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Organohalogen contaminants and vitamins in Northern Fur Seals (*Callorhinus ursinus*) collected during subsistence hunts in Alaska

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Abstract

During native subsistence hunts from 1987 to 2007, blubber and liver samples from 50 subadult male northern fur seals (*Callorhinus ursinus*) were collected on St. Paul Island, Alaska. Samples were analyzed for legacy persistent organic pollutants (POPs), recently phased out/current-use POPs, and vitamins. The legacy POPs measured from blubber samples included polychlorinated biphenyl congeners, dichlorodiphenyltrichloroethanes (DDT and metabolites), chlorobenzenes, chlordanes, and mirex. Recently phased-out/current-use POPs included in the blubber analysis were the flame retardants, polybrominated diphenyl ethers, and hexabromocyclododecanes. The chemical surfactants, perfluorinated alkyl acids and vitamins A and E were assessed in the liver samples. Overall, concentrations of legacy POPs are similar to levels seen in seal samples from other areas of the North Pacific Ocean and the Bering Sea. Statistically significant correlations were seen between compounds with similar functions (pesticides, flame retardants, vitamins). With sample collection spanning two decades, the temporal trends in the concentrations of POPs and vitamins were assessed. For these animals, the concentrations of the legacy POPs tend to decrease or stay the same with sampling year; however, the concentrations of the current-use POPs increased with sampling year. Vitamin concentrations tended to stay the same across the sampling years. With the population of northern fur seals from St. Paul Island on the decline, a detailed

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Compliance with Ethical Standards

Conflict of Interest: The authors declare that they have no conflict of interest.

assessment of exposure to contaminants and the correlations with vitamins fills a critical gap for identifying potential population risk factors that might be associated with health effects.

Introduction

Pinnipeds, fin-footed marine mammals, occupy a high trophic level in the marine environment, making them susceptible to the bioaccumulation of chemical contaminants such as persistent organic pollutants (POPs). Exposure to legacy POPs (including polychlorinated biphenyls (PCBs) and chlorinated pesticides) has been associated with health problems, including immunotoxicity and reproductive impairments in pinnipeds (Beckmen et al. 1999; Hutchinson and Simmonds 1994; Tabuchi et al. 2006). Many of the potential health problems resulting from long-term exposure to legacy POPs are not fully understood in wild populations of pinnipeds (Towell et al. 2006). An assessment of the POPs burden in these animals over the decades may be important to help understand trends in past and current levels of POPs since pinniped populations have recently exhibited a low reproduction rate and POPs concentrations may be associated with this rate (Towell et al. 2006). The Arctic has been shown to be a major sink for a variety of legacy POPs, although the levels of POPs in the Arctic vary depending on the environmental matrix (Muir et al. 1992). While many legacy POPs have been banned from use for decades, in general they have been slow to decline in top predators, especially mammals because of their persistent nature especially in the Arctic (Muir et al. 1992; Riget et al. 2010).

In addition to legacy POPs, pinnipeds are exposed to currently-used (and recently banned) pollutants, including polybrominated diphenyl ethers (PBDEs), hexabromocyclododecanes (HBCDs), and perfluorinated alkyl acids (PFAAs). PBDEs were extensively used as flame retardants in industrial and commercial applications (Darnerud et al. 2001). With the phase out of many PBDE commercial mixtures in Europe and the United States starting in 2006, HBCDs were considered a likely replacement, and they are often used in buildings and upholstery (Covaci et al. 2006). PFAAs were globally used for many applications, mainly as a surface protector for furniture, carpets, paper, and packaging (Prevedouros et al. 2006). PBDEs, HBCDs, and PFAAs are similar to legacy POPs because they are known to be persistent in the environment and have bioaccumulative properties. Many of these current-use POPs have been shown to accumulate in the arctic marine environment (Braune et al. 2005; Butt et al. 2010; Hart et al. 2009; Muir et al. 1992; Smithwick et al. 2006; Tomy et al. 2009; Verreault et al. 2005).

One group of pinnipeds, the northern fur seals (*Callorhinus ursinus*), are otariid (externally eared) seals that have a wide geographical range throughout the North Pacific Ocean extending from Japan, eastward to Northern California, and northward to the Bering Sea (Gentry 1997). While their habitat is not completely Arctic, in their northern range, northern fur seal habit overlaps with arctic pinnipeds such as the ringed seal (*Phoca hispida*). The seals are philopatric, having well established breeding colonies at several locations in the Bering Sea including the Pribilof Islands, Alaska. Within the Pribilof Islands, the animals using St. Paul Island have seen a pupping decline of nearly three-fold since the early 1970s (Towell et al. 2006). Hypotheses for the cause of the decline include climate changes,

commercial fisheries interactions, and/or predation (Towell et al. 2006). Chemical contamination is another suspected cause for the declining population; however, there have been few studies looking at chemical contaminants in northern fur seals from St. Paul Island (Wang et al. 2010). These studies focus primarily on legacy POPs and have not provided information about current-use POPs in the population.

