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Levels of brominated flame retardants in Northern Fulmar (*Fulmarus glacialis*) eggs from the Faroe Islands

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Abstract

Eggs from Northern Fulmars (*Fulmarus glacialis*) were sampled in the Faroe Islands. The content of the brominated flame retardants tri- to decabromodiphenyl ethers (BDEs) and 1,2-bis(2,4,6-tribromophenoxy)ethane (BTBPE) were determined in nine samples in concentrations ranging from non-detectable to 7 ng g^{-1}(l.w.). The BDE levels were similar as in an earlier study of BDE levels in the fulmar eggs from the Faroe Islands but 10–1000 times lower compared to studies of eggs from seabirds and birds of prey from Europe. The two hexaBDEs #153 and #154 were the most abundant congeners, which represented around 50% of the total mean BDE concentration. The levels of BDE #209 were below the limit of detection (1.24 ng g^{-1} l.w.) except for one sample, which showed a concentration of 7.18 ng g^{-1} l.w. BTBPE was detected in eight samples and the mean level was 0.11 ng g^{-1} l.w. This concentration was 150 times lower than the average total BDE concentration (including BDEs #28, #47, #100, #99, #154, #153, #183, #209). BTBPE has only been detected once before in biota. Also other bromo-containing compounds were detected in the fulmar eggs. One group identified was the polybrominated biphenyls (PBBs), but because of the absence of reference standards in the lab, these could not be quantified.

Keywords: Fulmar; Egg; PBDEs; BTBPE

1. Introduction

The Northern fulmars, *Fulmarus glacialis*, have been the object for studies investigating the occurrence of organohalogen pollutants in the environment (Braune et al., 2001; Ericson et al., 2005; Fängström et al., 2005a). In the Faroe Islands, the fulmar is believed to be well suited as an indicator species on the environmental pollution status in situ because ringing evidence suggests adults do not truly migrate, and therefore the pollutants present in the birds have been picked up in the foraging area in the Faroe Islands (Cramps, 1980). The diet of the Northern fulmars of the Fair Isle (Shetland, UK) and Iceland has been described to consists mainly of fish like sandeel and capelin, but also of crustaceans and squid depending on the season, and in more southerly areas also of fish offal (Phillips et al., 1999). The diet of the fulmars at the Faroe Islands has been studied during recent years in connection with the counting of stomach plastic litter as is done as a means of

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describing the pollutants load in the North Sea (van Franeker et al., 2005), but results are not available yet as the data remain to be analysed (Danielsen pers. commn.).

The burden of brominated flame retardants (BFRs) in the fulmar eggs is not only of interest for surveillance purposes, but also because fulmar and fulmar eggs are part of the Faroese subsistence diet and may thus, along with the pilot whale, be a substantial source of lipid soluble, persistant pollutants to the human population. The population of fulmars in the Faroe Islands is estimated to be 800 000 pairs (Skov et al., 2001).

PBDEs are used as additive flame retardants in textiles and various plastic polymers such as polyurethane foam, polyesters, acrylonitril butadiene styrene (ABS), etc. (IPCS, 1994, 1997). The total production of PBDEs was 67 390 metric tons in 2001 (BSEF, 2005), of which the commercial decaBDE-mixture consisted of 56 100 metric tons. The $\log K_{ow}$ for the PBDEs is between 4 to 10 (increasing value with increasing bromination), which means they are lipophilic and subject to bioaccumulation due to their stable structure. PBDEs have been found in sediment, sewage sludge, air, fish, marine mammals, birds and humans and the levels have been summarised in Hites (2004) and Watanabe and Sakai (2003). Of the three technical PBDE mixtures used as flame retardants, the pentaBDE mixture needs the comparably lowest dose to cause toxicological effects and the decaBDE mixture the highest dose to cause effects. The three mixtures give rise to different effects and a review written by Darnerud (2003) summarises the knowledge in this area. The pentaBDE mixture affects neurobehavioural development and thyroid development in the offspring. The octaBDE product causes fetal toxicity/teratogenicity in rats and rabbits and the decaBDE mixture gives rise to morphological effects in the liver, kidney and thyroid of adult animals.

