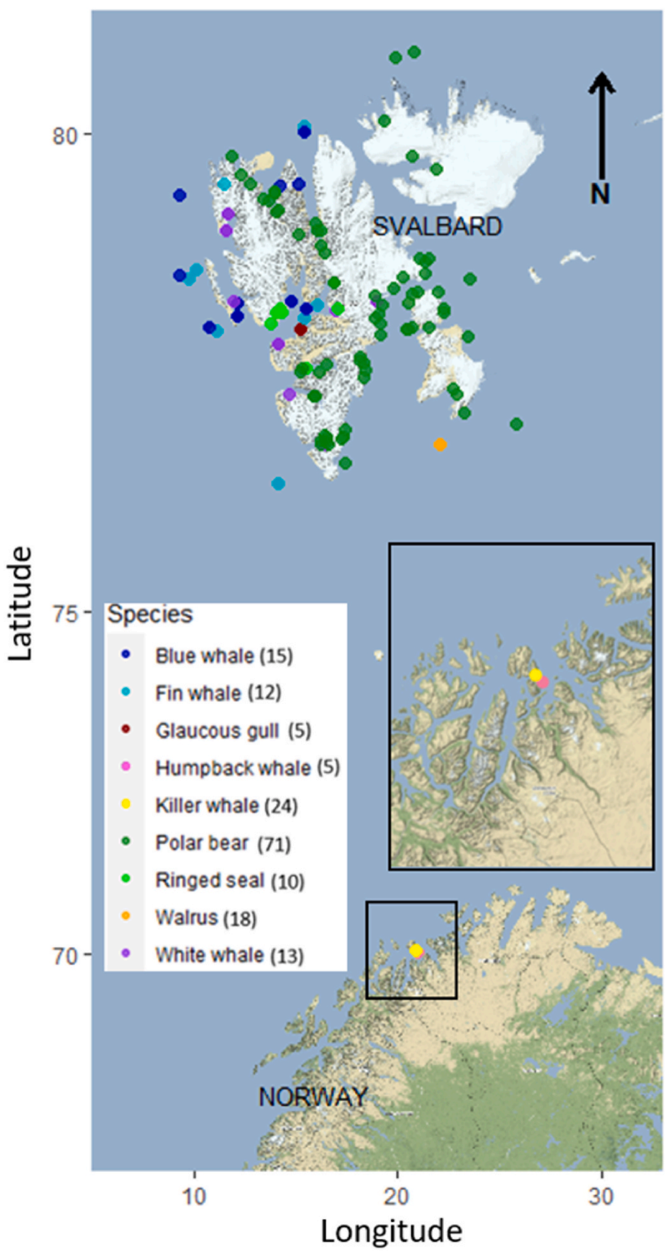

Fig. 1. Sampling locations and sample size for the various species in this study.

arrow and the arrow was attached to a string that ensured recovery of the whale biopsy sample. The upper layer of the biopsy, approximately 1–5 cm from the surface of the skin, was used for contaminant analyses. Adult white whales were live-captured in Svalbard in July–August 2014–2016 (2014: n = 6, 2015: n = 2, 2016: n = 5) in a nylon net set from the shore. Vertical blubber cores (from the skin through to the inner blubber layer (next to the muscle)), were collected using a custom-made steel collection tube (8 mm diameter) from the back of the animal, just in front of (cranially to) the dorsal ridge. The blubber from approximately 1-4 cm below the cork and epidermis was used for contaminant analyses.

Blubber was collected from the mid dorsal region of 10 ringed seals shot in western Svalbard in May and September in 2014 by local sport hunters (Fig. 1). The ages of the seals were determined by counting cementum layers in decalcified, stained longitudinal sections of the canine teeth (Lydersen and Gjertz, 1987).