A potential indicator of population decline could be the reduction or disruption of vitamin homeostasis in northern fur seals. Alterations in vitamin levels are a potential biomarker of exposure to legacy POPs. A possible link between exposure to PCBs and chlorinated pesticides with decrease levels of circulating retinol (vitamin A) and hepatic vitamin E has been observed in beluga whales, ringed seals, grey seals, and harbor seals (Desforges et al. 2013; Kakela et al., 1999; Nyman et al., 2003; Routti et al., 2005; Simms et al. 2000). It has been hypothesized that changes in vitamin A, essential in growth and development, may in turn correlate with population changes (Simms and Ross 2000). Biological factors, including size and reproductive status, may affect vitamin biomarkers (Loseto et al. 2009). However, it is difficult to understand what changes in vitamin levels elicit effects in wild populations because there is a lack of information on the concentrations of vitamins in wildlife, specifically northern fur seals.

The primary aim of this study was to measure concentrations of legacy POPs (PCBs and chlorinated pesticides), recently phased out/current-use POPs (PBDEs, HBCDs, and PFAAs), and vitamins (A and E) in northern fur seal blubber and liver tissue collected from four rookeries on St. Paul Island between 1987 and 2007. In order to assess contaminant-related issues in pinnipeds, an understanding of their exposure to the myriad of POPs and health status markers is critical. Since factors such as age, weight, sex, and body condition, can influence the health and accumulation of POPs, we specifically looked at subadult (2–3 years of age) males for this study. Secondly, since previous studies have hypothesized vitamin levels potentially correlate with POPs concentrations, we examine correlations among different POPs classes and vitamins. Thirdly, the temporal trends of POPs and vitamins were assessed in the samples (from 1987 to 2007), and a comparison among the legacy POPs, recently phased out/current-use POPs, and vitamin concentrations spanning the 20 year time period was performed. With the population of northern fur seals from St. Paul Island on the decline, a detailed assessment of exposure to contaminants, the information for recently phased out/current-use POPs and vitamins fills an important data gap which helps to put in context POPs exposure with other risks affecting this population.

Materials and Methods

Sample Collection and Preparation

Full-depth blubber (n=50) and liver (n=49) tissue samples from northern fur seals were collected as part of the Alaska Marine Mammal Tissue Archival Project (AMMTAP). The standardized AMMTAP protocols have been described previously (Becker et al. 1993). Briefly, samples were collected during native subsistence hunts from subadult males (2–3 years of age) from three rookeries on St. Paul Island, Alaska, from 1987 to 2007 (Supplemental Information, Table S1). The blubber and liver tissue samples were processed in the field using established protocols designed to minimize sample contamination (Becker

and Wise 2006). Then samples were placed in liquid nitrogen vapor-phase shippers and sent to the National Institute of Standards and Technology's (NIST) Marine Environmental Specimen Bank (Marine ESB) for storage. All samples were stored in liquid nitrogen vapor-phase freezers (−150° C) in a clean room environment prior to further processing and analysis. Once the samples were chosen for analysis, each blubber and liver tissue was cryogenically homogenized using methods described elsewhere in Zeisler, et al. (1983) and Pugh et al. (2007). This process resulted in approximately 20 to 25 aliquots (i.e. sub-samples) of a fresh, frozen powder homogenate, approximately 5g to 6g each.

Analytical Methods

Persistent Organic Pollutants (POPs)—The extraction methods have been previously explained in detail in Bachman et al. (2014). Briefly, blubber samples were mixed with sodium sulfate and transferred to pressurized fluid extraction (PFE) cells for extraction. The internal standard solution was added to the samples, and then samples were extracted with dichloromethane using the PFE. Extracted samples were subsequently cleaned up using size exclusion chromatography, followed by solid phase extraction using acidified silica to fractionate the HBCDs (Bachman et al. 2014). PCBs, pesticides, and PBDEs were quantified by gas chromatography with mass spectrometric detection (GC-MSD) operated in either the electron impact mode or the negative chemical ionization mode with selected ion monitoring. HBCDs were quantified using liquid chromatography tandem mass spectrometry detection (LC-MS/MS) in the multiple reaction monitoring (MRM) mode. Details of the instrumental methods can be found in Kucklick et al. (2013).

Perfluorinated Alkyl Acids (PFAAs)—Extraction and cleanup methods for PFAAs have been previously described in Reiner et al. (2012). Briefly, liver samples were extracted using basic methanol, filtered, and further cleaned using graphitized non-porous carbon solid phase extraction. PFAAs were determined using LC-MS/MS in the MRM mode. Further details of the extraction, cleanup, and instrumental methods can be found in Reiner et al. (2012).