There is little information available about BTBPE. The total annual world production is around 5000 metric tons (IPCS, 1997) and it is used as an additive flame retardant in thermoplastics, ABS polymer systems, high-impact polystyrene and in polycarbonate coatings (IPCS, 1997). Compared to the PBDEs, the compound is less lipophilic ($\log K_{ow}$ 3.14) and should therefore be expected to bioaccumulate to a lesser extent than the PBDEs. A survey of the tissue disposition and excretion of BTBPE in rat has been performed (Hakk et al., 2004). When BTBPE was administered orally at a single dose more than 94% was excreted in the feces after 72 h. The remaining part of the dose was mainly present in the lipophilic tissues. BTBPE has been found in the air from electronic recycling plants (Sjödin et al., 2001; Pettersson-Julander et al., 2004) and in dust from households (unpublished). Information on BTBPE in biological samples is scarce in the international literature, but because this compound is used as a replacement for PBDEs, levels in the environment are expected to rise.

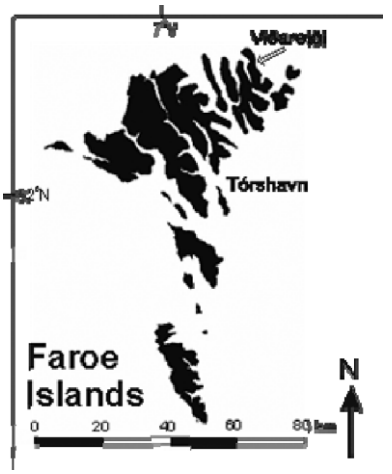

Fig. 1. Map of Faroe Islands indicating the Viðareiði sampling site of fulmar eggs in May 2003 as well as the capital Tórshavn (courtesy of Lis Mortensen at the Faroese Geological Survey).

The aim of this study was to determine the content of some brominated flame retardants, e.g. tri- to decabromodiphenyl ethers (BDEs) and 1,2-bis(2,4,6-tribromophenoxy)ethane (BTBPE) in the eggs from Northern fulmars sampled at the Faroe Islands.

2. Experimental

2.1. Materials and chemicals

Potassium hydroxide (reagent grade) was supplied by Scharlau (Barcelona, Spain). Dichloromethane, toluene and *n*-hexane were all purchased from Riedel de Haën (Envisolv; Seelze, Germany). Sulfuric acid (95–97% p.a.) was from Merck (Darmstadt, Germany). Sodium sulfate (p.a) and *n*-tetradecane (olefine free, >99.5%) was from Fluka (Steinheim, Germany). Nitrogen and helium were from Scangas (Quality 4.5, 99.995% and Quality 5.5, 99.9995%, respectively; Stockholm, Sweden). Methane was obtained from AGA (Quality 4.5, 99.995%). The silica used was purchased from Aldrich (70–230 mesh, grade 60; Steinheim, Germany).

All standards except ^{13}C-labeled BDE #77 which was purchased from the Cambridge Isotope Laboratories (Andover, Massachusetts, USA), were obtained from the Wellington Laboratories (Guelph, Ontario, USA).

2.2. Sampling

Nine eggs were collected from the nests of Northern fulmars in May 2003 in Viðareiði, Faroe Islands (Fig. 1). The egg content was transferred to heat treated (450 °C for 4 h) glass jars and homogenised using a fork. Thereafter the contents were covered with likewise heat treated aluminum foil and a screw cap. The homogenized eggs were stored at −20 °C until extraction.

2.3. Extraction and clean-up

A portion of 20 g containing both egg white and yolk was ground with anhydrous sodium sulfate. Blank samples containing only sodium sulfate were extracted and handled in the same way as the egg samples. The homogenates were put into the glass columns and spiked with internal standards (^{13}C-labeled BDE #77, #139, #209 and BTBPE). Afterwards the lipids were eluted with *n*-hexane/dichloromethane, 1:1. The lipid content was determined gravimetrically by evaporating the extracts to dryness in a rotary evaporator.

