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Screening of selected alkylphenolic compounds, biocides, rodenticides and current use pesticides

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Preface

NIVA was commissioned by the Norwegian Climate and Pollution Agency (Klif) to establish the occurrence of nonylphenol, octylphenol and bisphenol A in the marine and freshwater aquatic environments, the antifouling biocide zineb and its transformation product ethylenethiourea (ETU) in harbours, the fungicide transformation product *N,N*-dimethylsulfamide (DMS), selected second generation anticoagulant rodenticides (SGARs) in the livers of selected raptor species, and nonylphenol, octylphenol and selected pesticides in Arctic biota. The results of the screening study are reported here.

Sample collection was coordinated by Sigurd Øxnevad and Eirik Fjeld with sample analysis coordinated by Katherine Langford and Alfhild Kringstad. Jarle Håvardstun, Linda Marie Skryseth, Torbjørn Martin Johnsen and Merete Schøyen contributed to sample collection. Andreas Høgfeldt, Kine Bæk and Malcolm Reid all contributed to sample analysis. We thank Harald Normann Hillersøy and Lars Moe for fishing for crab and cod, and Pål Wergeland for his assistance with sampling at Kviturspollen. We also thank the Anuschka Polder and Vidar Berg from the Norwegian School of Veterinary Science and Lena Haugland Moen and Kjell Handeland the NorwegianVeterinary Institute for providing the liver samples of selected raptor species, The Norwegian Polar Institute, in particular Kjetil Sagerup, for providing the arctic samples and the Water and Wastewater division of Oslo Kommune, in particular Thomas Martinsen, for collecting the samples for DMS analysis.

The project was lead by Katherine Langford and the report written by Langford, K.H., Beylich, B.A., Bæk, K., Fjeld, E., Kringstad, A., Høyfeldt, A., Øxnevad, S. and Thomas, K.V. The risk assessment and quality assurance was completed by Kevin Thomas.

NIVA, Oslo, April 2012

Katherine Langford
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Contents

1. ABSTRACT

This screening survey investigated the occurrence of the alkylphenolic compounds, nonylphenol (nNP, straight chained isomer and tNP, branched chain mixture), nonylphenol monoethoxylate (NP1EO), octylphenol (4nOP straight chained isomer and tOP, branched chain mixture), octylphenol monoethoxylate (OP1EO) and also bisphenol A (BPA) in the marine and freshwater aquatic environments, the antifouling biocide zineb and its transformation product ethylenethiourea (ETU) in marinas, the fungicide transformation product N,N-dimethylsulfamide (DMS) in lakes, selected second generation anticoagulant rodenticides (SGARs) in the livers of selected raptor species, and selected alkylphenols (4nOP, tOP, OP1EO, nNP, tNP and NP1EO) and pesticides in Arctic biota.

For the screening of alkylphenolic compounds and BPA, 2 marine locations were chosen in western Norway: one was a harbour, impacted by industrial and human activities and the other a relatively untouched fjord. Two freshwater lakes (1 large and one smaller impacted) were also sampled. For the screening of zineb, 2 marine small-boat marinas were sampled and one reference location with limited boat activity. Two small lakes in central Oslo and one reference mountain lake were sampled for the screening of DMS. Thirty liver samples from birds of prey collected from all over Norway were provided by the Norwegian School of Veterinary Science and analysed for SGARs, and in addition Arctic biota was provided by the Norwegian Polar Institute.

Concentrations of OP, NP1EO and BPA measured in the samples were all lower than reported threshold values. The tNP $PNEC_{sediment}$ (predicted no effect concentration for tNP in sediment) value was exceeded for in all but 4 of the sediment samples collected suggesting that there is a risk to sediment dwelling organisms in these areas. OP1EO was also detected in sediments at concentrations high enough to cause concern. The concentrations of tNP and OP1EO detected in biota species and water samples were all of less significance than those in sediment.

Zineb was not detected above the limit of detection in any of the dissolved, particulate or sediment samples, while the zineb transformation product ETU was detected at concentrations of between 1 and 16 ng/L ww on suspended particulate matter from the two marinas sampled.

DMS was detected at concentrations of between 104 and 774 ng/L in two urban lakes. These levels represent no risk to aquatic organisms based upon the low aquatic toxicity of DMS as the PNEC (Predicted No Effect Concentration) in water is 140 µg/L. All samples contained levels above the drinking water quality standard or 100 ng/L, although it has to be considered that these lakes are not drinking water sources.

The SGARs brodifacoum, bromadiolone, difenacoum and flocoumafen were detected in golden eagle and eagle owl livers at a total SGAR concentration of between 11 and 255 ng/g in approximately 70% of the golden eagles and 50% of the eagle owls examined in this study. In the absence of specific golden eagle and eagle owl toxicity thresholds for SGARs, a level >100 ng/g (based upon a probabilistic characterization of toxic liver concentrations in barred owl, barn owl, eagle owl and red-tailed hawk) was used as a potential lethal range, accepting that poisoning may occur below this level. Thirty percent (7/24) of the golden eagle and eagle owl livers contained total SGAR residue levels above this threshold. Further estimation of the potential mortality impact on the sampled raptor populations was not possible.

The selected current use pesticides (CUPs) chlorpyriphos, dacthal, methoxychlor, pentachlorophenol (PCP), and trifluralin were not detected at concentrations above the limits of detection in any of the polar cod or capelin (whole fish), seal blubber, sea bird eggs or blood/plasma samples analysed, apart

from a single measurement of 1.4 ng/g of chlorpyrifos in a sample of ringed seal blubber. The same samples when analysed for octylphenol, nonylphenol and their ethoxylated homolgoues also contained levels below the limits of detection.

2. SAMMENDRAG

Dette screeningprosjektet har undersøkt forekomsten av alkyfenolløsninger, nonylfenol (nNP, rettkjedet isomer og tNP, forgrenet kjedeblanding), nonylfenol monoetoxylat (NP1EO), oktylfenol (4nOP rettkjedet isomer og tOP, forgrenet kjedeblanding), oktylfenol monoetoxylat (OP1EO) og bisfenol A (BPA) i marine lokaliteter og i ferskvann. I tillegg er det analysert for bunnstoffet zineb og dets nedbrytingsprodukt etylentiourea (ETU) i småbåthavner. Nedbrytingsproduktet *N,N*-dimetylsulfamid (DMS) fra soppmiddlene diklofluanid og tolylfluanid er undersøkt i ferskvannslokaliteter og noen annengenerasjons rottegiftmidler (SGARs) er undersøkt i leverprøver av utvalgte rovfuglarter. Noen alkyfenoler (4nOP, tOP, OP1EO, nNP, tNP og NP1EO) og pesticider I arktisk biota.

For screening av alkylfenolløsninger og BPA ble det valgt ut to marine lokaliteter på Vestlandet, hvorav den ene er påvirket av menneskelig aktivitet og industri mens den andre er en relativt upåvirket fjord. For analyser av disse stoffene ble det også tatt prøver fra to innsjøer (én stor innsjø som er påvirket av menneskelig aktivitet, og én liten innsjø). For screening av zineb ble det tatt prøver fra to småbåthavner og en referanselokalitet hvor det er lite båtaktivitet. For screening av DMS ble det tatt prøver av to små innsjøer i Oslo, og fra en fjellsjø som referanselokalitet. Det ble også analysert 30 leverprøver av rovfugler for kartlegging av annengenerasjons rottegiftmidler. Leverprøvene ble samlet inn fra hele Norge med hjelp fra Norges Veterinær Høgskole. Arktisk biota ble skaffet av Norsk polar institutt.

De målte konsentrasjonene av OP, NP1EO og BPA var alle lavere enn grenseverdiene for disse stoffene. Grensen for $PNEC_{sediment}$ (predicted no effect concentration av tNP i sediment) ble overskredet for tNP i alle unntatt fire av sedimentprøvene. Dette betyr at tNP utgjør en risiko for sedimentlevende organismer i disse områdene. OP1EO ble også påvist i sedimentprøver i så høye konsentrasjoner at det gir grunn til bekymring. Det ble bare funnet lave konsentrasjoner av tNP og OP1EO i prøvene av biota og vann.

Det ble ikke påvist zineb løst i vann, på partikler eller i sedimentprøvene. Nedbrytingsproduktet ETU ble imidlertid påvist i konsentrasjoner mellom 1 og 16 ng/L (ww) på suspenderte partikler fra to av de undersøkte småbåthavnene.

Det ble påvist konsentrasjoner av DMS på mellom 104 og 774 ng/L i to innsjøer i urbane områder. Disse nivåene utgjør ingen risiko for akvatiske organismer siden DMS er lite toksisk i akvatisk miljø når PNEC i vann var 140 µg/L. Alle prøvene inneholdt nivåer av DMS som er høyere enn standarden for drikkevann på 100 ng/L. Dette skal likevel ikke utgjøre noen risiko siden disse innsjøene ikke er drikkevannskilder.

De annengenerasjons rottegiftmidlene brodifacoum, bromadiolon, difenacoum og flocoumafen ble påvist i leverprøver av kongeørn og hubro. Totalkonsentrasjonene av SGAR var på mellom 11 og 255 ng/g i omtrent 70% av kongeørnene og 50% av hubroene som ble analysert i denne undersøkelsen. Siden det ikke finnes grenseverdier for giftighet av SGAR for kongeørn og hubro, er nivåer >100 ng/g (basert på sannsynlighets beregning av toksiske leverkonsentrasjoner i høvdingugle, tårnugle, hubro og rødhalevåk) brukt som potensielt dødelig nivå, og verdier under dette kan ha toksiske effekter. 30% (7/24) av leverprøvene av kongeørn og hubro inneholdt restkonsentrasjoner av total SGAR som var høyere enn denne grenseverdien. Det er ikke mulig å gi noen videre vurdering av virkningen disse stoffene kan ha på dødelighet i de undersøkte rovfuglpopulasjonene.

De valgte nåværende bruk plantevernmidler (CUPs; current use pesticides) klorpyrifos, dacthal, metoksyklor, pentaklorfenol (PCP) og trifluralin hadde konsentrasjoner under deteksjonsgrensen i prøver analysert fra polartorsk eller lodde (hel fisk), selspekk, sjøfuglegg, eller blod/plasmaprøver, med unntak fra en enkelt prøve hvor det ble målt 1,4 ng/g klorpyrifos i ringselspekk. De samme prøvene hadde også konsentrasjoner av oktylfenol, nonylfenol og etoksylater lavere enn deteksjonsgrensen.

