



## White-tailed eagle (*Haliaeetus albicilla*) feathers from Norway are suitable for monitoring of legacy, but not emerging contaminants

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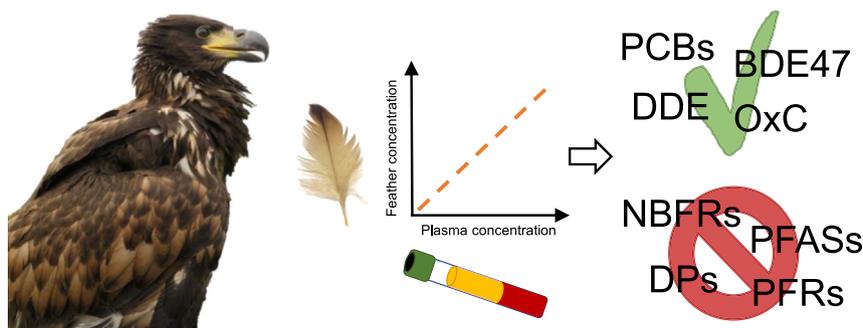
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### HIGHLIGHTS

- Feathers are suitable for monitoring of internal concentrations of legacy POPs.
- PFASs had higher detection frequencies in plasma than in feathers.
- Plasma is the preferred matrix for monitoring of internal concentrations of PFASs.
- Emerging flame retardants had higher detection frequencies in feathers than plasma.

### GRAPHICAL ABSTRACT



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### ABSTRACT

While feathers have been successfully validated for monitoring of internal concentrations of heavy metals and legacy persistent organic pollutants (POPs), less is known about their suitability for monitoring of emerging contaminants (ECs). Our study presents a broad investigation of both legacy POPs and ECs in non-destructive matrices from a bird of prey. Plasma and feathers were sampled in 2015 and 2016 from 70 whitetailed eagle (*Haliaeetus albicilla*) nestlings from two archipelagos in Norway. Preen oil was also sampled in 2016. Samples were analysed for POPs (polychlorinated biphenyls (PCBs), polybrominated diphenyl ethers (PBDEs) and organochlorinated pesticides (OCPs)) and ECs (per- and polyfluoroalkyl substances (PFASs), dechlorane plus (DPs), phosphate and novel brominated flame retardants (PFRs and NBFRs)). A total of nine PCBs, three OCPs, one PBDE and one PFAS were detected in over 50% of the plasma and feather samples within each sampling year and location.

Significant and positive correlations were found between plasma, feathers and preen oil concentrations of legacy POPs and confirm the findings of previous research on the usefulness of these matrices for non-destructive monitoring. In contrast, the suitability of feathers for ECs seems to be limited. Detection frequencies (DF) of PFASs were higher in plasma (mean DF: 78%) than in feathers (mean DF: 38%). Only perfluoroundecanoic acid could be quantified in over 50% of both plasma and feather samples, yet their correlation was poor and not significant. The detection frequencies of PFRs, NBFRs and DPs were very low in plasma (mean DF: 1–13%), compared to feathers (mean DF: 10–57%). This may suggest external atmospheric deposition, rapid internal biotransformation

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or excretion of these compounds. Accordingly, we suggest prioritising plasma for PFASs analyses, while the sources of PFRs, NBFRs and DPs in feathers and plasma need further investigation.

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## 1. Introduction

Polychlorinated biphenyls (PCBs), organochlorinated pesticides (OCPs) and polybrominated diphenyl ethers (PBDEs) are compounds previously used in industrial applications, agriculture and consumer products (Mackay et al., 2006). Classified as persistent organic pollutants (POPs), these compounds are generally lipophilic, semi-volatile and resistant to chemical and biological degradation (Buccini, 2003; Mackay et al., 2006). Consequently, POPs persist in the environment (Letcher et al., 2010; Mackay et al., 2006) and may result in high uptake in biota, followed by bioaccumulation and biomagnification, especially in long and lipid-rich food webs (Borgå et al., 2004; Jones and de Voogt, 1999). As replacements for the legacy POPs regulated by the Stockholm Convention (UNEP, 2009), new and (re-) emerging contaminants (ECs) have entered the market. Those include phosphorus flame retardants (PFRs; van der Veen and de Boer, 2012), “novel” brominated flame retardants (NBFRs; Covaci et al., 2011), dechlorane plus (DPs; Sverko et al., 2011) and certain per- and polyfluoroalkyl substances (PFASs; Lau et al., 2007). These ECs exhibit different physicochemical properties than the legacy POPs and may accumulate in other matrices, such as protein-rich tissues (Lau et al., 2007), or become rapidly metabolised and excreted (Briels et al., 2018; Covaci et al., 2011; van der Veen and de Boer, 2012).