Lipids and other interferences were removed by applying the extracts to mixed layer silica columns containing neutral, basic- and acid-treated silica gel. The extracts were dissolved in *n*-hexane and elution was performed with *n*-hexane. Evaporation was done using a rotary evaporator until around 0.5 ml solvent remained. For an additional cleaning the extracts were put onto small silica columns containing basic- and acid-treated silica gel and the analytes were eluted with *n*-hexane. *N*-tetradecane (30 μl) was added and the extracts were evaporated under a gentle stream of N_2-gas until no *n*-hexane was left. The extracts were finally transferred to GC-vials containing the recovery standard (^{13}C-labeled PCB #202) and were left to evaporate to a final volume of about 30 μl *n*-tetradecane.

To be able to determine the tri- to heptaBDE concentrations, an extra cleanup step using gel permeation chromatography (GPC) was performed. This method has been reported in detail elsewhere (Strandberg et al., 1997). The extracts at a volume of about 30 μl in *n*-tetradecane where introduced onto a GPC-system. The mobile phase consisted of *n*-hexane/dichloromethane (65:35, v/v). The eluates were collected in 10 min fractions and the analytes eluted in fractions of four and five. These two fractions were pooled and 25 μl *n*-tetradecane was added. The extracts were reduced to the final volume of 25 μl *n*-tetradecane and transferred to GC–MS vials.

2.4. GC–MS analysis

All samples were analysed using a gas chromatograph and detection was made with a mass spectrometer in negative chemical ionisation (NCI) mode using methane as the reagent gas. For tri- to heptaBDE, a DB-5MS capillary column (J and W Scientific, Folsom, USA) was used (30 m × 0.25 mm ID, 0.25 μm film thickness). For the analysis of BDE #209 and BTBPE, an Equity-5 column (Supelco, Bellefonte, USA) was used (10 m × 0.25 mm ID, 0.25 μm film thickness).

Detection was made in the single ion monitoring (SIM) mode using m/z 79 and 81 for tri- to heptaBDEs, m/z 484.7, 486.7, 494.7 and 496.7 for ^{12}C-and ^{13}C-decaBDE, m/z 249.8, 251.8, 257.8 and 259.8 for ^{12}C- and ^{13}C -BTBPE, m/z 442 for ^{13}C-PCB #202.

2.5. QA/QC

The recoveries for the ^{13}C-labeled internal standards BDE #77, #139, #209 and BTBPE were in the range of 65–138%, 85–138%, 8–150% and 50–166%, respectively. The laboratory participates on a regular basis in international QA/QC studies with good results. This includes studies specifically on PBDEs, unfortunately QA/QC studies on the newly discovered BTBPE are not available yet. During extraction all glassware was wrapped with aluminum foil to minimize UV-degradation of the analytes. The signal ratio between the two ions determined for every analyte and internal standard was within 15% of the ratio found in the quantification standard. Limit of detection (LOD) was set as the limit of quantification (LOQ) and was determined as three times the blank amount. The LODs for the different BFRs are given in Table 1. For sample levels below LOD, the LOD/2 values (Hornung and Reed, 1990) were used when the mean concentrations were calculated.

3. Results and discussion

3.1. PBDEs

The levels of PBDEs (Table 1) were in the same range as earlier published results of Northern fulmar eggs sampled on the Faroe Islands in 2000 and 2001 (Fängström et al., 2005a). All congeners analysed were detected in all samples, though some concentrations were below LOD (Table 1).