Blubber biopsies were collected from 18 adult male walruses from Svalbard in July 2014 (Fig. 1). Walruses were immobilized on shore with an intramuscular injection of etorphine hydrochloride, with naltrexone as a reversal agent (Ølberg et al., 2017). Tusk volume based on tusk length and girth at the proximal end was used as a proxy for age (Skoglund et al., 2010). Blubber biopsies were collected from the mid dorsal region using a custom-made hollow, stainless steel corer.

Polar bears were immobilized by remote injection of tiletamine hydrochloride and zolazepam hydrochloride (Zoletil Forte Vet®; Virbac, France) from a helicopter (Eurocopter AS350 Ecureuil). Adipose tissue was collected from six adult male polar bears captured in April 2012 in Svalbard, using a sterile disposable 6 mm biopsy punch (Fig. 1). The tissue was collected about 15 cm lateral to the base of the tail. Plasma samples were collected from 65 female bears sampled in late March and April 2014–2017 (2014: n = 6, 2015: n = 17, 2016: n = 23; 2017: n = 19) from Svalbard (Fig. 1). Blood was collected from the femoral vein into heparinized vacutainers and plasma was separated within 12 h by centrifugation.

Five adult glaucous gulls were collected at Fuglefjella close to Grumantbyen, in Isfjorden, Svalbard (Fig. 1). The birds were killed using a shotgun during the post-breeding period, in late August 2011. Subcutaneous fat samples were collected from frozen carcasses.

All sampling procedures were approved by the National Animal Research Authority of Norway and the Governor of Svalbard.

2.2. Sample packing and storage

Blue whale and fin whale biopsies collected in 2017 were packed in cleaned glass vials and aluminum foil was placed inside of a polypropylene top cap, with septum lined with polytetrafluoroethylene (PTFE) foam urethane (National Scientific, Austin, Texas, USA). Both the vials and aluminum foil were cleaned at 450 °C for 8 h. The caps were cleaned using an ultrasonic bath (10 min) and rinsed with acetone. Biopsy darts for whale sampling, tweezers and scissors were sterilized by boiling them for 15 min and then they were washed with acetone and packed individually in aluminum foil. Longitudinal slices of killer whale and humpback whale blubber cores were wrapped in acetone-cleaned aluminium foil and then placed into an acetone cleaned glass vial. The remaining blubber/fat samples were packed in aluminum foil and placed into plastic bags. Polar bear plasma samples were stored in cryovials. All samples were stored at −20 °C until analyses.

2.3. Molecular sexing

Sex of the whales was identified from molecular sexing using skin samples (see Berube and Palsbøll, 1996). Briefly, two sets of three oligonucleotides primers were used for PCR amplification of the ZFX/ZFY sequence specific to mysticetes and odontocetes.

2.4. Analyses of EBFRs and OPEs in blubber/adipose tissue samples

The list of full names, abbreviations and CAS numbers are given in Table S1. The samples were analysed in five batches in 2016–2019 and included 4–14 EBFRs and 14–17 OPEs (Table S2). Not all compounds were analysed in all batches because of tissue availability. But, adjustments of the methods over the years allowed for analyses of a larger number of compounds during later years of the study (Table S2).