Vitamins—Details of the extraction and cleanup for vitamin analysis can be found in Kucklick et al. (2013). Liver samples were digested using a potassium hydroxide heated (40 °C) extraction method. After extraction, a liquid-liquid extraction was performed three times using a hexane:petroleum ether mixture (1:1 volume fraction). The organic layer was removed, evaporated to dryness, and reconstituted in a 9:1 (volume fraction) ethanol: ethyl acetate mixture. All steps were performed in a room with subdued lighting, and when possible, the samples were fully shielded from the light. Instrumental analysis was performed using LC with ultraviolet/fluorescence detection.

Quality Control

Blanks, calibration solutions, NIST Standard Reference Materials (SRMs), and quality control materials were processed alongside the blubber and liver samples. SRM 1945 Organics in Whale Blubber was used as the control material in blubber sample analyses, and QC97LH2 Beluga Whale Liver Homogenate and SRM 1946 Lake Superior Fish Tissue were used as the control materials for the liver sample analyses. To assess if methods were in

control, the POPs measured in SRMs 1945, 1946, and QC97LH2 had to agree with the certified and reference values provided on the Certificates of Analysis. A compound was considered to be significantly above the reporting limit (RL) if the mass of an analyte in the sample was greater than the mean plus three standard deviations of all blanks.

Statistical Methods

Statistical analyses were performed on lipid-normalized data (ng/g lipid mass) for the sums of 64 PCB congeners (8, 18, 28, 31, 44, 49, 52, 56, 66, 74, 79, 87, 92, 95, 99, 101, 105, 110, 112, 114, 118, 119, 121, 127, 128, 132, 137, 138, 146, 149, 151, 153, 156, 158, 157, 159, 163, 167, 170, 172, 174, 175, 176, 177, 178, 180, 183, 185, 187, 191, 193, 194, 195, 196, 197, 199, 200, 201, 202, 205, 206, 207, 208, and 209), six DDT-related compounds (2,4'- and 4,4'-DDE, DDD, and DDT), five chlordanes (oxychlordane, *trans*-chlordane, *cis*-chlordane, *trans*-nonachlor, and *cis*-nonachlor), three hexachlorocyclohexanes (α-, β-, and γ-HCH), pentachlorobenzene, hexachlorobenzene, mirex, six PBDE congeners (47, 99, 100, 153, 154, and 155), and α-HBCD, on wet mass data (ng/g wet mass) for 15 PFAAs (PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnA, PFDoDA, PFTriA, PFTA, PFBS, PFHxS, PFOS, and PFOSA), and on wet mass data (μg/g wet mass) for vitamin A (retinol) and two vitamin E compounds (α- and γ-tocopherol). Values below the RL were imputed as the limit of quantitation divided by square root of two.

All statistical analyses were performed in Stata/SE 11.2 (more details can be found in the SI). Spearman rank correlation coefficients, or Spearman's rho (ρ), were run to determine statistical dependence among the analytes. Proportionate percentile parametric quantile regression models (Cox et al. 2007; Pierce et al. 2011; Gribble et al. 2013) were used to summarize the percent change in chemical level per year over 1987–2007 in the seal population of St. Paul Island as a linear function of year. In proportionate percentile models, the percent change 'trends' for the median are the same percent change 'trends' for all other quantiles of the chemical distribution. Clustering of seals within rookeries was acknowledged by cluster-robust standard errors. Most models assumed lognormal distributions, except for the vitamin E model which was Weibull.

Results and Discussion

Most classes of POPs and vitamins were detected in each of the fur seal samples (Table 1). The exceptions were mirex and α-HBCD, which were detected in 68 % and 96 % of the samples, respectively. Examining the legacy POPs in blubber tissue, the concentrations of Σ_{64} PCB and Σ_6 DDT made up more than 70 % of the total POPs measured in the northern fur seal samples. The Σ_{64} PCB median concentration was 2130 ng/g lipid mass and the Σ_6 DDT median concentration was 1240 ng/g lipid mass. Of the 64 PCBs determined in the northern fur seal samples, concentrations of hexa- and heptachlorobiphenyl, especially PCBs 138, 153, and 180 were found in higher amounts (over 68 % of the total PCB burden) compared to lower chlorinated (tetra- and pentachlorobiphenyl; 27 % of the total PCB burden) PCB homologs (Supplemental Information, Figure S1). The most persistent DDT metabolite, 4,4'-DDE was the dominant DDT compound detected in the northern fur seal blubber samples, making up more than 70 % of the Σ_6 DDTs in the samples. Detected in much lower amounts, Σ_5 Chlordane median concentration was 652 ng/g lipid mass. *Trans*-

nonachlor, one of the major constituents of the insecticide chlordane, was the highest detected of the Σ_5 Chlordanes measured in the northern fur seals. Σ_3 HCH median concentration of 331 ng/g lipid mass, pentacholorobenzene median concentration was 3.59 ng/g lipid mass, hexachlorobenzene median concentration was 0.775 ng/g lipid mass, and mirex median concentration was 12.6 ng/g lipid mass.