3. ABBREVIATIONS

4nNP	4-n-nonylphenol (straight chain)
tNP	Technical nonylphenol (branched chain)
NP1EO	Nonylphenol monoethoxylate
4nOP	4-n-octylphenol (straight chain)
4tOP	4-tertiary-octylphenol (branched chain)
OP1EO	Octylphenol monoethoxylate
AP	Alkylphenol
APEO	Alkylphenol ethoxylate
BPA	Bisphenol A
ETU	Ethylenethiourea
DMS	*N*,*N*-Dimethylsulfamide
SGAR	Second generation anticoagulant rodenticide
CUP	Current use pesticide
LC/MS/MS	Liquid chromatography-tandem mass spectrometry
GC-ToF-MS	Gas chromatography-time of flight- mass spectrometry
GC-CI-MS	Gas chromatography-chemical ionization-mass spectrometry
GPC	Gel permeation chromatography
PNEC	Predicted No Effect Concentration

4. INTRODUCTION

This report presents the results of screening for the occurrence of contaminants from five different groups; alkylphenols and bisphenol A, the antifouling biocide zineb and its transformation product ethylenethiourea, the fungicide transformation product *N,N*-dimethylsulfamide, second generation anticoagulant rodenticides, brodifacoum, bromadiolone, difenacoum difethialone and flocoumafen, and current use pesticides chlorpyriphos, dacthal, methoxychlor, pentachlorophenol (PCP), and trifluralin.

4.1 Alkylphenolic compounds and bisphenol A

Long chain alkylphenol polyethoxylates (APnEO) have been used in industrial and domestic products and processes as surfactants for 60 years (EU 2002; EA 2005). Bisphenol A is been used as an antioxidant in plastics and epoxy resin manufacturing and during paper recycling, amongst other applications (EU 2003). Bisphenol A is a high production plasticizer (29 tonnes registered sold in Norway for 2009) and is ubiquitous in the aquatic environment as are the alkylphenols and their ethoxylate homologues.

Nonylphenol (NP) and nonylphenol monoethoxylate (NP1EO), and octylphenol (OP) and octylphenol monoethoxylate (OP1EO) are degradation products of the anthropogenic longer chain ethoxylated compounds, NPnEO and OPnEO respectively and their presence and persistence in the environment has been studied for many years. Bisphenol A has a log K_{ow} slightly less than the AP and APnEO compounds but is still likely to partition to the sediment phase. The Log K_{oc} values in Table 1 also highlight the likelihood of sediment concentrations being higher than those in water.

Their structures and physic-chemical properties mean NP and OP, as well as their corresponding monoethoxylates, act as endocrine disrupting compounds (Jobling *et al.* 1993; Jobling *et al.* 1996). Biological and chemical processes during wastewater treatment processes and those occurring in the in the aquatic environment, result in the removal of the ethoxy units from the long chain homologues to form shorter chain homologues and alkylphenols. As the ethoxylated chain becomes shorter, the log K_{ow} value, toxicity and environmental stability increase (Langford *et al.* 2005; Langford *et al.* 2005; Langford *et al.* 2007). Wastewater treatment works effluent is a significant point source of NP and NP1EO, and OP and OP1EO to surface waters but high log K_{ow} values of APs, APEOs and BPA mean the most likely environmental sink is through sorption to sediments and particulates (Yang *et al.* 2011). In Tokyo, 20% of the NP in river water was sorbed to the particulate phase (Isobe *et al.* 2001). Partition coefficients are shown in table 1.

Table 1. Partition coefficients normalized to organic carbon content

Compound	Log K_{oc}	Reference
NP	5.22 ± 0.38	Isobe 2001
NP1EO	5.46	Ferguson 2001
OP	4.65 ± 0.42	Isobe 2001

A tissue bioaccumulation factor of 50 in wild fish muscle compared to measured water samples has been calculated for NP in a UK river (Blackburn *et al.* 1999). Fish from Japanese rivers also contained residues of NP and OP and a seasonal variation was also observed with higher concentrations in the summer season than in winter (Tsuda *et al.* 2000). 4NP and BPA have both recently been identified as

anti-androgenic compounds occurring in fish bile exposed to wastewater effluent (Rostkowski *et al.* 2011).

4.2 Zineb and ethylenethiourea

Zineb is an antifouling biocide frequently used together with copper compounds in antifouling paints in Norway, although to date there is a lack of environmental occurrence data. In water, zineb hydrolyses quickly to its metabolite ethylenethiourea (ETU) (Thomas *et al.* 2002) (Figure 1). This rapid transformation of zineb may be one of the reasons for the absence of occurrence data. It is also very difficult to analyse zineb directly and a more robust methodology is to convert zineb to carbon disulphide (CS_2) for analysis (Thomas *et al.* 2010). Evaluating ETU and CS_2 concentrations enables an assessment of zineb concentrations. ETU has shown teratogenic and mutagenic properties.

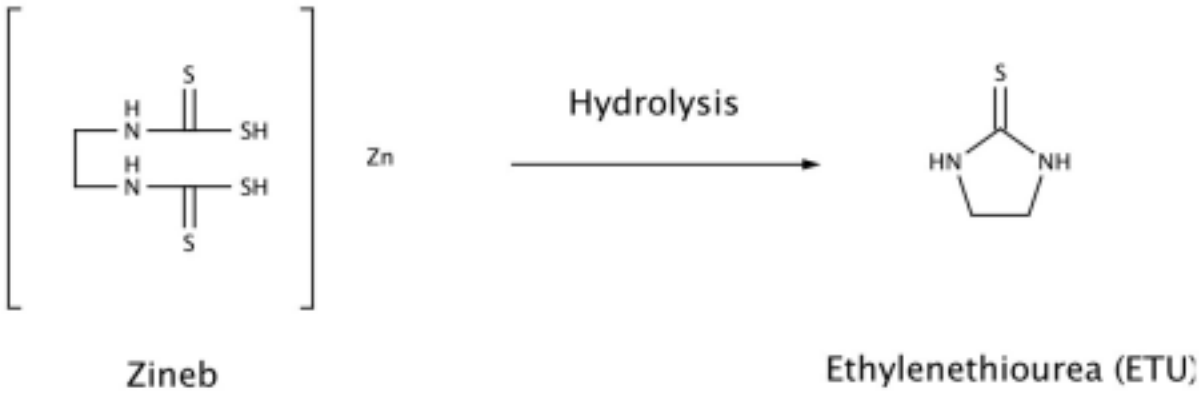

Figure 1. Transformation of zineb to ethylenethiourea (ETU)

4.3 *N,N*-dimethylsulfamide (DMS)

Tolylfluanid and dichlofluanid are sulfonamide fungicides used as wood preservative in external paint products for wooden buildings, as pesticides in the agricultural industry to protect crops from spoilage, and also as antifoulant biocides in paint coatings for boats and ships. Both of these compounds degrade to *N,N*-dimethylsulfamide (DMS) (Figure 2). DMS is a polar transformation product and shows little or no sorption to sediments (Dalkmann *et al.* 2012). Due to its high polarity, its removal from wastewater is poor and ozonation of drinking water results in the formation of N-nitrosodimethylamine (NDMA). During ozonation 30-50% of the DMS was converted to NDMA in laboratory studies (Schmidt *et al.* 2008). NDMA is a genotoxic, mutagenic and carcinogenic substance.

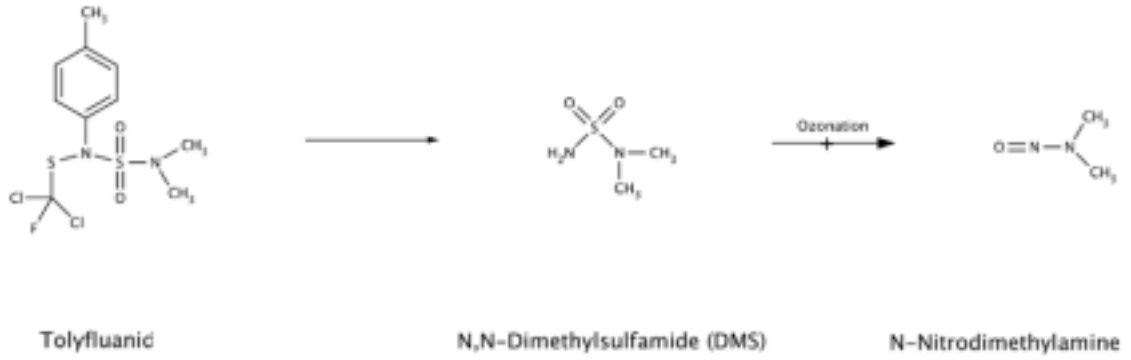

Figure 2. Formation of *N-N*-dimethylsulfamide (DMS) and N-nitrodimethylamine (NDMA) from tolylfluanid

DMS does not fulfil the criteria for a PBT (persistent, bioaccumulative and toxic) substance, however it can be considered a vP compound with a half-life (DT_{50}) in water >1000 days (967 days).

A ground water study spanning 23 European Countries (Loos *et al.* 2010) highlighted DMS as one of the pesticide degradation products that was most frequently detected in amounts exceeding the European ground water quality standard of 100 ng/L (EC 2006). It exceeded the quality standard in 8 of the 23 samples with an average concentration of 332 ng/L and a maximum of 52 000 ng/L. In another study on German groundwater, raw water, surface water and drinking water, DMS was detected in approximately 65% of the 600 samples at concentrations of up to 63 000 ng/L (Kowal *et al.* 2009).

4.4 Second generation rodenticides

The first generation rodenticides were introduced for pest control in the 1940s but after some rodents developed resistance to these compounds, second-generation anticoagulant rodenticides (SGARs) were developed and introduced in the 1970s. The SGAR group includes brodifacoum, bromadiolone difenacoum, difethialone, and flocoumafen. They act as vitamin K antagonists and interfere with the synthesis of blood clotting agents in vertebrates which makes vertebrates vulnerable to hemorrhage (Stone *et al.* 2003; Vandenbroucke 2008).

Compared to the first generation of rodenticides such as warfarin, SGARs are more likely to have effects on non-target species due to their extremely slow elimination rate from the target species and their higher vertebrate liver toxicity. They are likely to accumulate in non-target species which consume either bait or poisoned rodents. Exposed rodents can survive for several days after consumption of SGARs and continue to consume bait which in turn increases their body burden allowing an even greater exposure potential to non-target predators. SGARs are considered high potency anticoagulants and the substances are retained in the liver for 6-12 months after exposure, compared to up to 1 month for warfarin, a first generation rodenticide (Eason *et al.* 2002). Flocoumafen, for example, demonstrated an elimination time of greater than 100 days in exposed barns owls (*Tyto alba*) (Eason *et al.* 2002).