EBFRs and OPEs were extracted in blubber and adipose tissue samples in a similar manner. Approximately 0.3 g of sample was cut into pieces and transferred into 50 mL glass vials that were sealed with aluminium foil and a metal lid. Ten grams of burnt Na_2SO_4 (600 °C/8 h) was added and the sample was left in the freezer (−20 °C) over night. For EBFRs analyses, 20 ng of isotopically labelled internal EBFR standard consisting of ^{13}C decabromodiphenylethane (DBDPE), ^{13}C 1,2-bis(2,4,6-tribromophenoxy) ethane (BTBPE), ^{13}C pentabromobenzene (PBBz), ^{13}C hexabromobenzene (HBBz) and ^{13}C d17 2-ethylhexyl 2,3,4,5-tetrabromobenzoate (EH-TBB) (Cambridge Isotope Laboratories Inc., CIL, MA, USA) were added to the preparation. For OPEs analyses, 10 ng of deuterated d12-TCEP, d18-tris(2-chloroisopropyl) phosphate (TCIPP), d18-TDICPP, d27-tris-n-butyl phosphate (TNBP) and d15-tris(phenyl) phosphate (TPhP) purchased from CIL and Chiron (Trondheim, Norway) were added as internal standards to the preparation. Contaminants were extracted with 40 mL of 1:3 acetone:cyclohexane, sonicated for 10 min. The extraction was repeated with 30 mL of the solvent mixture and then with 30 mL of 1/1 acetone/cyclohexane. The extracts were pooled and evaporated in a Rapidvap vacuum evaporation system (Labconco, MO, USA) until constant weight (dryness) was achieved to determine lipid content. Extracts for EBFRs were redissolved in 2 mL dichloromethane. The cleanup of the extracts for EBFR analyses was performed by gel permeation chromatography (GPC) on two serially connected 19 mm × 150 mm and 19 mm × 300 mm Envirogel columns (Waters Inc. MA, USA) using dichloromethane at 5 mL/min. The fraction collected between 12.5 and 25 min was further cleaned up using RapidTrace® Automated Solid Phase Extraction (SPE) Workstation (Biotage AB, Uppsala, Sweden) with 1 g of activated Florisil (450 °C/8h). The fraction was eluted using 14 mL 25% dichloromethane in n-hexane. The fraction was then concentrated to 50 μL and 4 ng of recovery standard (^{13}C PCB 159) were added. Extracts for OPE analyses were cleaned up using Supelclean EZ-POP NP columns (Sigma-Aldrich inc., MO, USA), eluted using 14 mL of acetonitrile, concentrated to 0.1 mL, and recovery standard (10 ng of d15-TPP) and 50 μL cleaned water (MilliQ Advantage A10, Millipore, Merck KGaA, Darmstadt, Germany) were added.

EBFR analyses (before 2018) were performed on a gas chromatography (GC) coupled to a tandem quadrupole mass spectrometer (MS) (Quattromicro, Waters Inc. MA, USA). More recent EBFR analyses were performed using a Thermo Scientific Exactive high resolution, accurate Mass Spectrometer (HRAM), coupled to a GC (Thermo Fisher Scientific, Waltham, MA USA). Analyses of OPEs were run on an ultra-high-pressure liquid chromatograph (UPLC) connected to a triple quadrupole MS (TSQ Vantage, Thermo Scientific Inc.). Details for instrumental analyses are given in the supporting information.

2.5. Quality assurance of EBFR and OPE analyses in blubber/adipose tissue samples

The quality of the analyses was monitored using sample blanks and reference samples of standard reference material EDF-2524 (clean fish reference material from Cambridge Isotope Laboratories, Andover, MA, USA) spiked with native OPEs and fish oil from the first worldwide interlaboratory study on OPE analyses (Brandsma et al., 2013). Recovery of internal standards for EBFRs and OPEs, and native OPE standards are reported in Table S3-4. Recovery for DBDPE was not acceptable (<10%) for batches 1–3, and hence the results are not reported. Limits of detection (LODs) and quantification (LOQ) (Table S3-4) were calculated as the average concentration in the procedural blanks plus three and ten times the standard deviation, respectively. Possible OPE contamination during field sampling was monitored using a field blank in 2017, which were prepared of 0.25 g of seal blubber sample in cleaned glass vials with cleaned aluminum foil and screw caps. One field blank was stored in the sample processing laboratory (−20 °C) as a reference and was processed at the same time as the whale samples and field blank. The field blank was open in the ambient air for a few minutes. Differences in OPE concentrations between the reference samples and field blank were minor as reported in Table S4.