The recently phased out/current-use POPs were detected in concentrations were much lower in concentrations (less than 0.5 % of the total POPs measured) compared to Σ_{64} PCB and Σ_6 DDT concentrations measured in this study. The median concentration in blubber for Σ_6 PBDEs in fur seal blubber samples was 26.3 ng/g lipid mass, with a significant contribution of the total coming from BDE 47 (median concentration of BDE 47 16.0 ng/g lipid mass, average percent of total PBDEs 61 %). The only HBCD isomer detected in blubber tissue was α-HBCD with a median concentration of 2.49 ng/g lipid mass. The median hepatic concentration for Σ_{15} PFAAs was 37.0 ng/g wet mass, with significant contribution to the total coming from six of the longer chain perfluorocarboxcyclic acids (Σ_6 PFCAs, median concentration 29.1 ng/g wet mass), especially the odd chain PFCAs perfluorononanic acid (PFNA), perfluoroundecanoic acid (PFUnA), and perfluorotridecanoic acid (PFTriA). One of the most frequently detected PFAA, perfluorooctane sulfonate (PFOS) was found at lower concentrations compared to the long-chain PFCAs (Supplemental Information, Table S2 and Figure S2).

In this study, vitamins A and E were determined in northern fur seal liver samples since in some studies the concentrations measured in blubber, plasma, and liver tissues have been shown to be affected by POP's exposure in other marine mammal species (Kakela et al. 1999; Nyman et al. 2003; Routti et al. 2005; Desforges et al. 2013). To the authors' knowledge this is the first time vitamins A and E have been examined in wild northern fur seals. The concentrations of vitamin A (in the form of retinol) ranged from 4.8 μg/g wet mass to 671 μg/g wet mass (median concentration of 59.9 μg/g wet mass). This concentration is within the same range as hepatic concentrations of vitamin A seen in ringed seals, but lower than hepatic concentrations of retinol measured in male juvenile bowhead whales (Kakela et al. 1999; Rosa et al. 2007). The concentration of vitamin E (in the form of α-tocopherol and γ-tocopherol) ranged from <RL to 24100 μg/g wet mass (median concentration of 11300 μg/g wet mass), with the majority of vitamin E quantified being in the form of α-tocopherol. In comparison to vitamin E concentrations measured in ringed seals and bowhead whales, the concentrations measured in this study are higher (Kakela et al. 1999; Rosa et al. 2007).

In order to understand if POPs exposures come from common routes, it is important to examine relationships among the different classes of compounds. There were strong correlations among classes of compounds; many of these pairwise correlations were significant ($p<0.05$; Table 2). Some of the correlations correspond to similar commercial uses (i.e. pesticides, flame retardants) so the correlations among these chemicals may be partly explained by common routes of application or use. The concentration of Σ_{64} PCBs were positively correlated with most the concentrations of most legacy POPs, including Σ_6 DDTs, Σ_5 Chlordanes, Σ_3 HCHs, pentachlorobenzene, and mirex ($\rho_{PCBs/DDT} = 0.3$, $\rho_{PCBs/chlordanes} = 0.7$, $\rho_{PCBs/HCHs} = 0.6$, $\rho_{PCBs/pentachlorobenzene} = 0.4$, $\rho_{PCBs/mirex} = 0.7$). Σ_{64}

PCBs was negatively correlated with hexachlorobenzene ($\rho_{PCBs/hexachlorobenzene} = -0.5$), suggesting there are differences in exposure and/or metabolism of these compounds. Similarly, the concentrations of Σ_6 DDTs was positively correlated with the concentrations of Σ_5 Chlordane ($\rho_{DDTs/chlordanes} = 0.8$), Σ_3 HCH ($\rho_{DDTs/HCH} = 0.5$), pentachlorobenzene ($\rho_{DDTs/pentachlorobenzene} = 0.6$), and mirex ($\rho_{DDTs/mirex} = 0.4$), the concentrations of Σ_5 Chlordane was positively correlated with the concentrations of Σ_3 HCH ($\rho_{chlordanes/HCH} = 0.8$), pentachlorobenzene ($\rho_{chlordanes/pentachlorobenzene} = 0.6$), and mirex ($\rho_{chlordanes/mirex} = 0.5$), and the concentrations of Σ_3 HCH was correlated with the concentrations of pentachlorobenzene ($\rho_{HCH/pentachlorobenzene} = 0.5$) and mirex ($\rho_{HCH/mirex} = 0.5$). Since most legacy POPs correlate with each other, one can hypothesize there are common routes of exposure for most legacy POPs.