Exposure can occur indirectly as a result of avian and mammalian predators consuming exposed target or non-target rodent species, or indirectly through direct consumption of the baits. Indirect exposure is the biggest concern for predatory species in Norway but despite the wide use of SGARs in Norway, no data are available on the occurrence of SGAR residues in non-target species (Laakso *et al.* 2010).

Studies outside of Norway have shown that these compounds can affect non-target species, however, LC50 data are only available for very few predatory species. The lack of toxicity data for predatory species makes it difficult to confirm the link between SGARs and cause of death in most cases as does the likelihood that some species are more sensitive than others (Thomas *et al.* 2011). LD50 data is presented in tables 2 and 3.

In a UK study, 20% of dead tawny owls (*Strix aluco*) contained detectable concentrations of one or more SGARs although this species appears to be less vulnerable to poisoning than other species according to the Walker *et al.* (2008). In a US study, containing data from New York from 1998-2001, 49% of 12 raptor and vulture species contained traces of SGARs, although only 7.2% of the deaths were directly attributed to SGAR poisoning (Stone *et al.* 2003). In one particular species, the eagle owl (*Bubo bubo*), SGARs were detected in 81% of the dead birds. A Canadian study attributed only 4% of 164 deaths to exposure to SGARs (brodifacoum) (Albert *et al.* 2010).

Brodifacoum is highly insoluble in water and very persistent (DT50 157 days) and immobile in soil (K_{oc} > 9155 l/kg) (EU 2010). If bait is placed in an outdoor environment, a likely sink for uneaten bait is the soil. The environmental occurrence of brodifacoum was investigated in New Zealand (Ogilvie 1997). Aerial application of brodifacoum was used on a small island to eradicate rats. After a single aerial spraying episode, no residues were detected in water or soil, or in the beetles found on the bait although it is possible that the sampling campaign was not extensive enough. However, residues were detected in one anthropod (*Gymnoplectron* spp), and in the livers of one owl (*Ninox novaeseelandiae*) and one parakeet (*Cyanoramphus novaezelandiae*). Clearly it is difficult to draw conclusions from such a small study but it does highlight the potential of exposure. The parakeet likely consumed bait directly and the owl was probably exposed via a secondary exposure route. The occurrence of residues in the anthropod raise concerns about insectivore exposure whereas other studies have all focused on carnivorous species such as raptors and vultures.

In a Canadian study of 3 different owl species, 70% of the birds had residues of at least one SGAR and 41% of these had residues of more than one SGAR (Albert *et al.* 2010).

Table 2. LD50 values for different second generation anticoagulant rodenticides for non-target species

Species	**Anticoagulant**	**LD50 (mg/kg)**	**Reference**
Mink (*Mustela lutreola*)	Brodifacoum	9.2	(Erickson *et al.* 2004)
Australasian harrier (*Circus approximan*)	Brodifacoum	10.0	(Eason *et al.* 1995)
Raven (*Corvus corax*)	Brodifacoum	0.56	(Howald *et al.* 1999)
Canada goose *(Branta canadensis)*	Brodifacoum	<0.75	(USEPA 1998)
Mallard *(Anas platyrhynchos)*	Brodicacoum	0.26	(USEPA 1998)
Northern bobwhite *(Colinus virginianus)*	Difethialone	0.26	(USEPA 1998)
Northern bobwhite *(Colinus virginianus)*	Bromadiolone	138	(USEPA 1998)

Table 3. LD50 values for different anticoagulant rodenticides for mice (Vandenbroucke 2008)

Anticoagulant rodenticide	LD50 (ng/kg body wt)
Brodifacoum	0.4
Difenacoum	0.8
Flocoumafen	0.8
Difethialone	1.29
Bromadiolone	1.75
Warfarin (1st generation)	374

4.5 Pesticides in the Arctic

Chlorpyrifos, dacthal, methoxychlor, pentachlorophenol (PCP) and trifluralin were selected for screening in Arctic samples. All five pesticides are or have been used on a large scale globally as agrochemicals and are commonly referred to as current use pesticides (CUP) with a recent review showing that they been reported to occur in the Arctic environment (Hoferkamp *et al*. 2010). CUPs that have been identified as of concern in the Arctic are currently or have been produced in high volumes in temperate regions, have relatively high air-water partitioning, and have the potential to bioaccumulate and biomagnify (Hoferkamp *et al*. 2010; Table 4). Chlorpyrifos is an organothiophosphate insecticide that is used globally in high volumes, however use in Norway is restricted. Dacthal is an organochlorine herbicide with no reported agricultural use in Norway. Methoxychlor is an organochlorine insecticide with a high global production (8,500 to 50,000 tonnes/year), however it is no longer registered for use in Norway, the EU or USA. PCP is a chlorinated herbicide/fungicide. Its use in Norway is strictly controlled with a reduction in release of over 99% between 1995 and 2009, although some release is known to occur in leachate from landfill sites (Klif, 2009). PCP has been proposed for inclusion under the Stockholm Convention, however currently it is not included and under consideration since there is disagreement as to whether it should be included for further evaluation alongside its transformation product pentachloroanisole since PCP does not meet all of the selection criteria. Previous screening of PCP in Barents Sea sediments reported less than detection limit levels (Gabrielsen *et al*. 2008), while screening for PCP on mainland Norway in freshwater sediments from three different locations (the Lågen river delta in Oppland and the lakes Mjøsa and Selbusjøen) showed very low or below detection limit levels. Trifluralin is a dinitroaniline herbicide that is not registered for use in Norway and its use is prohibited in the EU.

Table 4. Technical data for the selected current use pesticides (CUPs) screened in the Arctic (Hoferkamp *et al*. 2010)

Common name	Class	Soil DT50 (days)	Solubility (mg/L)	Log K_{OW}	AC-BAP	CTD (km)
Chlorpyrifos	Organophosphate insecticide	30	0.4	4.96	NA	430
Dacthal	Organochlorine insecticide	45	0.5	4.28	No	2690
Methoxychlor	Organochlorine insecticide	120	7	5.08	NA	55
Pentachlorophenol (PCP)	Chlorinated herbicide/fungicide	10-100	14	5.12	NA	1320
Trifluralin	Dinitroaniline herbicide	60	0.18	5.34	NA	110

AC-BAP: Arctic accumulation potential, CTD: Characteristic travel distances, NA: Not available.

5 MATERIALS AND METHODS

5.1 Sample collection

Figure 3. Sample location summary

5.1.1 Marine Sampling

5.1.1.1 Sampling locations for screening of alkylphenolic compounds and bisphenol A.

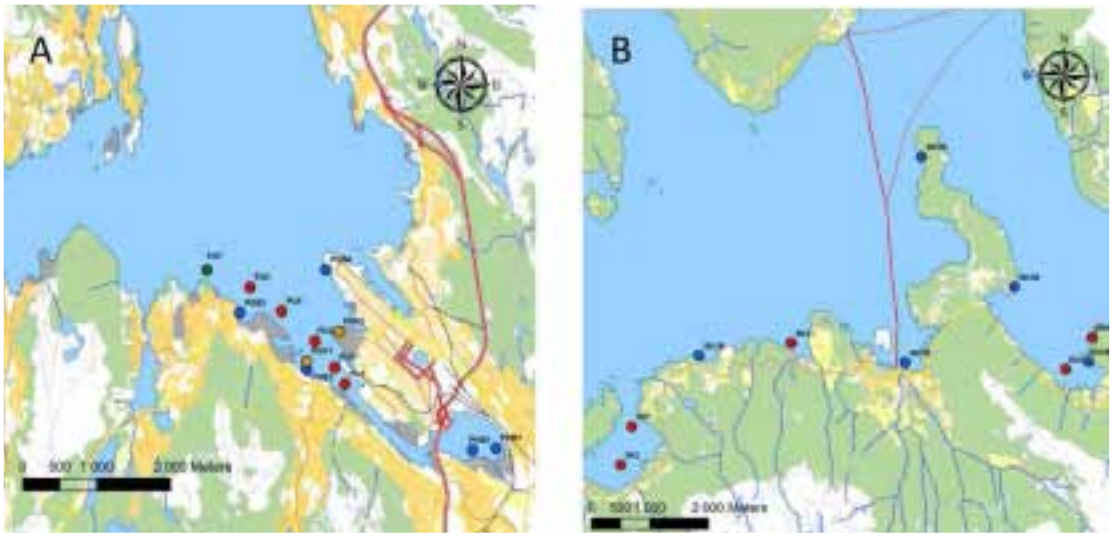

Figure 4. Sampling locations for the screening of alkylphenolic compounds and bisphenol A in the marine environment. A. Puddefjorden, Bergen B. Skånevikfjorden. (green=cod, red=water and sediment, blue=blue mussels and orange=crab)

Samples were collected from Puddefjorden and Byfjorden (Bergen, Western Norway), and from Skånevikfjorden (Western Norway) for the screening of alkylphenolic compounds and bisphenol A (Table 5). The samples from the Skånevikfjord were used as reference material.

Table 5. Overview of sample locations and collection dates for the screening of alkylphenolic compounds and bisphenol A in the marine environment

		Lat	Long	Depth (m)
Pudderfjorden	05.10.2011			
Blue mussel 1		60,38142	5,34978	
Blue mussel 2		60,38078	5,34413	
Blue mussel 3		60,38706	5,30123	
Blue mussel 4		60,39876	5,30241	
Blue mussel 5		60,39230	5,28310	
Sediment 1		60,38604	5,31094	16
Sediment 2		60,38778	5,30789	26
Sediment 3		60,39044	5,30225	39
Sediment 4		60,39323	5,29318	56
Sediment 5		60,39542	5,28465	79
Water 1		60,38604	5,31094	14
Water 2		60,38778	5,30789	21
Water 3		60,39044	5,30225	26
Water 4		60,39323	5,29318	26
Water 5		60,39542	5,28465	26
Skånevikfjorden	12.09.2011			
Blue mussel 1		59,73143	5,87493	
Blue mussel 2		59,73437	5,93528	
Blue mussel 3		59,76593	5,93168	
Blue mussel 4		59,74810	5,96500	
Blue mussel 5		59,73833	5,99040	
Sediment 1		59,71893	5,85603	51
Sediment 2		59,71288	5,85425	52
Sediment 3		59,73490	5,90027	9
Sediment 4		59,74207	5,99042	10
Sediment 5		59,73670	5,98375	10
Water 1		59,71893	5,85603	9
Water 2		59,71288	5,85425	9
Water 3		59,73490	5,90027	9
Water 4		59,74207	5,99042	9
Water 5		59,73670	5,98375	9

5.1.1.2 Sampling locations for screening of zineb and ethylentiourea

Samples were collected from two small craft harbours: Oslo motorbåtforening (Bestumkilen, Oslo) and Bergens seilforening (Kviturspollen, Bergen). Skånevikfjorden was used as the reference location (Table 6).