2.6. Analyses of EBFRs in plasma samples

Analyses of EBFRs in polar bear plasma were conducted at the Laboratory of Environmental Toxicology at The Norwegian University of Life Sciences in Oslo (NMBU), located in Ås. The laboratory is accredited by the Norwegian Accreditation for testing the analysed chemicals in biological material according to the requirements of the NS-EN ISO/IEC 17025 (TEST 137). EBFRs were extracted simultaneously with POPs (Lippold et al., 2019) based on a previously described liquid/liquid extraction method (Brevik, 1978), modified by Gabrielsen et al. (2011) to extract OH-metabolites simultaneously. Description of further treatment of the extracts, specification of analytical instruments, quantification and quality assurance is described by Polder et al. (2016).

For extraction of EBFRs, 2 g of plasma was added to internal standards for PCB 29, 112 and 207 (Ultra Scientific, N. Kingstown, RI, USA), BDE 77, 119 and 181 and $^{13}C_{12}$-BDE 209 (Cambridge Isotope Laboratories) and 2-endo,3-exo,6-exo,8,9,10,10-heptachlorobornane (DETOX 409; LGC Standards GmbH, Wesel, Germany). The lipid content of the samples was determined gravimetrically.

Quality control included analyses of a blind sample, several spiked recoveries, blanks, and standard reference materials. Average recovery of pentabromotoluene (PBT), pentabromoethylbenzene (PBEB), 2,4,6-tribromophenyl 2,3-dibromopropyl ether (TBP-DBPE) and HBBz in the spiked samples was 87–96%.

2.7. Statistical analysis

The datasets are available in the Norwegian Polar Data Centre repository (data.npolar.no) (Routti et al., 2022). Statistical analyses were carried out using R version 3.6.1 (R Core Team, 2019). All concentrations were lipid corrected (concentration / % lipid content * 100). LODs and LOQs were lipid transformed using the median % lipid content per species to avoid large variations. Median contaminant concentrations and summary statistics for boxplots were calculated with the R-package NADA (Lee, 2020), which was specifically developed for left-censored environmental data analysis (Helsel, 2012). The Robust Regression on Order Statistics (Helsel, 2012) was applied for all compounds that were detected above LOQ in ≥50% of the samples. This method has the benefit of handling multiple censored limits and is more accurate than applying the commonly used substitutions (Helsel, 2012). For contaminants that were detected above LOQ in less than 50% of samples, the range of the detected concentrations was reported. To assess the goodness of fit and validity of distributional assumptions probability plots were used (Fig. S1) for censored data from the R package *EnvStats* (Millard, 2013). Species and sex differences for compounds detected in >80% of samples from one or more species were tested using linear models. Values below the detection limit were replaced by half of the limit of detection. 95% confidence intervals (CI) of the estimates were used to determine whether the differences were significant from 0 at the 5% confidence level.

3. Results and discussion

Concentrations of EBFRs and OPEs in ng/g lipid weight (lw) are given in Tables 2 and 3. Wet weight concentrations are provided in Table S5 and S6.

Table 2
Concentration ranges for emerging brominated flame retardants (ng/g lipid weight) in adipose tissue/blubber/plasma of marine wildlife sampled from the Svalbard Archipelago and northern Norway. Limits of detections and quantification (in italics) were transformed from wet weight to lipid weight using the median lipid percentage for each species (Table 1). Different analytical batches are separated by a slash. Number of samples with detectable concentrations is shown in parentheses. n.a.: not analysed