The concentrations of the brominated flame retardants (Σ_6 PBDEs and α-HBCD) were positively correlated with each other ($\rho_{PBDE/\alpha\text{-}HBCD} = 0.5$). Both Σ_6 PBDEs and α-HBCD are recent use flame retardants and their correlation with one another suggests similar sources of exposure. Interestingly, Σ_6 PBDEs correlated positively with Σ_{64} PCBs ($\rho_{PBDEs/PCBs} = 0.3$) and negatively with hexachlorobenzene ($\rho_{PBDEs/hexachlorobenzene} = -0.4$). The concentrations of Σ_{15} PFAAs negatively correlated with concentrations of many legacy POPs, including, Σ_6 DDTs, Σ_5 Chlordane, Σ_3 HCH, and pentachlorobenzene ($\rho_{PFAAs/DDT} = -0.4$, $\rho_{PFAAs/chlordanes} = -0.4$, $\rho_{PFAAs/HCHs} = -0.5$, $\rho_{PFAAs/pentachlorobenzene} = -0.4$), but positively correlated with concentrations of Σ_6 PBDEs and α-HBCD ($\rho_{PFAAs/PBDEs} = 0.4$, $\rho_{PFAAs/\alpha\text{-}HBCD} = 0.4$). Since Σ_{15} PFAAs negatively correlate with all legacy POPs, this suggests there are differences in uptake and metabolism. PFAAs are not lipophilic like most legacy POPs therefore it was not expected to see positive correlations among these classes of compounds. With correlations shown among many POPs classes, it is important to consider exposure of multiple POPs groups when assessing animal health and not just one class of compounds.

There have been few studies done examining the influence of POPs on vitamin A and E levels in marine mammals (Kakela et al. 1999; Nyman et al. 2003; Routti et al. 2005; Desforges et al. 2013). Previous studies have shown a decrease in retinol levels in northern elephant seals (*Mirounga angustirostris*) and harbor seals (*Phoca vitulina*) associated with PCBs and 4, 4'-DDE (Beckmen et al. 1997; Brouwer et al. 1989; De Swart et al. 1994). It has been suggested by Nyman et al. (2003) that elevated levels of vitamin E could potentially be used as a biomarker of exposure to higher POPs loads. The concentrations of the vitamins retinol and α-tocopherol were positively correlated with one another ($\rho_{retinol/\alpha\text{-}tocopherol} = 0.5$); however, vitamins did not correlate with any POPs in this study.

All samples were combined to assess the temporal trends of POPs and vitamins in northern fur seals from St. Paul Island, with the standard errors being corrected for clustering by rookeries (Table 3). The Σ_{64} PCBs, Σ_6 DDTs, and mirex concentrations showed no significant increase or decrease in northern fur seals from 1987 to 2007. When looking at homolog groups for PCBs there is were no significant changes based on homolog group. The concentrations of the other legacy POPs showed a decline in northern fur seals from 1987 to 2007. There was a significant decrease of 5 % per year of Σ_5 Chlordanes and hexachlorobenzene concentrations (95 % CI: −7, −1 and −7, −2, respectively), a 4 %

decrease annually of Σ_3 HCH concentrations (95 % CI: −6, −3), and a 3 % decrease annually of pentachlorobenzene (95 % CI: −4, −2) in the northern fur seal blubber samples (Table 3). The steady state and decrease in legacy POPs, including PCBs and pesticides, has been shown in other marine mammal studies from the Arctic (Hoguet et al. 2013; Lebeuf et al. 2007; Riget et al. 2010).

Unlike the legacy POPs, the recently phased out/current-use POPs showed increases over this 20-year study time frame. Σ_6 PBDE and α-HBCD concentrations both significantly increased from 1987 to 2007 at an average rate of 9 % (95 % CI: +5, +13) and 12 % (95 % CI: +8, +16) annually, respectively (Table 3; Figure 1). Σ_{15} PFAA concentrations increased at an average rate of 9 % (95 % CI: +8, +10) annually (Table 3; Figure 1). The increases in Σ_6 PBDE concentrations are being driven primarily by the increase in BDE 47. The increase in Σ_{15} PFAA is being driven by the increases in the PFCAs and there was no significant change in concentrations of PFOS from 1987 to 2007 (Table 3). The increase of the recently phased out/current-use POPs is not unexpected as other studies have shown increases of the concentrations of PBDEs, α-HBCD, and PFAAs in marine mammals from the Alaskan Arctic (Ikonomou et al. 2002; She et al. 2002; Reiner et al. 2011; Hoguet et al. 2013).

This was the first time vitamins have been measured in wild northern fur seals. Temporally, both vitamins A and E showed no significant changes in the northern fur seal samples from 1987 to 2007. Because of the limited knowledge of vitamin concentrations in wild populations, this vitamin information can potentially be used as baseline vitamin A and E measurements for wild northern fur seals. Although no correlations were shown among vitamins and POPs in northern fur seals, knowing typical vitamin A and E levels expected in wildlife can aid future researchers who may want to use these vitamins as potential biomarkers. Additionally, vitamin A and E concentrations in liver tissue are dependent on diet, so these baseline vitamin concentrations may be used to understand potential changes in the northern fur seals feeding habits.