Table 6. Overview of sample locations and matrices for the screening of zineb and ethylenethiourea (ETU) in the marine environment

	Lat	Long	Depth (m)
Skånevikfjorden 12.09.2011			
Sediment 1	59,71893	5,85603	51
Sediment 2	59,71288	5,85425	52
Sediment 3	59,73490	5,90026	9
Sediment 4	59,74207	5,99041	10.2
Sediment 5	59,73670	5,98375	10.2
Water/particulate 1	59,71893	5,85603	9
Water/particulate 2	59,71288	5,85425	9
Water/particulate 3	59,73490	5,90026	9
Water/particulate 4	59,74207	5,99041	9
Water/particulate 5	59,73670	5,98375	9
Kviturspollen 02.11.2011			
Sediment 1	60,26289	5,25371	13.1
Sediment 2	60,26264	5,25259	12.8
Sediment 3	60,26276	5,25082	11.7
Sediment 4	60,26329	5,24830	11
Sediment 5	60,26310	5,24164	12.9
Water/particulate 1	60,26289	5,25371	11
Water/particulate 2	60,26264	5,25259	11
Water/particulate 3	60,26276	5,25082	10
Water/particulate 4	60,26329	5,24830	10
Water/particulate 5	60,26310	5,24164	11
Bestumkilen 01.07.2011			
Sediment 1	59,55171	10,40403	4
Sediment 2	59,55133	10,30328	2
Sediment 3	59,55011	10,40063	5.5
Sediment 4	59,54849	10,39573	5
Sediment 5	59,54734	10,39343	9
Water/particulate 1	59,55171	10,40403	4
Water/particulate 2	59,55133	10,30328	2
Water/particulate 3	59,55011	10,40063	5.5
Water/particulate 4	59,54849	10,39573	5
Water/particulate 5	59,54734	10,39343	9

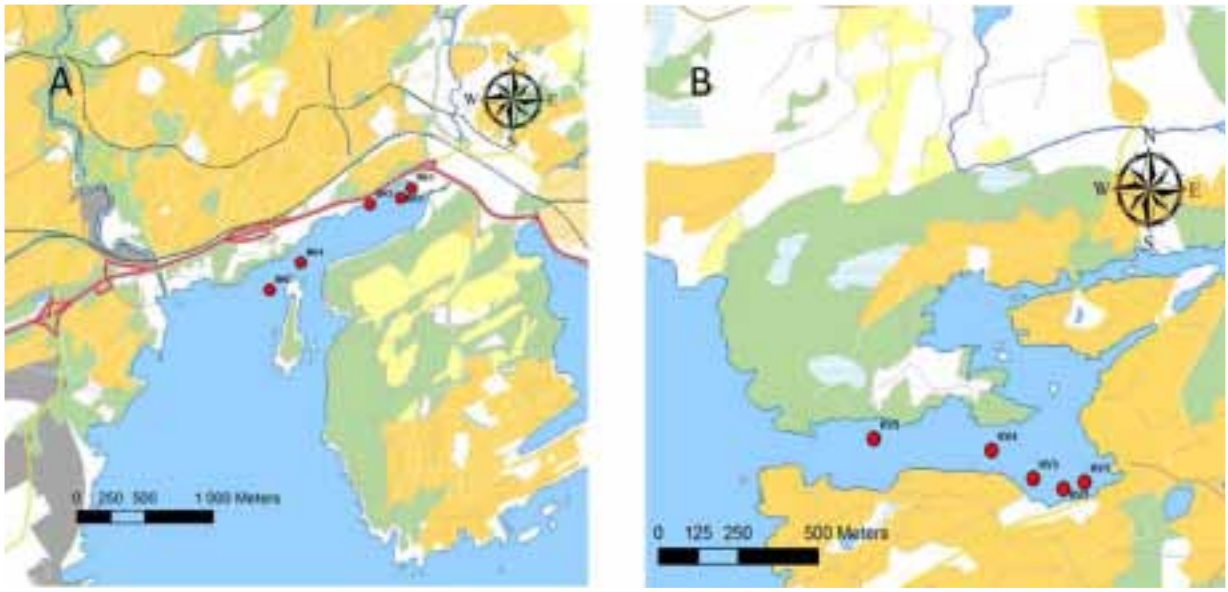

Figure 5. Sampling locations for water, particulate and sediment samples for zineb and ETU analysis. A. Bestumkilen, Oslo B. Kviturspollen, Bergen


Figure 6. Oslo motorbåtforening (Bestumkilen, Oslo)

Samples of biota and sediment were put in clean, baked (500 °C) glass jars. The water samples were put in clean, baked (500 °C) bottles (2.5 L).

- *Water and Particulate*

The water samples were collected by using a Nisikin water sampler. The samples collected from the small craft harbours were collected from near the bottom. The water samples at the other locations were taken from approximately 10 meters depth. Water samples were filtered through pre-cleaned GFC filters (0.45 µm, Whatmann) to collect the particulate fraction.

- *Sediment*

Sediment samples were collected from five stations at each location by using a van Veen grab. The sediment sample was collected from the upper 2 cm of sediment of three parallel grabs.

- *Blue mussel*

Blue mussels were collected from five stations at each location. At least 40 blue mussels (3-5 cm) were collected from each station. Blue mussels were frozen (-20 °C) upon arrival at NIVA from the field. The soft tissue of 30 blue mussels was pooled into one bulk sample from each station.

- *Cod*

Cod were caught with fish pots and gill nets by local fishermen and then sent to NIVA for analysis. The cod varied in size from 41 to 81 cm and 720 to 4750 grams (Table 7). Individual liver samples were taken for analysis.

Table 7. Length and weight of cod from the three locations, with weight of liver samples

Location	Length (cm)	Weight (g)	Liver (g)
Byfjorden	51 - 63	1343 - 2983	33.4 – 155.8
Skånevikfjorden	61 - 81	1893 - 3879	17.4 – 167.6
Lofoten	41 - 82	720 - 4750	7.7 – 155.6

- *Brown crab*

Brown crab was caught with crab pots by local fishermen, frozen and sent to NIVA for analysis. Approximately 20 g of soft tissue (white and brown tissue including hepatopancreas) of five crabs were pooled into one bulk sample. This resulted in four samples from each location.

5.1.2 Freshwater sampling

5.1.2.1 Sampling locations for screening of alkylphenolic compounds and bisphenol A.

Water samples, surface sediment samples and trout (*Salmo trutta*) were collected from Lake Mjøsa and Lake Spjeldsjøen (reference lake). The sites and sampling dates are given in Table 8.

Lake Mjøsa is Norway's largest lake with a surface area of 369 km^2, situated 123 m above sea level, with a maximum depth of 456 m. Approximately 150 000 people live in the surroundings of Lake Mjøsa. The sediment and water sampling took place at five different stations, spread out along a gradient that should reflect different degree of influence from the city of Gjøvik and the sewage treatment plant situated here.

Lake Spjeldsjøen is a small forest lake located approximately 15 km east of Lake Mjøsa. It has a surface area of 0.32 km^2, situated 603 m above sea level, with a maximum depth of 9 m. The lake is located in a remote area without any residents. Three cabins, mostly used by fishermen and hunters, are the only buildings in the lake catchment. The sediment sampling took place at the deepest part of the lake, whereas the water sampling was done in the outlet stream.

Figure 7. Sampling locations for water, particulate and sediment samples for screening of alkylphenolic compounds and bisphenol A

- *Water*

A portable high volume water sampling system was used to collect samples of suspended solids and filtered water. The system consists of a filter holder of stainless steel (Millipore, 293 mm) equipped with a GF filter (nominal pore size: 1.0 µm), a peristaltic pump with a manual speed regulator, and silicone tubing.

The GF filters were cleaned prior to sampling by soaking them in hexane and methanol (laboratory grade).

The sampling system was cleaned before collecting each sample by flushing it with laboratory grade acetone and methanol (approximately 500 ml of each). After air drying and before the GF filter was installed, traces of solvents were removed by pumping 30 L of untreated natural water from the sampling site through the system. All equipment handling were done wearing clean butyl-nitrile gloves. A total volume of 100 L was filtered at each sampling station. The water flow was held constant at 2.5 L/ min. Samples of filtered water were taken after 50 L of the total volume had been filtered. The filtered water samples were collected in clean 5 L glass bottles. The GF filters with the suspended solids were removed by using stainless steel tweezers. The filters were folded, wrapped in

clean (ignited) aluminium foil, put in zip-lock polyethylene bags, and kept frozen until transferred to the NIVA laboratory.

The water samples from Lake Mjøsa were collected at a depth of 10 m to avoid debris floating in the surface water due to flooding. The water samples from Spjeldsjøen were collected from the outlet stream. The sampling here occurred at the onset of the autumn circulation, and they should therefore represent a mixed water column.

Table 8. Sites and dates for the sampling of sediments and lake water.

Location Description	UTM33 Longitude	UTM33 Latitude	Sediment depth (m)
Lake Mjøsa			
(NVE No. 118)			
1. Ringsakerfjordenat Kolberg	267020	6753600	130
2. Rambekk sewage treatment plant	266660	6745850	50
3. Hunnselva. South of river outlet	266400	6747390	45
4. Gjøvik South	269350	6745070	190
5. Gjøvik North	266830	6748960	235
Lake Spjeldsjøen			
(NVE No. 33698)			
Central part (sediment replicates)	289700	6777460	9 m
Outlet stream (water replicates)	289923	6778415	

- *Sediment*

The sediment sampling in Lake Mjøsa took place at five different stations, whereas only the deepest part of Lake Spjeldsjøen was sampled (Table 8).

The samples consisted each of three subsamples of surface sediments (0–2 cm). They were sampled with a gravity corer with a stainless steel tube. For Lake Mjøsa the sampler was equipped with an all metal core catcher mechanism to avoid the soft sediments slipping out of the tube.