	Blue whale blubber n = 11	Fin whale blubber n = 6	Humpback whale blubber n = 5	White whale blubber n = 6–8	Killer whale blubber n = 24	Walrus blubber n = 18	Ringed seal blubber n = 10	Polar bear adipose tissue n = 6	Polar bear plasma n = 65	Glaucous gull adipose tissue n = 5
TBP-AE rowhead	n.a.	n.a.	<0.16	<0.06	<0.22	n.a.	n.a.	<0.12	n.a.	<0.06
TBP-BAE rowhead	n.a.	n.a.	<0.16	<0.03	<0.22	n.a.	n.a.	<0.05	n.a.	<0.02–0.11 (2)
TBP-DBPE rowhead	<0.02	<0.02	<0.16	<0.01	<0.22	<0.39	<0.13	<0.02	<0.48	<0.01
α-DBE-DBCH rowhead	n.a.	n.a.	<0.16	<0.38	<0.22	n.a.	n.a.	<0.73	n.a.	<0.35
β-DBE-DBCH rowhead	n.a.	n.a.	<0.16	<0.25	<0.22	n.a.	n.a.	<0.5	n.a.	<0.23
γ,δ-DBE-DBCH rowhead	n.a.	n.a.	<0.23	<0.25	<0.32	n.a.	n.a.	<0.5	n.a.	<0.23
PBBz rowhead	n.a.	n.a.	<0.16	n.a.	<0.22	n.a.	n.a.	n.a.	n.a.	n.a.
HBBz rowhead	<0.09	<0.10	<0.16	<0.06[a]	<0.22	<0.39–1.45 (1)	<0.13	n.a.	<0.90	n.a.
PBT rowhead	<0.08	<0.09	<0.16	<0.01/ <0.05	<0.22	<0.39	<0.13	<0.02	<0.36	<0.01–0.05 (1)
PBEB rowhead	<0.09	<0.09	<0.16	<0.01/ <0.05	<0.22	<0.39	<0.13	<0.02	<0.42	<0.01
EH-TBB rowhead	n.a.	n.a.	<0.16	n.a.	<0.22–1.05 (1)	n.a.	n.a.	n.a.	n.a.	n.a.
BTBPE rowhead	n.a.	n.a.	<0.16	n.a.	<0.22	n.a.	n.a.	n.a.	n.a.	n.a.
BEH-TEBP rowhead	n.a.	n.a.	<4.29	n.a.	<6.09	n.a.	n.a.	n.a.	n.a.	n.a.
DBDPE rowhead	n.a.	n.a.	<1.13–3.93 (1)	n.a.	<1.60–12.9 (4)	n.a.	n.a.	n.a.	n.a.	n.a.

[a] n = 2 for analysed samples.

3.1. *Emerging brominated flame retardants*

The presence of the EBFRs in biota sampled from the Norwegian Arctic was very limited despite low LODs for all compounds except bis (2-ethylhexyl) tetrabromophthalate (BEH-TEBP) (Table 2). Five of the 14 targeted EBFR compounds or isomers were detected in at least one sample across the different species (Table 2). The most frequently detected EBFR, DBDPE, was present in four killer whale samples (≤12.9 ng/g lw) and one humpback whale sample (3.39 ng/g lw). DBDPE has also been reported in various samples from marine mammals from the Arctic (Harju et al., 2013; Vorkamp et al., 2015) and elsewhere (Aznar-Alemany et al., 2021; Zhu et al., 2014), although it is subject for partial degradation in liver microsomes of marine mammals (McKinney et al., 2011a). DBDPE, a currently-used alternative for decaBDE, has been proposed for restricted usage in Canada (Canadian Gazette, 2022) and is under assessment as a persistent, bioaccumulative and toxic substance in Europe (European Chemicals Agency, 2022b). Its production volume reached 30 000 t/y in China in 2016 (Shen et al., 2019), while in USA the reported production volumes were in the range of 9000–45 000 t/y in 2019 (U.S. Environmental Protection Agency, 2020).