Polybromobiphenyl (PBB) #153 coeluted with PBDE #154 on the GC column (Herzke et al., 2005) and these concentrations are therefore reported together as one value (assuming the same detector response for

Table 1
Concentrations in ng g^{-1} (l.w.) of brominated flame retardants detected in Northern fulmar eggs

Sample no.	1	2	3	4	5	6	7	8	9	Mean	LOD[a]
BDE #28	<0.13	<0.13	<0.13	<0.13	<0.13	<0.13	<0.13	<0.13	<0.13	0.07	0.13
BDE #47	1.79	<0.78	2.07	<0.78	2.58	2.37	1.67	3.09	1.89	1.80	0.78
BDE #100	0.80	1.73	0.95	0.56	0.67	0.81	0.62	1.50	0.54	0.91	0.28
BDE #99	3.16	3.92	2.66	1.99	4.51	2.70	2.20	3.61	3.09	3.09	1.16
BDE #154+BB #153[b]	9.43	29.55	9.4	15.13	5.51	16.86	7.89	10.67	7.37	12.43	0.53
BDE #153	3.56	7.04	3.07	7.41	3.97	6.01	3.97	4.99	4.22	4.92	0.39
BDE #183	0.37	0.49	<0.32	0.43	0.87	0.62	<0.32	<0.32	<0.32	0.38	0.32
BDE #209	<1.24	<1.24	<1.24	<1.24[c]	<1.24	<1.24	<1.24	<1.24	7.18	1.35	1.24
BTBPE	0.11	0.11	0.17	<0.02	0.10	0.12	0.15	0.14	0.06	0.11	0.02

[a] Limit of detection was set to 3× Blank concentration.
[b] BDE #154 and BB #153 coeluted in the GC analysis.
[c] Low recovery (8%) of the internal standard ^{13}C-BDE #209.

PBB #153 as for PBDE #154). The coelution was confirmed by reanalysing a sample in electron impact (EI) mode where the compounds were detected separately by their different molecular masses. Fängström et al. (2005a) determined the levels of PBDE #154 and PBB #153 separately in the fulmar egg samples with a c(PBB #153)/c(PBDE #154) ratio (where *c* is the concentration) close to two. With the assumption that the above ratio is valid also in this case, the mean concentration for BDE #154 in this study is expected to be around 4 ng g^{-1} (l.w.). Thus, together with BDE #153 (4.92 ng g^{-1} l.w.) the hexaBDEs constitutes 54% of the total BDE concentration. The third most abundant congener was BDE #99 with a mean concentration of 3.09 ng g^{-1} l.w. The levels of BDE #99, #154 and #153 is only slightly different from the one reported in the fulmar eggs by Fängström et al. (2005a), where these congeners were present at almost equal amounts (4.6, 5.0 and 5.0 ng g^{-1} l.w., respectively). The mean level of BDE #47 was in this study 1.8 ng g^{-1} l.w., whereas Fängström et al. (2005a) reported a level at 4.3 ng g^{-1}l.w. The contribution of this congener to the sumBDE concentration (including BDE #47, #100, #99, #154, #153 and #209, the congeners that were determined in both studies) were lower in this study (11%) than in the Fängström study (21%). The contribution of BDE #153, on the other hand, was higher in this study (29%) than in the Fängström study (24%). The fact that BDE #153 was more abundant than BDE #47 (4.9 and 1.8 ng g^{-1} l.w., respectively) is unusual, although exceptions exist (De Filip et al., 2003; Nagayama et al., 2001; Fängström et al., 2005a,b).

The mass distribution of tri- to heptaBDEs is compared with the distribution in the technical pentaBDE mixture Bromkal 70-5DE (Sjödin et al., 1998) in Fig. 2. The fulmar eggs have a larger contribution of the hexaBDEs #154 and #153 than the technical pentaBDE mixture, which indicates exposure from an octaBDE mixture that contains penta- to nonaBDEs (Ikonomou et al., 2002a). The possibility of selective uptake of the different BDE congeners in fulmars can however not be ruled out.The BDE levels in our study were 10–1 000 times lower compared to the studies of the concentrations in eggs from seabirds from areas closer to denser