The coring tube and the core catcher mechanism were rinsed with solvents (lab grade acetone and methanol) before sampling a new site.

The sediment samples (approximately 150 ml each) were kept in ignited (550 °C) glass jars sealed with ignited aluminium foil under the lids. The sediment samples were stored dark and cooled (≈ 4 °C) until they were transferred to the NIVA laboratory.

- *Trout*

Local fishermen collected fish from Lake Mjøsa (North, at Vingerom), whereas personnel from NIVA caught fish from Lake Spjeldsjøen (southern part of the lake). The fish were caught by gill nets. Mean length and weight of the analysed fish were: Lake Mjøsa, 69 (± 96) cm and 3.81 (± 2.22) kg; Lake Spjeldsjøen, 25.0 (± 5.2) cm and 0.203 (± 0.135) kg.

The fish were kept frozen (- 20 °C) in polyethylene bags until samples could be taken at the fish dissection lab at NIVA's office at Hamar. The sampling procedure followed the manual given by ICP Waters Programme Centre (Centre 2010). Dorsal skin- and bone free muscle fillets were cut free using stainless steel blades and tweezers. The equipment was rinsed in acetone and methanol (laboratory grade) between each sample to avoid cross contamination. All sampling were done wearing clean butyl-nitrile gloves and hand contacts with the samples were avoided. From each individual approximately 50–100 g muscle was stored in ignited (550 °C) glass jars sealed with ignited aluminium foil under the lids. The samples were kept frozen (-20 °C) until analysis.

The large difference in mean body size between the populations reflects their feeding habits. The trout population of Lake Mjøsa is piscivorous (fish eating) and the individuals may attain a body size of 10–12 kg, whereas the population of Lake Spjeldsjøen mainly feed on invertebrates and individuals rarely grow larger than 1 kg.

5.1.2.2 Sampling locations for screening of N,N-dimethylsulfamide

Samples were collected from 2 lakes in central Oslo (Figure 8) and from a control location approximately 130 km North West of the city. One city lake was Holmendammen on the West side of Oalo that receives runoff from a large residential area and also from woodlands used for recreation. The second city lake was Østensjø in the East of the city with a similar runoff influence as Holmendammen. The reference lake, Skodøltjenn, was situated in Trillemarka-Rollagsfjell Nature Reserve which is one of the few remaining untouched old growth forest areas in Norway. With no influence from residential or commercial buildings it can be considered a remote lake in the mountains. All samples were collected in November 2011 during dry weather conditions.

- *Water*

Water samples (2.5 L) were collected from 3 locations on Holmendammen and Østensjø. At Holmendammen, 2 x spot samples (21st November 2011) and a 96 hour composite sample were collected (17th to 21st November 2011). At Østensjø, 1 spot sample (21st November 2011) and 2 x 96 hour composite samples (17th to 21st November 2011) were collected. Water samples (2.5 L) were collected from 5 locations from the reference lake, Skodøltjenn (17th November 2011), in a transect running from the lake inflow to the outflow.

- *Sediment*

Sediment samples were collected from 3 locations along the river flowing into Holmendammen and 2 locations along the river flowing into Østensjø. 5 sediment samples were collected from the outflow point of the reference lake, Skodøltjenn.

Figure 8. Sampling locations in Oslo for the screening of N,N-dimethylsulfamide (DMS)

5.1.3 Sampling locations for screening of second generation rodenticides

Archived liver samples were kindly provided by the Norwegian School of Veterinary Science and the Veterinary Institute in Oslo (Table 9). The birds were all discovered already dead in the wild and were autopsied and the livers were then stored frozen. Cause of death was not established.

Table 9. Avian predator species collected for analysis of second generation anticoagulant rodenticides (SGARs)

Species	Location	Year	Age/Sex	Weight (kg)
Osprey	Øvre Rendal	2010	Adult/M	-
(Pandion haliaetus)	Sør Trøndelag	2009	Juvenile/-	1.135
	Valldal	2010	Adult/F	-
Golden Eagle	Løten	2010	Adult/M	3.745
(Aquila chrysaetos)	Flekkefjord	2009	Adult/F	-
	Vikeså	2009	Adult/F	4.8
	Vinje	2010	Juvenile/F	5.0
	Vik i Sogn	2010	Juvenile/M	-
	Hol i Tjeldsund	2010	Juvenile/M	3.4
	Hansnes	2011	Juvenile/-	-
	Åndalsnes	2009	Adult/F	5.4
	Vikeså	2010	Adult/F	-
	Lyngdal	2011	Juvenile/M	3.76
	Oppdal	2011	Juvenile/M	4.5
	Engerdal	2009	Juvenile/F	3.8
	Balestrand	2009	Juvenile/M	2.78
	Rena	2009	Juvenile/F	4.05
	Tolga	2011	Adult/F	5.02
	Kyrksæterøra	2009	Juvenile/M	2.8
Eagle owl	Hitra	2011	Adult/F	3.0
(Bubo bubo)	Hitra	2011	Adult/F	-
	Kopervik	2011	Adult/F	1.6
	Hitra	2010	Adult/F	2.345
	Mandal	2011	Adult/F	-
	Halden	2010	Adult/F	1.7
	Hardbakke	2011	Adult/F	2.522
	Fitjar	2009	-/-	1.78
Gyrfalcon	Berlevåg	2011	Juvenile/F	1.48
(Falco rusticolus)				
Peregrine Falcon	Roa	2010	-/M	0.77
(Falco peregrinus)	Gaupne	2009	Adult/F	-

- denotes where no data were available

5.1.4 Arctic sampling

Samples from Svalbard were kindly collected by The Norwegian Polar Institute during the Autumn of 2011 (Tables 10, 11 and 12)

Table 10. Sample data for glaucous gull and kittiwake samples collected from Svalbard

	Date	Location	North	East	Weight (g)	Sex
Glaucous gull - serum						
1-2011	07.06.2011		7855670	1157328	1740	M
2-2011	08.06.2011		7855889	1217847	1500	F
3-2011	10.06.2011		7859153	1157897	1440	F
4-2011	10.06.2011		7859721	1202255	1570	F
5-2011	10.06.2011		7900184	1204655	1400	F
6-2011	11.06.2001		7855069	1201082	2030	M
7-2011	11.06.2011		7856662	1215259	1410	F
8-2011	14.06.2011		7855069	1201082	1380	F
9-2011	14.06.2011		7855889	1217847	1760	M
10-2011	15.06.2011		7856014	1212482	1740	M
Kittiwake - whole blood						
K56-07		Kongsfjorden	788962	121950		
K57-07		Kongsfjorden	788962	121950		
K71-07		Kongsfjorden	788962	121950		
K75-07		Kongsfjorden	788962	121950		
K97-07		Kongsfjorden	788962	121950		

Table 11. Sample data for seal samples collected on Svalbard

	Date	Location	Location	Weight/length/abdominal measure of animal	Sex	Age (adult/juvenile)
Bearded Seal 1	23.08.2011	Billefjorden	0541800, 8732800	230 kg, 230 cm, 124 cm	M	adult
Bearded Seal 2	23.08.2011	Billefjorden	0541800, 8732800	240 kg, 248, 132 cm	F	adult
Bearded Seal 3	23.08.2011	Billefjorden	0541800, 8732800	190kg, 208, 113 cm	M	juvenile
Ringed Seal 1	24.08.2011	Nordenskjoldsbreen	0541600, 8732500	- , 113 cm, 83 cm	M	juvenile
Ringed Seal 2	27.08.2011	Kapp Ekholm	0533974, 8722260	ca 80kg, 155 cm, 88 cm	M	adult

Table 12. Sample data for egg samples collected on Svalbard

	Date	Location	North	East
Eider duck				
2010 - 1	16.06.2010	Kongsfjorden	789325	122186
2010 - 3	19.06.2010	Kongsfjorden	789325	122186
2010 - 9	16.06.2010	Kongsfjorden	789325	122186
20120 - 18	19.06.2010	Kongsfjorden	789325	122186
2010 - 26	17.06.2010	Kongsfjorden	789325	122186
2010 - 53	17.06.2010	Kongsfjorden	789325	122186
2010 - 56	17.06.2010	Kongsfjorden	789325	122186
2010 - 72	22.06.2010	Kongsfjorden	789325	122186
2010 - 82	19.06.2010	Kongsfjorden	789325	122186
2010 - 128	25.06.2011	Kongsfjorden	789325	122186
Common guillemot				
P1-2010	15.06.2010	Kongsfjorden	788962	121950
P2-2010	17.06.2010	Kongsfjorden	788962	121950
P3-2010	17.06.2010	Kongsfjorden	788962	121950
P4-2010	17.06.2010	Kongsfjorden	788962	121950
P5-2010	17.06.2010	Kongsfjorden	788962	121950
P1-2011	14.06.2011	Kongsfjorden	788962	121950
P2-2011	14.06.2011	Kongsfjorden	788962	121950
P3-2011	14.06.2011	Kongsfjorden	788962	121950
P4-2011	14.06.2011	Kongsfjorden	788962	121950
P5-2011	14.06.2011	Kongsfjorden	788962	121950

5.2 Analytical methodology

5.2.1 Alkylphenolic compounds

5.2.1.1 Biota extraction

Biota samples (cod liver, blue mussel and crab) were homogenized and 2 g aliquots taken. The samples were shaken for 2 hours with 50:50 cyclohexane:isopropanol and then centrifuged for 10 min at 3500 rpm. The solvent was decanted off and the procedure was repeated and the two extracts were combined. 0.5% NaCl acidified with 0.1% H_2SO_4 was added and samples were shaken and centrifuged. The upper organic phase was removed and the solvent was evaporated yielding a fatty extract. Approximately 0.15 g of fat was removed and deuterated internal standards were added prior to dissolving the extract in dichloromethane. The samples were then run on GPC (Waters 2695 separations module coupled to a Waters 486 absorbance detector at 254 nm) and separated using Envirogel columns (19 mm x 150 mm + 19 mm x 300 mm; Waters). The elution solvent was dichloromethane with a flow rate of 5 ml/min and the extract was collected between 12.45 min and 20.2 min. The extracts were solvent exchanged into cyclohexane and evaporated to less than 0.5 ml and derivatised as described below.