Wild birds are important biomonitors for numerous environmental contaminants (Burger and Gochfeld, 2004; Furness, 1993). Due to ethical and species conservational aspects, non-destructive sampling methods such as the collection of blood or addled eggs are often applied in environmental monitoring programs of wild birds (Espín et al., 2016). The contaminant concentrations detected in blood plasma provide a snapshot of recent exposure through diet (Henriksen et al., 1998), but during periods of low food availability or starvation concentrations can also originate from internal fat reserves (re-exposure) (Fenstad et al., 2014). Egg concentrations on the other hand reflect maternal concentrations deposited during the egg formation (Becker and Sperveslage, 1989). Feathers, either plucked or moulted, present another non-destructive sampling matrix. Feathers are connected to the blood circulation during formation and growth, and during this period the internal contaminant concentrations may thereby be transferred and deposited into the feather (Jaspers et al., 2006; García-Fernández et al., 2013).

The use of feathers as a non-destructive matrix for biomonitoring is increasing (García-Fernández et al., 2013; Gómez-Ramírez et al., 2014). While feathers have been used for decades as a matrix for monitoring environmental concentrations of metal (Burger, 1993), it was only in the early 2000s that feathers were proposed for legacy POP analyses (Dauwe et al., 2005; Jaspers et al., 2006). Recently, feathers have also been investigated as a matrix for analysing and monitoring PFASs (Gómez-Ramírez et al., 2017; Jaspers et al., 2013; Li et al., 2017; Meyer et al., 2009), and only a few studies published to date have investigated the suitability of NBFRs and PFRs monitoring in feathers (Eulaers et al., 2014; Svendsen et al., 2018). Consequently, little is known about the exposure to and deposition of these ECs into feathers. Preen oil has also been proposed as a non-destructive matrix for monitoring PCBs, PBDEs and OCPs (Eulaers et al., 2011b; Van den Brink, 1997), but few studies have collected preen oil for contaminant analyses (Eulaers et al., 2011a, 2011b; Van den Brink, 1997).

Studies investigating non-destructive sampling matrices in birds have been conducted on a wide variety of bird species (García-Fernández et al., 2013). However, there is a general lack of studies with larger sample sizes that have investigated both legacy POPs and ECs in several non-destructive matrices (Espín et al., 2016; García-Fernández et al., 2013). This may improve the evaluation of the suitability of these matrices for monitoring purposes. An overview of contaminant monitoring activities in Europe revealed that 100 monitoring programs from 28 countries have included feathers samples from birds of prey (Espín et al., 2016).

Due to their apex trophic position, large body size and long lifespan, birds of prey such as the white-tailed eagle (*Haliaeetus albicilla*), are good sentinel species for monitoring the presence of contaminants in the environment (Burger and Gochfeld, 2004). White-tailed eagle nestlings are stationary in their nests and therefore good indicators of local exposure to a wide range of environmental contaminants (Olsson et al., 2000). They are also relatively easy to sample while still in the nest (Espín et al., 2016; Eulaers et al., 2011b). The white-tailed eagle was listed as threatened by the International Union for Conservation of Nature in 1988, but today it is listed as of least concern (Birdlife Int., 2016).

In this study, we aimed to evaluate if body feathers and preen oil from white-tailed eagle nestlings present a good non-destructive matrix to monitor internal concentrations of both legacy POPs and ECs. Consequently, we investigated concentrations of legacy POPs and ECs in plasma, feathers and preen oil from 70 white-tailed eagle nestlings. Furthermore, we investigated correlations of POP and EC concentrations in these matrices and evaluated the consistency of these results by including samples from two field locations during two consecutive years. As the sampled feathers were still growing and connected to the blood circulation, we expected to find strong correlations between feathers and plasma concentrations of POPs and ECs. We also expected to find strong correlations between plasma and preen oil, as the oil is produced by an internal gland which is connected to the blood circulation.

## 2. Materials and methods

### 2.1. Field sampling

The study was conducted on 70 white-tailed eagle nestlings from two archipelagos in Norway, Smøla (63.35°N; 8.03°E) and Steigen (67.93°N; 14.98°E), during the breeding seasons of 2015 and 2016. We sampled 13 nestlings in Smøla in 2015 and 22 nestlings in 2016. In Steigen, 14 nestlings were sampled in 2015 and 21 nestlings in 2016. All nestlings, aged from 8 to 12 weeks old, were caught at the nest site and handled for approximately 15 min. Body feathers were gently pulled from the dorsal region, approximately 10 per individual, and stored in polyethylene zipper bags (VWR, USA) at  $-20^{\circ}\text{C}$ . A blood sample of 8 mL was collected in heparinised vacutainers through brachial venepuncture. The blood samples were centrifuged (860 g), after which plasma was transferred to cryogenic tubes (Nalgene®, USA) and stored at  $-20^{\circ}\text{C}$ . Preen oil could only be collected in a sufficient amount in 2016. It was collected in a 1.5 mL Eppendorf tube (VWR, USA) by massaging the preen gland using disposable gloves and avoiding traces of feathers in the sample. The sampling was approved by the Norwegian Food Safety Authority (Mattilsynet; 2015/6432 and 2016/8709) and the

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