2,4,6-tribromophenyl allyl ether (TBP-BAE) and PBT were found in two and one glaucous gull sample, respectively. HBBz was found in one walrus sample and EH-TBB in one killer whale sample at low but quantifiable concentrations (≤1.45 ng/g lw). 2,4,6-tribromophenyl allyl ether (TBP-AE), TBP-DBPE, 4-(1,2-dibromoethyl)-1,2-dibromocyclohexane (DBE-DBCH) isomers, PBBz, PBEB, BTBPE and BEH-TEBP were not detected in any of the samples; it should be noted that not all compounds were analysed in all species (Table 2). A recent review on current-use halogenated FRs in the Arctic also concluded that the levels of EBFRs in Arctic biota and abiotic media are generally low (Vorkamp et al., 2019). For example, PBT, TBP-DBPE, PBEB and HBBz were below quantifiable levels in polar bears from the Canadian and Norwegian Arctic (McKinney et al., 2011b; Tartu et al., 2017), HBBz was detected at low concentrations in 31% and PBEB in 4% of ringed seal samples from the Canadian Arctic (LOD ≤0.004 ng/g ww) (Houde et al., 2017). Additionally, HBBz and PBEB were detected on average ≤ 1 ng/g ww in blubber of white whales and minke whales from the St Lawrence Estuary and glaucous gull egg yolks from the Norwegian Arctic (Simond et al., 2019; Verreault et al., 2007). EBFRs in plasma of mother and pup hooded seals (*Cystophora cristata*) sampled from east of Greenland were also below the detection limit (<0.03 ng/g ww) (Villanger et al., 2013), while β-DBE-DBCH was detected in white whale blubber (1.1–9.3 ng/g lw) from Canada (Tomy et al., 2008). Studies on the same killer whale population sampled in the current study, reported HBB and PBT ranged from <LOD (<0.017 ng/g ww) to 2 and 14 ng/g lw in blubber, respectively, whereas DPTE was detected only in one sample (Andvik et al., 2021, 2020).

3.2. *Organophosphate esters*

Tris(2-ethylhexyl) phosphate (TEHP) was the most frequently detected and abundant OPE in blue whales, fin whales and white whales (Table 3). This compound was detected in 93% of the blue whales and all of the fin whales, with concentrations ranging from <0.61 to 164 ng/g lw, and, in two out of seven white whale samples (≤23 ng/g lw) (Table 3). The differences in TEHP concentrations in fin whales *vs* blue whales (+58%) and in males *vs* females (−20%) were not statistically

Table 3
Concentration ranges for organophosphate esters (ng/g lipid weigh) in adipose tissue of blue whales, fin whales and white whales sampled from the Svalbard Archipelago. Limits of detections and quantification (in italics) were transformed from wet weight to lipid weight using the median lipid percentage for each species (Table 1). Different analytical batches are separated by a slash. Number of samples with detectable concentrations is shown in parentheses. Medians for compounds with >50% of the samples > LOQ were calculated using Robust Regression on Order Statistics. n.a.: not analysed.

	Blue whale n = 15	Fin whale n = 12	White whale n = 7
TCEP rowhead	<3.9/<3.0	<4.2/<3.3	<2.42
TCIPP rowhead	<0.8–14.5 (5)	<0.9–40.9 (7)	<3.29
TDCIPP rowhead	<1.25	<1.34–6.15 (1)	<0.82
TEP rowhead	<0.58[a]	<0.63[c]	<0.36
TPP rowhead	<0.07/<0.20	<0.08/<0.22	<0.05
TNBP rowhead	<2.18/<0.61	<2.40/<0.67	<1.38
TIBP rowhead	<3.39-<3.94 (1)/<8.91	<3.72/<9.79	<2.13
TBOEP rowhead	<2.63-<3.17 (1)	<2.89-<3.45 (1)	<1.94-<5.95 (1)
TEHP rowhead	<0.61–164 (14)	3.12–106 (12)	<0.38–22.7 (2)
Median rowhead	4.86	13.5	
dBPhP rowhead	<0.13/<0.21	<0.14/<0.23	<0.08
BdPhP rowhead	<0.25/<0.42	<0.27/<0.45	<0.15
TPhP rowhead	<0.41–18.5 (2)	<3.06/<0.45–0.76[a] (2)	<1.78–8.39 (2)
TMPP rowhead	<0.13/<0.42	<0.13–4.68 (2)	<0.08
TXP rowhead	<1.01[b]	<1.11[c]	n.a.
TTBPP rowhead	<0.41[b]	<0.45[c]	n.a.
TIPPP rowhead	<2.03[b]	<2.23[c]	n.a.
EHDPP rowhead	<0.61-<5.43 (3)/<5.36	<0.67/<5.88–9.12 (2)	<3.36-<10.2 (1)

[a] n = 11.
[b] n = 4.
[c] n = 6 for analysed samples.