There are few studies looking at legacy POPs in northern fur seals from the Pribilof Islands (Loughlin et al. 2002; Wang et al. 2010). These studies show similar concentrations of PCBs, DDTs, HCHs, and hexachlorobenzene measured in northern fur seal blubber tissue (Table 4). The recently phased out/current-use POPs have not been previously examined in northern fur seals from the Alaskan Arctic; however, PBDEs have been examined in female northern fur seal blubber samples collected from Japan (Kajiwara et al. 2004). The Kajiwara et al. study and ours showed similar PBDEs concentrations (2004). Although α-HBCD has not previously been measured in northern fur seals, α-HBCD has been measured in California sea lions (*Zalphophus californianus*) blubber samples (Stapleton et al. 2006). The values of α-HBCD reported by Stapleton et al. (2006) tend to be higher in California sea lions. This difference can be explained by proximity of California sea lions to higher ambient sources originating from California watersheds, and subsequently different food web sources. One PFAA, PFOS, has been measured in northern fur seals twice previously (Giesy and Kannan 2001; Kannan et al. 2001). The concentrations of PFOS measured in this study (Supplemental Information, Table S2) are similar to concentrations measured in other northern fur seal liver samples from the Pribilof Islands (Giesy and Kannan 2001; Kannan et al. 2001).

Alaskan natives rely on the annual northern fur seals harvests' from the Pribilof Islands as part of their subsistence diet. It is important from a human health perspective to consider the meat and blubber from the northern fur seals as a potential route of dietary exposure to legacy and current-use POPs. POPs are primarily sequestered in blubber tissue, so the consumption of marine mammal blubbers has been suggested as a source of POPs to Nunavik Inuits (Dallaire et al. 2009; Weihe et al. 2008). It would be rational to assume Alaskan natives who consume subsistence food, such as northern fur seals, are exposed to many POPs, though the health effects of consuming food with levels reported here is unclear.

Conclusions

This study demonstrates that recently banned and current use POPs are increasing in the arctic region, while the legacy POPs are either declining or remaining constant. Similar trends have been seen in arctic species studied. Monitoring recently banned and current use POPs in future studies will be important to see if these POPs begin to decline in the arctic region. This study also gives baseline information about the concentrations of vitamins A and E in northern fur seals. The identification of POPs in this population of northern fur seals can be used to help understand potential risk factors for this declining population and also fills a critical gap in contaminant information for this sub-arctic species. Although the toxicological implications of the concentrations measured in the blubber and liver of northern fur seals are unknown, their potential impact on both northern fur seal health and human health still exists and should be considered.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

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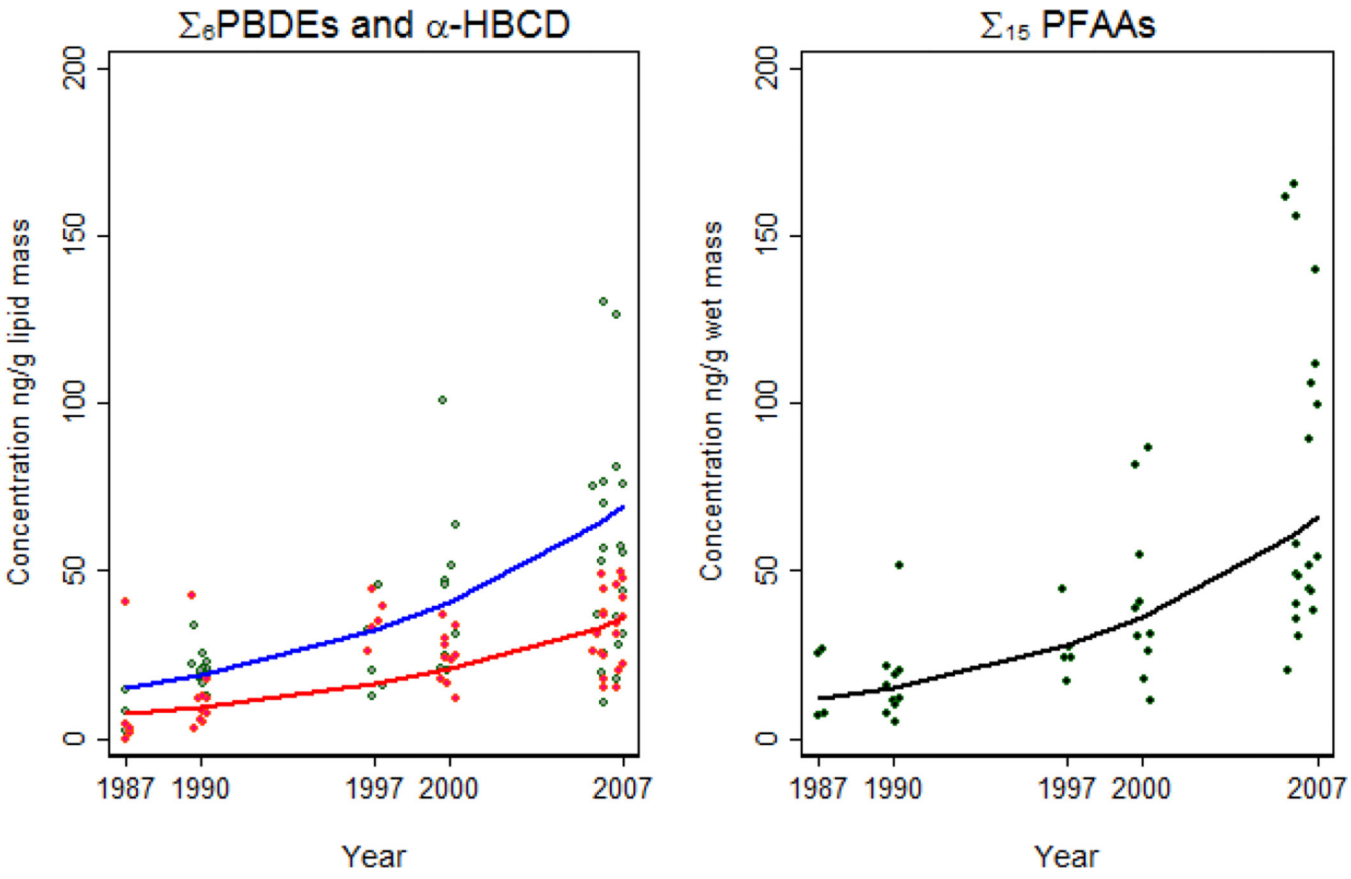