5.2.1.2 Sediment extraction

Dried sediment (1 g) and particulate (whole filter paper) samples were extracted following the same method with the only difference being the addition of copper powder to remove sulphur from the sediment samples during extraction. Deuterated internal standards were added to all samples prior to a double sonication extraction initially with hexane/acetone (60/40) followed by dichloromethane. The particulate extracts were solvent exchanged to cyclohexane and were derivatised using the method described for biota and analysed without further cleanup. The sediment extracts were solvent exchanged to methanol and water in preparation for cleanup using HLB solid phase extraction cartridges (Waters, Sweden). The extracts were then derivatised using the method described below and analysed without further cleanup.

5.2.1.3 Water extraction

Deuterated internal standards were added to water samples (2.5 L) before extraction using HLB solid phase extraction cartridges (Waters, Sweden). The analytes were eluted with dichloromethane and 10% ether in dichloromethane. Extracts were evaporated under nitrogen and derivatised using the method described below and analysed without further cleanup.

5.2.1.4 Derivatisation

Extracts were derivatized methods modified from those reported in the literature (Meier *et al.* 2005) using pentafluorobenzoyl chloride and pyridine at 60 °C overnight. Excess reagent was removed by adding 6 ml of 0.5 M sodium thiosulfate and 6 ml ultrapure water. Samples were then shaken and allowed to stand for approximately an hour. The water was removed and 6 ml of ultrapure water was added followed by shaking and removal of water phase again. The organic phase is dried with anhydrous sodium sulfate. Biota extracts were then evaporated to less than 0.5 ml and cleaned over silica SPE cartridges (1 g) with 10% ether in iso-hexane as the elution solvent. All sample extracts were evaporated to approx. 0.5 ml ready for analysis.

5.2.1.5 Analysis

All samples were analysed by GC-CI-MS (Hewlett Packard HP6890 and HP5973 Mass Selective Detector). The injector temperature was 280 °C with a 2 µl splitless injection with a purge time of 2 mins. Analytes were separated on a DB-5 column (15 m x 0.25 mm x 0.1 µm; Agilent) with a constant flow (1.1 ml/min) of helium. The GC temperature programme was an initial temperature of 80 °C and held for 2 mins, 5 °C /min to 225 °C, 25 °C/min to 325 °C and held for 2 mins. The chemical ionization gas was methane. The MS interface was 280 °C, the source 150 °C and the MS quad 106 °C. The SIM masses are shown in table 13, 2 masses were used for each compound. The uncertainty (RSD%) ranged from 1-10% for most compounds with the exception of OP1EO and NP1EO in crab which had uncertainties of 80%.

Table 13. Single ion monitoring ions (*m/z*) for the determination of alkylphenolic compounds and bisphenol A

Compound	Target Ion (*m/z*)	Qualifier Ion (*m/z*)
4tOP/4nOP	400	401
4tNP/4nNP	414	414
OP1EO	444	445
NP1EO	458	459
BPA	616	406

Table 14. Recovery data (%) for alkylphenolic compounds and bisphenol A

	Crab	Blue mussel	Trout	Cod liver	Polar cod	Capelin	Blood/plasma	Seal blubber
4tOP	84 ± 9	48	66	167	62 ± 3	132	157	104
4nOP	82 ± 2	61	72	108	111 ± 3	84	43	94
4nNP	102 ± 2	376	368	166	98 ± 3	205	43	106
OP1EO	333 ± 87	96	36	221	67 ± 1	175	78	112
NP1EO	225 ± 81	291	184	257	125 ± 10	216	99	134
BPA	176 ± 6	13	12	150	104 ± 1	129	90	56

5.2.2 Zineb and ethylenethiourea (ETU)

Water samples (2.5 L) were filtered through pre cleaned 0.45 µm GFC filters (Whatmann) in order to analyse dissolved and sorbed compounds separately.

5.*2.2.1 Zineb*

Zineb analysis was performed indirectly by hydrolyzing zineb to form carbon disulfide (CS_2) which is then measured by headspace-GC/MS. The method used here was modified from those in the literature (Jongen *et al.* 1991; Vuik *et al.* 1992). Aliquots (10 ml) of filtered water samples, sediment (1 g) or whole filter papers were placed straight into a headspace vial with 1 ml stannous chloride (66.5 mM $SnCl_2$ + 5 M HCl). The samples were heated at 70 ^oC for 1 hour. They were then equilibrated and agitated at 40 ^oC for 45 mins before headspace injection (2.5 ml).

Headspace-GC/MS (Hewlett Packard HP6890 and HP5973 Mass Selective Detector) using a DB-5 column (15 m x 0.25 mm x 0.1 µm; Agilent) with a constant flow (1.1 ml/min) of helium was used to determine CS_2. The injector temperature was 230 ^oC. The oven temperature program was 55 ^oC and held for 10 mins, 15 ^oC/min to 280 ^oC and held for 10 mins. The detector temperature was 280 ^oC.

5.2.2.2 Ethylenethiourea (ETU)

- *Water*

The method was modified from those reported in the literature (USEPA 1992; Fustinoni *et al.* 2005). Internal standard (ETU-d_4) was added to each water sample (2.5 L). Dichloromethane (DCM) (50 ml) was added to each sample which was then stirred under vortex for an hour. This extraction was then repeated for a double solvent extraction. The DCM extracts were dried over sodium sulphate and evaporated under nitrogen and solvent exchanged to iso-octane before derivatisation with BSTFA+1% TMCS. Extracts were derivatised at 60 ^{o}C overnight before analysis by gas chromatography-time of flight-mass spectrometry (GC-ToF-MS).

- *Particulates and sediment*

Internal standard (ETU-d_4) was added to each sediment (1 g) and particulate (whole filter paper). Samples were shaker extracted by double solvent extraction with hexane/acetone (60/40). Extracts were evaporated under nitrogen and solvent exchanged to iso-octane before derivatisation with BSTFA + 1% TMCS. Extracts were derivatised at 60 ^{o}C overnight before analysis by gas chromatography-time of flight-mass spectrometry (GC-ToF-MS).

- *Analysis*

GC-ToF-MS (GCT, Waters,) analysis was performed in electron impact mode (70 eV) using full scan (m/z 50-400 mD) and a source temperature of 180 ^{o}C. Gas chromatographic separation was carried out using a DB-5MS column (30 m x 0.25 mm x 0.25 µm) (J&W Scientific). The temperature program was as follows 60 ^{o}C for 2 mins, 15 ^{o}C/min to 280 ^{o}C, held for 5 mins. Splitless mode injection at 240 ^{o}C was used. The accurate masses used for compound determination were as follows ETU; 231.059+159.049+73.047 and ETU-d_4; 235.077+163.072+73.047. The uncertainty (RSD%) of ETU analysis was 10%

5.2.3 *N,N*-dimethylsulfamide (DMS)

Water samples (2.5 L) were extracted by solid phase extraction (SPE) following the addition of NDMA-d_6 as an internal standard. The SPE cartridges were connected in series for extraction. The first cartridge was Oasis HLB (Waters, Sweden) and this was connected to an active carbon cartridge. This allowed for cleaner extracts as many interfering compounds were retained by the HLB cartridge while the DMS and NDMA-d_6 passed through and were retained on the activated carbon cartridge. The analytes were eluted from the activated carbon with dichloromethane and evaporated and solvent exchanged to methanol and water in preparation for LC/MS/MS analysis.

NDMA-d_6 was added to sediment samples prior to shaker extraction with dichloromethane followed by ethyl acetate. The extracts were combined and evaporated under nitrogen and were cleaned up using the water extraction method described above.

Analytes were separated on a Acquity BEH C18 column (50 mm x 2 mm x 1.7 µm) with water (0.1% formic acid) and MeOH (0.1% formic acid) mobile phases using a gradient elution program from 99% water (0.1% formic acid) to 50% water (0.1% formic acid) with a flow rate of 0.6 ml/min. The mass spectrometry parameters are shown in table 15. The uncertainty (RSD%) of DMS analysis 13%.

Table 15. Mass transitions and mass spectrometer conditions for the determination of *N,N*-dimethylsulfamide (DMS)

	Parent Ion (*m/z*)	**Daughter ion (*m/z*)**	**Cone (V)**	**Collision energy (V)**
DMS	124.9	107.8/44.4	18	12
NDMA-d6	80.6	46.0	22	15

5.2.4 Second Generation Anticoagulant Rodenticides (SGARs)

Liver samples (0.5 g) were solvent extracted. Coumachlor was added to each sample as an internal standard. 200 µl of zinc chloride solution (10%) was added and mixed thoroughly before the addition of 1.5 ml ACN and further mixing and precipitation. Samples were centrifuged for 5 mins at 13000 rpm and the top layer removed. 1 ml heptane was added to the ACN extract and shaken before centrifugation again. The ACN layer was then removed in preparation for analysis by LC/MS/MS (Acquity UPLC – Quattro Premier XE (Micromass, Sweden)).

Analytes were separated on a Acquity BEH C18 column (50 mm x 2 mm x 1.7 µm) with water (10 mM ammonium acetate) and MeOH (10 mM ammonium acetate) mobile phases using a gradient elution program over a period of 3 minutes with a flow rate of 0.6 ml/min. The MS parameters, method recoveries and analytical uncertainty (RSD%) are shown in table 16.

Table 16. Mass transitions and mass spectrometer conditions for the determination of second-generation anticoagulant rodenticides

Compound	**Mass Transition (*m/z*)**	**Cone Voltage (V)**	**Collision Energy (V)**	**Method Recovery and Uncertainty (%)**
Coumachlor (Int Std)	343>163 343>285	+35	17	66 ± 1
Flocoumafen	543>159	+45	40	59 ± 2
Difethialone	539>335 541>337	+45	25	54
Difenacoum	445>179	+45	32	64.6 ± 0.5
Bromadiolone	525>250 527>250	-50	37	74 ± 3
Brodifacoum	523>335 525>337	+45	22	53.5 ± 2.5

5.2.5 Arctic samples

5.2 5.1 Extraction

Biota samples (seal blubber, egg, whole polar cod and capelin) were homogenized and 1 to 5 g aliquots (500 µl aliquots for whole blood and plasma) were taken for analysis. The samples were shaken for 2 hours following the addition of 50:50 cyclohexane:isopropanol and then centrifuged for 10 min at 3500 rpm. The solvent was decanted off and the procedure was repeated and the two extracts combined. NaCl (0.5% acidified with 0.1% H_2SO_4) was added and the samples shaken and centrifuged before the upper solvent phase was removed and reduced in volume to yield a fatty extract.