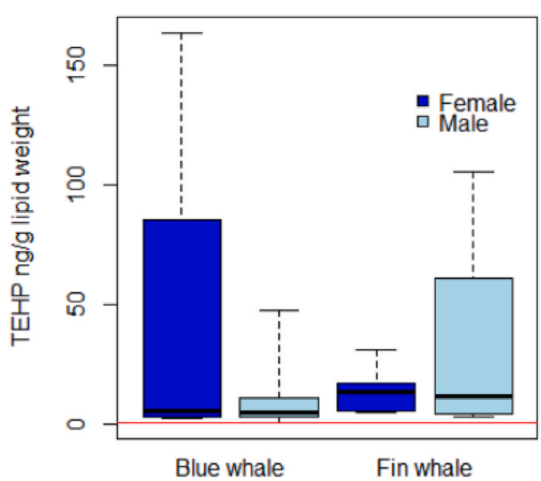

Fig. 2. Boxplot showing concentrations of TEHP in blubber from female and male blue whales (n = 4 and 11, respectively) and fin whales (n = 8 and 4, respectively) sampled from the Norwegian Arctic. Since TEHP values were partly below the limit of detection, robust Regression on Order Statistics were used to estimate the ranges. The limit of detection is marked with the red line. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

significant (95% CIs: [−42, 330] and [−70, 119], respectively) (Fig. 2). TCIPP was present in 44% of the baleen whale samples (<0.9–41 ng/g lw). The detection frequency for these two compounds in blue whales and fin whales was higher than the detection frequency for any OPEs in phocid seals, polar bears, black-legged kittiwakes, Brunnich guillemots, glaucous gulls or Arctic foxes (*Vulpes lagopus*) from Svalbard sampled in 2007–2010 (Hallanger et al., 2015). Furthermore, OPEs were quantified only at low levels in ringed seal blubber and polar bear adipose tissue from East Greenland (Strobel et al., 2018b).

A different set of OPEs was analysed in fin whale muscle and krill from Iceland in a study by Garcia-Garin et al. (2020). Fin whales sampled in Iceland and those used in our study sampled in Svalbard likely belong to the same population (Lydersen et al., 2020). The most abundant OPE compounds in both the whales and krill in Garcia-Garin et al.'s (2020) study were TNBP, isopropylated triphenyl phosphate and triphenylphosphine oxide, whereas isopropylphenyl diphenyl phosphate was the most frequent OPE in fin whales. Although the sum of OPEs detected was on average ~1000 ng/g lw in fin whale muscle samples, neither TCIPP or TEHP were detected. This may be due to high detection limits of OPEs, which were up to 19 ng/g lw (Garcia-Garin et al., 2020). Levels of OPEs in cetaceans from the Arctic were an order of magnitude lower than levels reported for blubber of common dolphin collected from the Mediterranean Sea (Sala et al., 2019).

The presence of OPEs in the whale samples is potentially related to their limited capacity for biotransformation. Serum hydroxylases, mainly paraoxonases, are involved in hydroxylation of organophosphate triesters to their respective diesters (Van den Eede et al., 2016), whilst hepatic phase I and II enzymes catalyze oxidative metabolism, dealkylation and conjugation of OPEs (Van den Eede et al., 2013). Interestingly, marine mammals have lost paraoxonase 1 and important transcription factors of phase I enzymes in the course of evolution (Hecker et al., 2019; Meyer et al., 2018) and they also possess a low number of phase II enzymes (Kim et al., 2016; Tian et al., 2019), which make them more prone to accumulation of OPEs. *In vitro* metabolism in OPEs in hepatic microsomes has also been shown to be slower in ringed seals than in polar bears (Strobel et al., 2018a, 2018b).