Figure 1.

Temporal concentration trends of Σ_6PBDEs (blue) and α-HBCD (red) in northern fur seal blubber samples and Σ_{15}PFAAs (black) in northern fur seal liver samples.

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Table 1

Concentrations of a) POPs (ng/g lipid mass) in northern fur seal blubber samples and b) PFAAs (ng/g wet mass) and vitamins (μg/g wet mass) in liver samples.

a.	Polavina blubber (n=23)			Northeast blubber (n=2)			Zapadni blubber (n=9)			Reef blubber (n=16)		
	Range	Median	n > RL	Range	Median	n > RL	Range	Median	n > RL	Range	Median	n > RL
Σ_{64}PCBs	923–3650	2110	23	1360–2586	1980	2	1510–2600	1850	9	681–4410	2520	16
Σ_{6}DDTs	297–5720	1170	23	453–1100	775	2	1520–5000	2980	9	287–156000	867	16
Σ_{5}Chlordanes	305–2720	588	23	577–1050	815	2	649–1760	826	9	221–2080	616	16
Σ_{3}HCHs	98.0–725	326	23	420–796	608	2	264–583	438	9	124–736	264	16
Pentachlorobenzene	1.87–8.89	2.74	23	3.41–5.86	4.63	2	2.77–6.53	4.04	9	1.81–8.57	3.85	16
Hexachlorobenzene	<0.135–4.89	0.850	20	1.37–1.59	1.48	2	0.530–3.23	0.780	9	<0.135–2.56	0.723	13
Mirex	<0.0835–107	11.8	14	<0.0847–17.3	8.67	1	7.57–23.0	12.1	9	<0.0682–27.2	18.7	9
Σ_{6}PBDEs	1.46–126	35.1	23	3.33–9.30	6.31	2	11.4–45.1	21.6	9	12.2–132	24.5	16
α-HBCD	<0.0740–15.1	3.02	22	<0.0680–0.513	0.0935	1	0.296–6.99	2.90	9	0.436–30.7	1.99	16

b.	Polavina liver (n=23)			Northeast liver (n=1)			Zapadni liver (n=9)			Reef liver (n=16)		
	Range	Median	n > RL	Range	Median	n > RL	Range	Median	n > RL	Range	Median	n > RL
Σ_{15}PFAAs	6.89–137	42.7	23	27.1	--	1	8.59–45.9	19.6	9	4.86–164	39.4	16
Retinol	4.80–654	134	23	52.4	--	1	11.5–671	43.9	9	11.1–203	45.9	16
α-Tocopherol	<472–23400	11100	20	4790	--	1	<472–15300	12700	8	<472–16700	9820	14
γ-Tocopherol	<573–1310	738	12	657	--	1	<573–1290	618	3	<573–2010	877	12

Abbreviations: POPs=persistent organic pollutants, PCBs=polychlorinated biphenyls, DDTs= dichlorodiphenyltrichloroethanes, HCH= hexachlorocyclohexanes, PBDEs=polybrominated diphenyl ethers, α-HBCD=α-hexabromocyclododecanes, PFAAs=perfluorinated alkyl acids

Values shown as "<" a specified number describe the actual reporting limit (RL)

Table 2

Spearman correlations of POPs and vitamin concentrations in northern fur seal samples. Asterisks (*) indicate there were significant correlations ($p<0.05$).