Approximately 0.15 g of the fat (seal blubber, egg, whole polar cod, whole capelin) was taken and an internal standard (PCB 30, 53 and 204) added prior to dissolving the extract in dichloromethane for

cleanup by gel permeation chromatography (GPC; Waters 2695 separations module coupled to a Waters 486 absorbance detector at 254 nm) fitted with Envirogel columns (19 mm x 150 mm + 19 mm x 300 mm; Waters). The mobile phase was dichloromethane with a flow rate of 5 ml/min and the extract was collected between 12.45 and 20.2 min. The extracts were exchanged into cyclohexane and reduced in volume by nitrogen evaporation to less than 0.5 ml. Whole blood and plasma did not require GPC cleanup.

Extracts for the analysis of dacthal, chlorpyrifos, trifluralin and methoxychlor were then cleaned up further using silica SPE columns (1 g Si, Isolute). The analytes were eluted with 2 ml hexane followed by 5 ml hexane:dichloromethane (50/50). The extracts were reduced in volume under nitrogen to approximately 100 µl in preparation for analysis by GC/MS. The extracts for the analysis of pentachlorophenol required no additional silica cleanup and were derivatised prior to analysis.

5.2.5.2 Pentachlorophenol derivatisation

Extracts were derivatized using methods modified from those reported in the literature (Meier *et al.* 2005) using pentafluorobenzoyl chloride and pyridine at 60 °C overnight. Excess reagent was removed by adding 6 ml of 0.5 M sodium thiosulfate and 6 ml ultrapure water. Samples were then shaken and allowed to stand for about an hour. The water was removed and 6 ml of ultrapure water was added followed by shaking and removal of water phase again. The organic phase was dried with anhydrous sodium sulfate. Biota extracts were then reduced in volume to less than 0.5 ml and cleaned up over silica SPE columns (1 g Si, Isolute) with 10% ether in iso-hexane elution solvent. All sample extracts were evaporated to approx. 0.5 ml ready for analysis.

5.2.5.3 Analysis

Chlorpyrifos, dacthal, methoxychlor and trifluralin and were analysed by GC-EI-MS (Hewlett Packard HP7890A and HP5975C inert XL EI/CI Mass Selective Detector). The injector temperature was 250 °C with a 2 µl splitless injection with a purge time of 2 mins. Analytes were separated on a DB-5 column (30 m x 0.25 mm x 0.25 µm; Agilent) with a constant flow (1.1 ml/min) of helium. The GC temperature programme was an initial temperature of 60 °C and held for 2 mins, 5 °C /min to 250 °C, 15 °C/min to 290 °C and held for 5 mins. The MS interface was 280 °C, the source 230 °C and the MS quad 150 °C. 2 masses were used for each compound (Table 17).

Pentachlorophenol was analysed by GC-CI-MS (Hewlett Packard HP6890 and HP5973 Mass Selective Detector). The injector temperature was 280 °C with a 2 µl splitless injection with a purge time of 2 mins. Analytes were separated on a DB-5 column (15 m x 0.25 mm x 0.1 µm; Agilent) with a constant flow (1.1 ml/min) of helium. The GC temperature programme was an initial temperature of 80 °C and held for 2 mins, 5 °C /min to 225 °C, 25 °C/min to 325 °C and held for 2 mins. The chemical ionization gas was methane. The MS interface was 280 °C, the source 150 °C and the MS quad 106 °C. The SIM masses are shown in table 14 and the recovery and uncertainty (RSD%) are summarized in table 18.

Table 17. SIM ions monitored for the analysis of herbicides

Pesticide	Ions analysed (m/z)
Dacthal	301 + 332
Chlorpyrifos	314 + 349
Trifluralin	306 + 264
Methoxychlor	227 + 344
Pentachlorophenol (derivatised)	265 + 267

Table 18. Recovery and uncertainty data (%) for current use pesticide analysis

Matrix	Dacthal	Chlorpyrifos	Trifluralin	Methoxychlor	Pentachlorphenol
Polar cod	102 ± 1	119 ± 3	72 ± 1	142 ± 7	104
Whole capelin	67 ± 14	106 ± 0	57 ± 11	116 ± 2	216
Seal blubber	67 ± 14	106 ± 0	57 ± 11	116 ± 2	103
Whole blood/serum	88	159	97	126	106

6 RESULTS

6.1 Alkylphenolic compounds and bisphenol A

Low ng/g levels of 4-NP were detected in the crab samples from both Puddefjorden and Skånevikfjorden and t-NP was also detected in Puddefjord crab samples (Figure 9, Table 27). There was no significant difference between the concentrations of 4-NP in the different locations and the concentration range in Puddefjord was 5.3-6.6 ng/g compared to <2-4.8 ng/g in Skånevikfjorden.

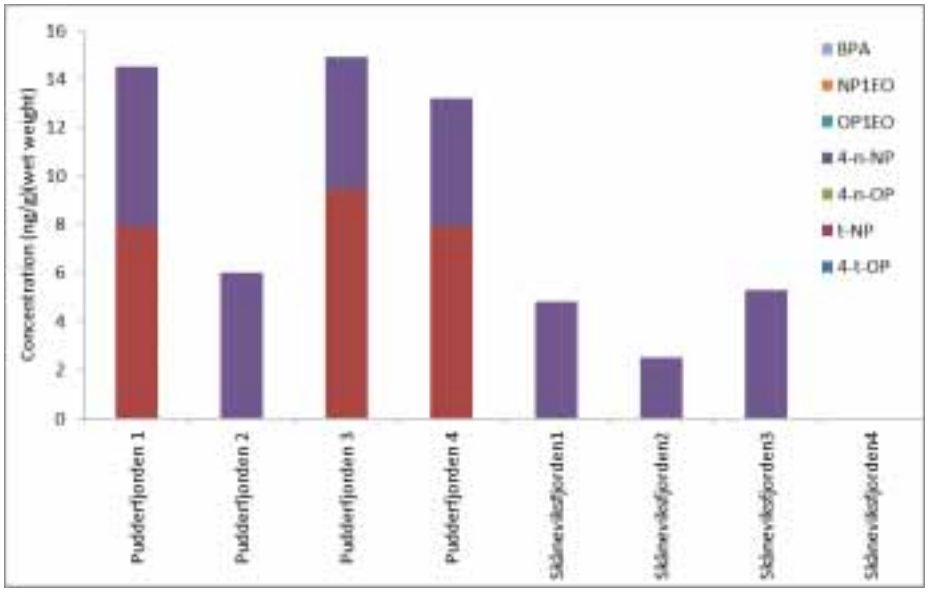

Figure 9. Concentration of alkylphenolic compounds and bisphenol A in crab (ng/g) (wet weight)

As with the crab, 4-NP is the dominant compounds in cod liver from Byfjord and was detected in all 15 samples from 1.5-114 ng/g (median 5.3 ng/g; Figure 10, Table 26). However, tNP was detected at much higher concentrations with a median concentration of 74 ng/g, and a maximum concentration of 383 ng/g in one sample. NP1EO was also detected in the majority of samples with a median concentration of 21.3 ng/g (<7-121 ng/g). OP oligomers were detected less frequently and at lower concentrations. BPA was detected in 75% of the Byfjord cod liver samples in the concentration range <4 ng/g – 46.3 ng/g.

No compounds were detected in blue mussels in either Puddefjorden or Skånevikfjorden (Table 28).

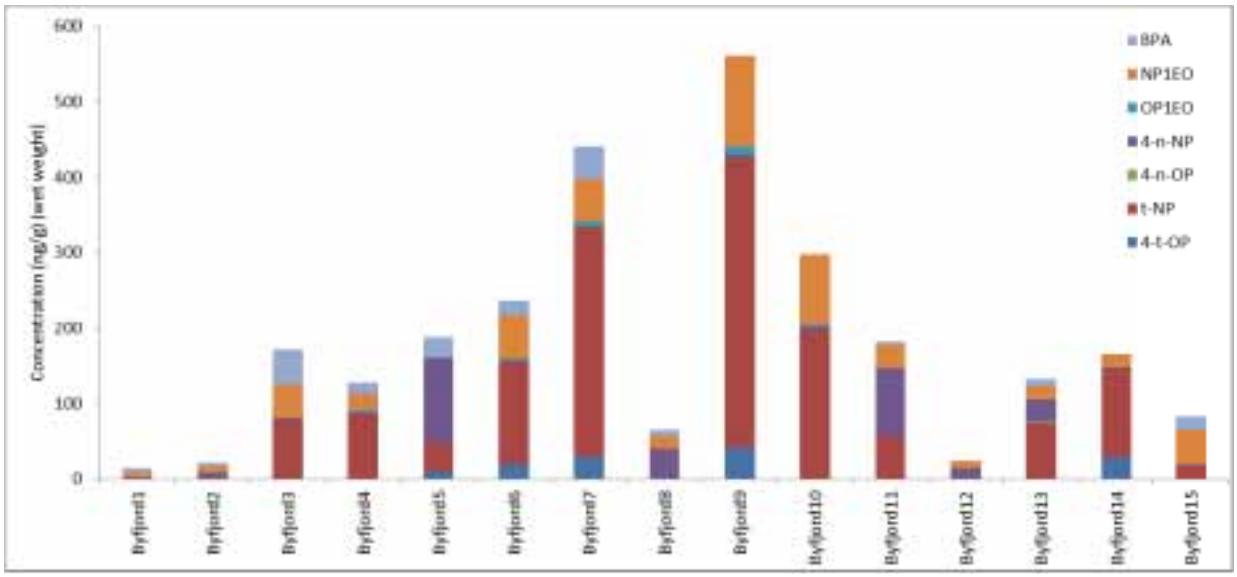

Figure 10. Concentration of alkylphenolic and bisphenol A compounds in cod liver (ng/g) (wet weight)

Only 3 compounds were detected in freshwater samples, 4-t-OP, OP1EO and t-NP. 4-t-OP and t-NP were detected in all water samples (Figure 11, Table 22). The freshwater reference location, Spjeldsjøen, had median concentrations of 7.9 and 5.9 ng/L for 4tOP and tNP respectively, compared to the marine reference location, Skånevikfjorden, with median concentrations of 15 ng/L for both compounds. The median concentrations for both compounds were higher in Mjøsa and Puddefjorden. In Mjøsa the medians were 30 and 26 ng/L for 4tOP and tNP respectively, compared to 50 and 82 ng/L in Puddefjorden. 4NP was also detected in all Puddefjorden water samples (5.8-111 ng/L). The only other compound detected with any frequency was OP1EO which was detected in all water samples except those from Spjeldsjøen. The concentrations were all lower than for the APs with median concentrations of 0.02, 0.39 and 0.39 ng/L for Mjøsa, Puddefjorden and Skånevikfjorden respectively.