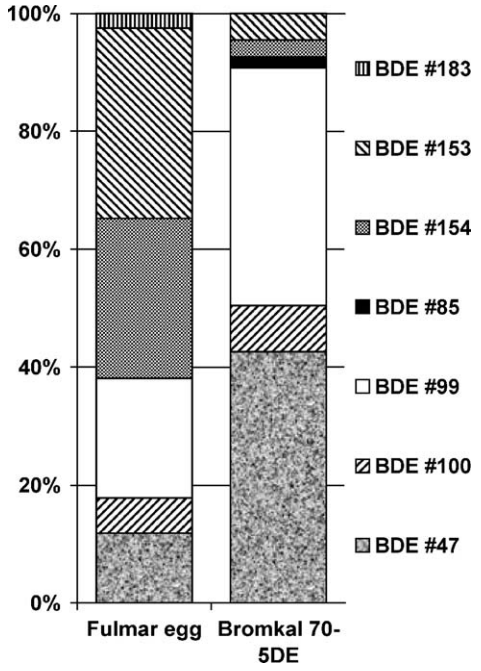

Fig. 2. Distribution (%) of the mean concentrations of tri- to heptaBDEs in the fulmar eggs and in the technical pentaBDE mixture Bromkal 70-5DE. Congeners below 1% are excluded. Concentration of BDE #154 is estimated at 4 ng g^{-1} (l.w.) in the fulmar egg.

population and industrial activity (Sellström et al., 1993; Lundstedt-Enkel et al., 2001; She et al., 2003), or birds of prey (Lindberg et al., 2004; Herzke et al., 2005; Jaspers et al., 2005). The sampling location seems to have more influence on the levels than the specific bird species. No clear difference between BFR levels in seabirds and birds of prey has been described in the literature. Our results therefore implies that the Faroe Islands should be considered to be a relatively low contaminated location with regard to the polybrominated diphenyl ethers in birds.

BDE #209 was found in only one sample at a concentration of 7 ng g^{-1} (l.w.), which is somewhat surprising because the other BFR levels did not differ substantially between the samples (Table 1). Of the three procedural blanks analysed together with the samples, the one with the highest BDE #209 content represented 6% of this sample level. Also in Fängström et al. (2005a), one fulmar muscle sample contained relatively high levels of BDE #209 (62 ng g^{-1} l.w.).The level found in one fulmar egg sample in this study was however low compared to the level found in the peregrine falcon egg in Sweden, which showed mean concentrations of 130 ng g^{-1} l.w. (southern Sweden) and 110 ng g^{-1} l.w. (northern Sweden), respectively of BDE #209 (Lindberg et al., 2004). The distribution of BDE #209 to the egg is currently unknown, but the congener in rat has been shown to be distributed to the plasma and well-perfused tissues such as the liver more readily than the fat tissues (Sandholm, 2003).

In addition to the PBDEs other organic bromocompounds were also detected in the samples. Two unknown peaks eluting just before BDE #47 and #100 respectively, were in a GC/MS analysis in the electron impact (EI) mode detected at m/z 469.7 and 549.6. These ions are consistent with the molecular masses for a tetraBB and a pentaBB, respectively. In a study of fat tissue from young fulmars sampled in the same area in 1999 comprising the five PBB congeners #15, #49, #52, #101 and #153, only two congeners could be detected (Vorkamp et al., 2004). The hexaBB #153 was found in concentrations of approximately 20 ng g^{-1} l.w., whereas the detectable tetraBB isomer #49 was quantified to be 0.37 ng g^{-1} l.w. in the young males. Herzke et al. (2005) reported the presence of di- to heptaBBs in Norwegian eggs of birds of prey. The major congener was PBB #153 and the sum of the PBBs was around 10 times lower than the sum of the PBDEs.