	Σ_{64}PCBs	Σ_6DDTs	Σ_5Chlordanes	Σ_3HCHs	Pentachlorobenzene	Hexachlorobenzene	Mirex	Σ_6PBDEs	α-HBCD	Σ_{15}PFAAs	Retinol	α-Tocopherol	γ-Tocopherol
Σ_{64}PCBs	-	**0.3***	**0.7***	**0.6***	**0.4***	**−0.5***	**0.7***	**0.3***	−0.08	−0.2	0.1	−0.02	0.1
Σ_6DDTs		-	**0.8***	**0.5***	**0.6***	−0.06	**0.4***	−0.2	−0.06	**−0.4***	0.06	0.07	0.2
Σ_5Chlordanes			-	**0.8***	**0.6***	−0.1	**0.5***	−0.03	0.06	**−0.4***	0.05	0.02	0.2
Σ_3HCHs				-	**0.5***	−0.09	**0.5***	−0.2	−0.2	**−0.5***	0.2	0.002	0.04
Pentachlorobenzene					-	0.05	**0.5***	−0.2	−0.1	**−0.4**	−0.08	0.02	−0.1
Hexachlorobenzene						-	**−0.4***	**−0.4***	−0.2	−0.007	−0.1	0.1	−0.1
Mirex							-	0.09	0.06	−0.1	0.09	0.1	0.02
Σ_6PBDEs								-	**0.5***	**0.4***	−0.06	0.1	0.05
α-HBCD									-	**0.4***	−0.1	0.1	−0.1
Σ_{15}PFAAs										-	−0.06	−0.04	−0.1
Retinol											-	**0.5**	−0.07
α-Tocopherol												-	0.01
γ-Tocopherol													-

Table 3

Temporal trend (% change per year) and 95 % confidence intervals of POPs and vitamins in northern fur seals from St. Paul Island from 1987 to 2007.

Compound	Trend	95 % CI
Σ_{64}PCBs	NS	−3, +1
di- and trichlorobiphenyls	NS	−2, +1
tetrachlorobiphenyls	NS	−3, 0
pentachlorobiphenyls	NS	−5, +1
hexachlorobiphenyls	NS	−3, 0
heptachlorobiphenyls	NS	−3, 0
octachlorobiphenyls	NS	−1, +1
Σ_6DDTs	NS	−17, +2
4,4'-DDT	NS	−25, +2
Σ_5Chlordanes	5 % decrease	−7, −1
Σ_3HCHs	4 % decrease	−6, −3
β-HCH	3 % decrease	−1, −4
Pentachlorobenzene	3 % decrease	−4, −2
Hexachlorobenzene	5 % decrease	−7, −2
Mirex	NS	−21, +4
Σ_6PBDEs	9 % increase	+5, +13
BDE 47	9 % increase	+5, +13
α-HBCD	12 % increase	+8, +16
Σ_{15}PFAAs	9 % increase	+8, +10
PFOS	NS	−2, +16
Retinol	NS	−5, +3
α-Tocopherol	NS	−2, 0
γ-Tocopherol	NS	−2, 0

Abbreviations: NS=no significant trend

Table 4

Comparison of mean concentrations of legacy POPs (ng/g lipid mass) in nortern fur seal blubber samples from the Priblof Islands collected as part of this study and published results.

Pribilof Island Location (Year; sample size)	ΣPCBs	ΣDDTs	ΣChlordanes	ΣHCHs	Pentachlorobenzene	Hexachlorobenzene	Mirex	
St. Paul Island (1987; n=5)	2020	2110	1130	526	5.22	2.35	17.2	This study
St. Paul Island (1990; n=10)	2900	32400	1370	502	5.25	1.34	17	This study
St. George Island (1995–1996; n=10)	3030	3300	NM	NM	NM	1.15	NM	Loughlin et al., 2002
St. Paul Island (1995–1996; n=10)	2400	1990	NM	NM	NM	1.36	NM	Loughlin et al., 2002
St. Paul Island (1997; n=5)	2000	2580	950	409	4.27	0.875	14.5	This study
St. Paul Island (2000; n=10)	2350	713	591	384	2.47	0.788	14.1	This study
St. Paul Island (2003–2004; n=10)	823	1090	NM	72.1	NM	0.42	NM	Wang et al. 2009
St. Paul Island (2006; n=10)	2030	612	468	202	3.1	0.729	10.4	This study
St. Paul Island (2007; n=10)	1900	1850	721	265	3.79	1.14	23.7	This study

Abbreviations: NM=not measured