Fin whale and blue whales may be exposed to OPEs when feeding in the Arctic, but recent studies indicate that both species may also feed at lower latitudes (Lydersen et al., 2020; Silva et al., 2019). The presence of TEHP in blue whales and fin whales, that feed extensively on krill, is likely related to the high bioaccumulation potential of TEHP at low trophic levels. In a marine food web from China, TEHP showed the highest bioaccumulation factor (mean log BAF = 4.6 from water to fish and invertebrates) and trophic magnification factor (2.52) among 20 OPEs studied (Bekele et al., 2019). Similarly, bioaccumulation from water to fish was the highest for TEHP among six to eight OPEs studied in freshwater fish (Bekele et al., 2018; Hou et al., 2017). The high bioaccumulation and biomagnification potential of TEHP is thought to be related to its high lipophilicity (Bekele et al., 2019).

The presence of TCIPP, the second most frequently detected OPE in whale samples from Svalbard, may be related to its high production volume and potential for long-range transport. TCIPP is a commonly used flame retardant with estimated annual production of 45000 tons/year (Huang et al., 2022; Wang et al., 2019). It is highly resistant to waste water treatment and thus it is discharged to natural waters (Cristale et al., 2016; Martínez-Carballo et al., 2007). The long half-life of TCIPP in water (213 days) (Zhang et al., 2016) makes it prone to long-range oceanic transport. Consequently, it is the most abundant OPE in seawater in the North Atlantic, Fram Strait and Arctic Ocean (Li et al., 2017; McDonough et al., 2018). TCIPP may also be transported via the atmosphere to remote areas and it is the dominant OPE (along with TCEP) in air samples from the North Atlantic, the Arctic and also in the Antarctica (Li et al., 2017; Sühring et al., 2021; Wang et al., 2020b). TCIPP was the most abundant OPE in fish samples from Svalbard (Hallanger et al., 2015). In addition, TCIPP has been shown to bioaccumulate in fish and marine invertebrates and to biomagnify in marine food webs (up to fish), but to a lesser degree than TEHP (Bekele et al., 2019; Bekele et al., 2018). The trophic magnification factor for TCIPP in an Antarctic marine food web ranging from algae to fish and birds (feathers) was 2.92 (Fu et al., 2020).

3.3. Limitations

The limitations of the study are related to the variation in the chemical analyses through time. The quality of the analyses was high (Table S3-4), although the limits of detection were variable among batches of samples, but adjustments to the methods over the years did allow us to increase the set of compounds analysed. An additional concern relates to sample size; due to the challenges of collecting samples from Arctic marine mammals and limitations in resources, the number of samples collected for some species were relatively small.

4. Conclusions

The present study provides knowledge on the occurrence and potential sources of EBFRs and OPEs in marine biota from the Norwegian Arctic, which is important for future monitoring and management of these toxic compounds. Various sources may contribute to the TEHP and TCIPP in pelagic migratory baleen whales. We recommend future studies to investigate the presence of a wide range of EBFRs and OPEs in various compartments of Arctic ecosystems. Future studies should cover potential metabolites of OPEs in Arctic biota. OPEs should also be studied in species, such as killer whales, that are known to be exposed to high concentrations of other pollutants (Andvik et al., 2020). Further studies should also focus on potential health risks related TEHP and TCIPP exposure in pelagic baleen whales.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The dataset is available in the Norwegian Polar Data Centre repository (Routti et al., 2022; https://doi.org/10.21334/npolar.2022.a3bc6d92).

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.envpol.2022.120395. It includes the detailed description of instrumental analyses of EBFRs and OPEs, an overview of the samples and compounds analysed in different batches, information regarding quality assurance for the different batches, EBFR and OPE concentrations in wet weight and results and QQ-plots of the Robust Regression on Order Statistics.

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