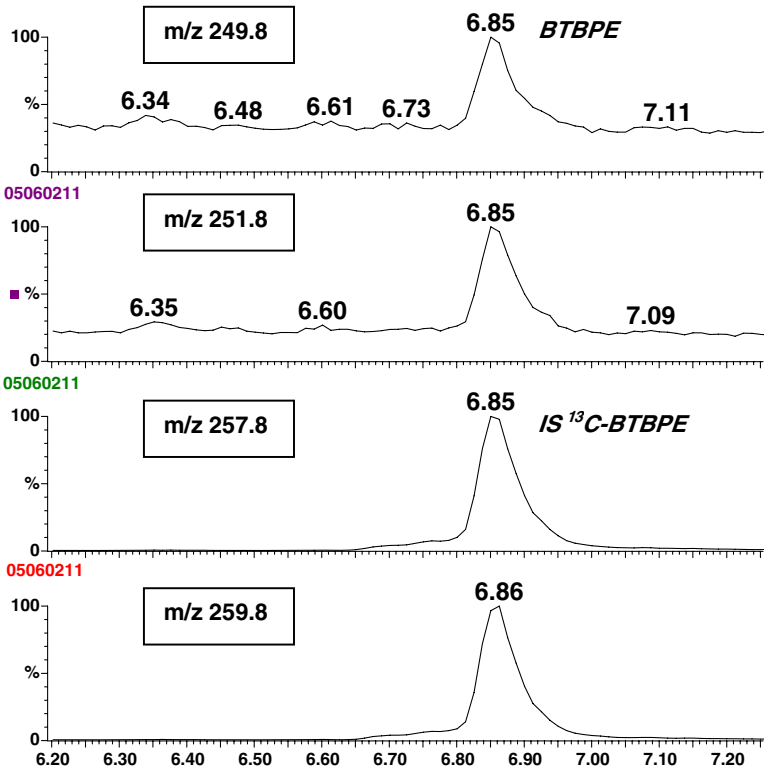

Fig. 3. Mass chromatograms of the GC/MS analysis of BTBPE in sample no. 1. Congeners are marked in the m/z windows used for quantification.

3.2. BTBPE

The analysis of BTBPE is shown in Fig. 3 and is illustrated by m/z 249.8, 251.8 (^{12}C-BTBPE), 257.8 and 259.8 (IS ^{13}C-BTBPE). No overlap was found between the ion clusters for the ^{12}C- and ^{13}C-isomers. The compound was present in all samples except one and the mean concentration was 0.11 ng g^{-1} l.w. (Table 1). This is the second report of this flame retardant in biota. Law et al. (2005) detected BTBPE in fish from Lake Winnipeg, Canada at a maximum concentration of 1.3 ng g^{-1} l.w.

Because the amounts of PBDEs were low in these eggs in relation to other studies, and because the reported amounts of BTBPE in fish are 10 times higher than in the fulmar eggs, one can suspect that the BTBPE levels in fulmars are relatively low compared to levels in other biota from other locations. On the other hand, levels of PCBs and dioxin concentrations are high in the fulmar eggs (Ericson et al., 2005; Fängström et al., 2005a). The low levels of BTBPE may be due to the fact that this compound class has been taken into use more recently, and hitherto in lower volumes.

According to Ikonomou et al. (2002b), PBDEs have been produced in large-scale since the early 1970s, while BTBPE has been on the market in at least 25 years (New materials international, 2005). Up to now the production of BTBPE has been about ten times lower than the total PBDE production. However, the distribution in the fulmar eggs was different with a mean BTBPE concentration 150 times lower than the average total BDE concentration (the sum of eight congeners determined). This fact may mirror the lower lipophilicity of BTBPE.

The three brominated flame retardant producers ICL Industrial Products (former Dead Sea Bromine Group), Great Lakes Chemical Corporation and Albemarle Corporation (representing 76% of the total manufacturing of brominated flame retardants (Swedish society for Nature conservation, 2005)) have ceased to produce the commercial pentaBDE and octaBDE products in the end of 2004 (ICL Industrial Products, 2005; Albemarle corporation, 2005; Great Lakes Chemical Corporation, 2005). This is done in compliance with the EU Directive 2003/11/EC that bans the marketing of the two mixtures in Europe by the end of 2004. BTBPE is used as a replacement of the octaBDE mixture (New materials international, 2005). A higher production of BTBPE is consequently expected and may lead to increasing levels of this flame retardant in the environment in the future. It is therefore of importance to further investigate the occurrence of BTBPE in the environment and if levels are increasing over time.

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