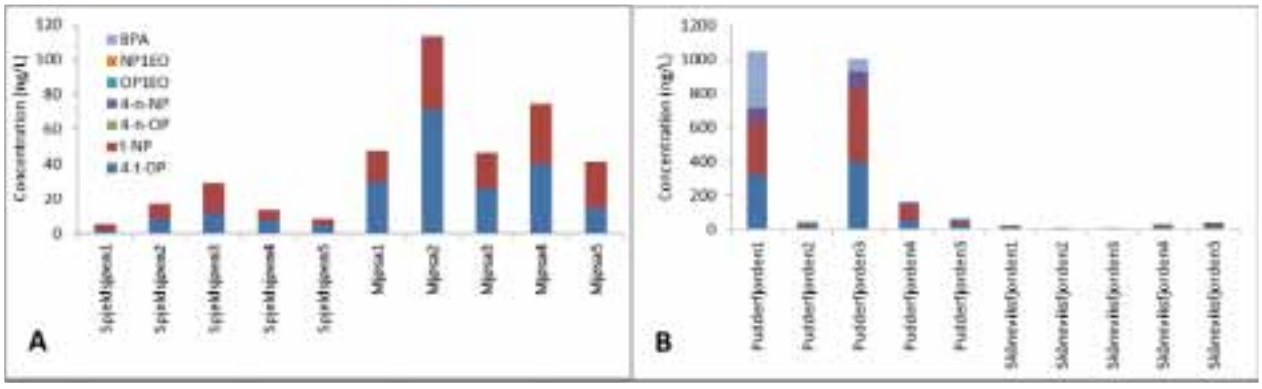

Figure 11. Concentration of alkylphenolic compounds and bisphenol A in water (ng/L) A. freshwater environment. B. Marine environment

BPA was detected with more frequency in particulate samples than in water or sediment (Figure 12, Table 23). The highest concentrations were detected in the freshwater particulate samples, both from Mjøsa and from the reference location, Spjeldsjøen. The median concentrations were 120 and 139

pg/L in Spjeldsjøen and Mjøsa respectively and in Puddefjorden and Skånevikfjorden the median concentrations were much lower at 0.45 and 0.04 pg/L respectively. As was observed with in the water samples, the most significant concentrations were observed for 4-t-OP and t-NP, and again were much higher in the freshwater environment than in the marine environment. In the reference locations, Speldsjøen and Skånevikfjorden, the concentration ranges for 4tOP were 193.8-472.2 pg/L and 1.5-3.9 pg/L respectively. These data compare to similar concentration ranges in the theoretically more contaminated locations. Concentration ranges of 255-363 pg/L in Mjøsa and 1.3-3.7 pg/L in Puddefjorden. tNP was also defected in higher concentration in the freshwater than the marine environment. The median particulate values were 506 and 721 pg/L in Speldsjøen and Mjøsa respectively compared to only 2 pg/L in Puddefjorden. OP1EO and NP1EO were detected in the freshwater reference location at higher concentrations than any of the other 3 locations. The median concentrations are shown in Table 19. No NP1EO was detected in Mjøsa compared to a maximum concentration of 146 pg/L in Speldsjøen. Puddefjorden also contained lower concentrations than the reference location, Skåvevikfjorden.

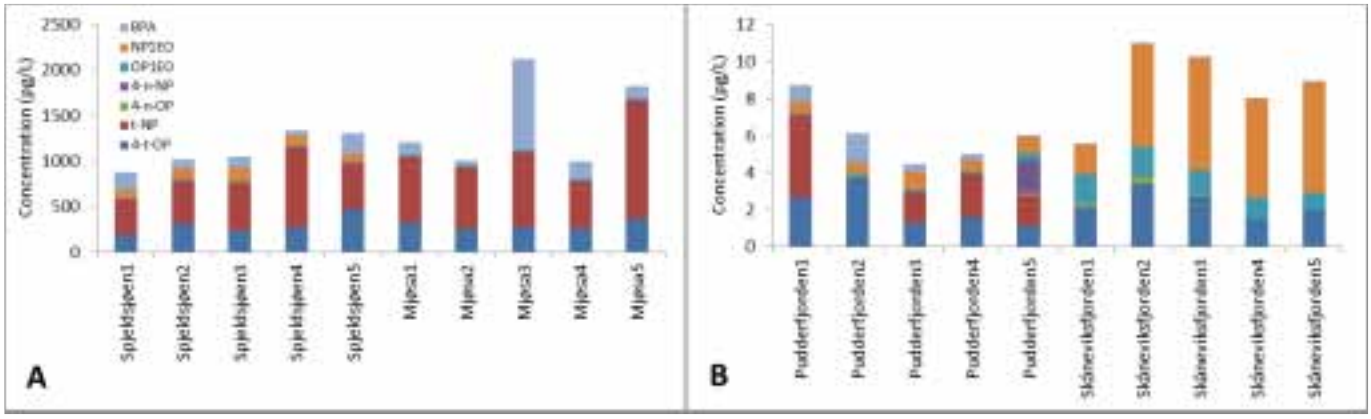

Figure 12 . Concentration of alkylphenolic compounds and bisphenol A in particulates (pg/L) A. freshwater environment. B. Marine environment

Table 19. Median concentration of ethoxylates in particulates (pg/L)

	OP1EO	NP1EO
Speldsjøen	15.8	108.5
Mjøsa	18.0	-
Puddefjorden	0.13	0.64
Skånevikfjorden	1.31	5.55

In sediment samples, the most frequently detected compounds were tNP, OP1EO and its degradation product 4nOP (Table 24). tNP and OP1EO were detected in the highest concentrations, and were highest in Speldsjøen, as was observed with the particulate samples. The median concentration of tNP in Speldsjøen was 245 ng/g compared to 158 ng/g in Mjøsa. In Puddefjorden the median concentration was 154 ng/g and was 44 ng/g in Skånevikfjorden. In Spjeldsjøen the median concentration of OP1EO was 20 ng/g and its degradation product 4nOP had a median concentration of 0.3 ng/g. OP1EO was also detected in all sediment samples collected from Mjøsa (1.9-13.6 ng/g) and Puddefjorden (3.6-9.6 ng/g), where 4nOP was also detected with a median concentration of 0.46 ng/g. BPA was detected in 4

out of 5 Puddefjorden samples with a concentration range of <1-20 ng/g, otherwise it was only detected in 2 other samples, 1 from Mjøsa and 1 from Speldsjøen, both a low ng/g concentrations.

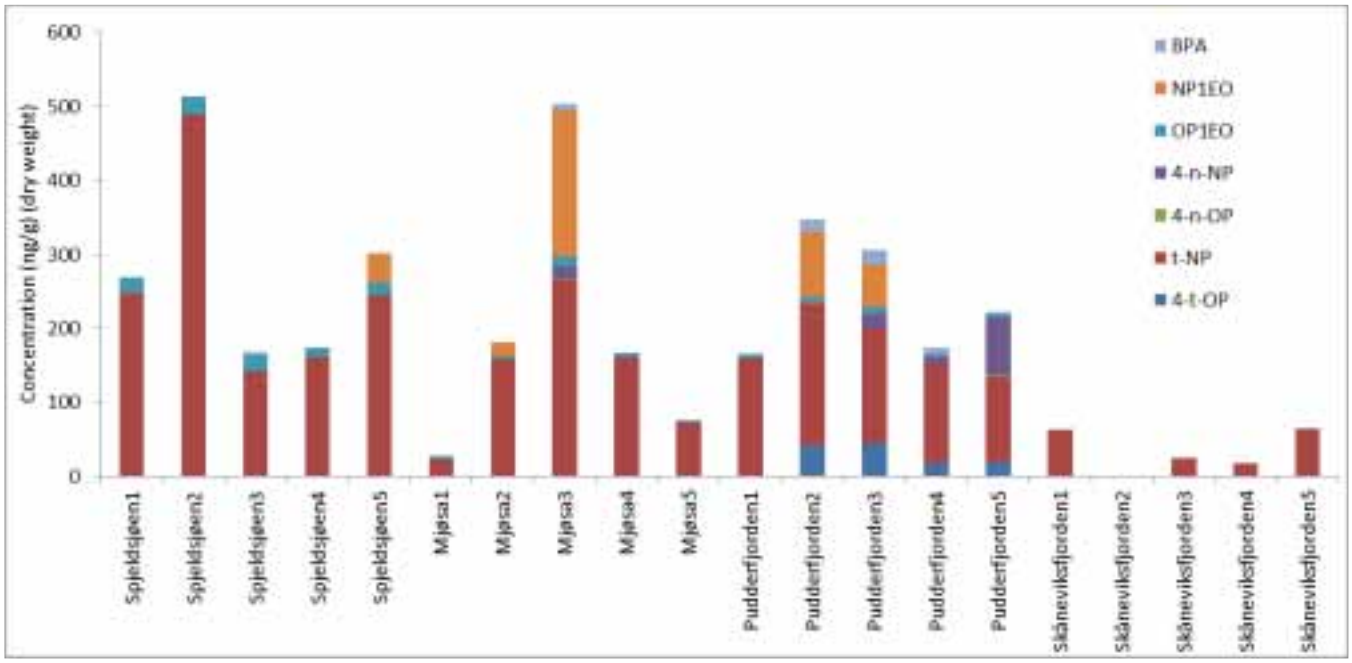

Figure 13. Concentration of alkylphenolic compounds and bisphenol A in sediments (ng/g) (dry weight)

The Arctic samples of whole polar cod and capelin, seal blubber, kittiwake and glaucous gull blood/plasma, and eider and common guillemot eggs collected from the Arctic contained levels of 4nOP, tOP, OP1EO, nNP, tNP and NP1EO below the limits of detection for each analyte (Tables 35-38).

6.2 Zineb and ethinylthiourea (ETU)

Neither zineb nor ETU were detected in the dissolved aqueous phase of the water samples collected (Table 29). In the particulate phase ETU was detected at concentrations of between 1.3 and 2.2 ng/L in the samples collected from Bestumkilen, Oslo and between 3.3 and 15.5 ng/L in the samples collected at Kviturspollen, Bergen (Figure 14, Table 30). The concentrations in the particulate samples collected from Skånevikfjorden were below the limit of detection (<1 ng/L) as were the levels of zineb in all particulate samples. The concentrations of zineb and ETU were also below the limit of detection in the sediment samples collected (Table 31).

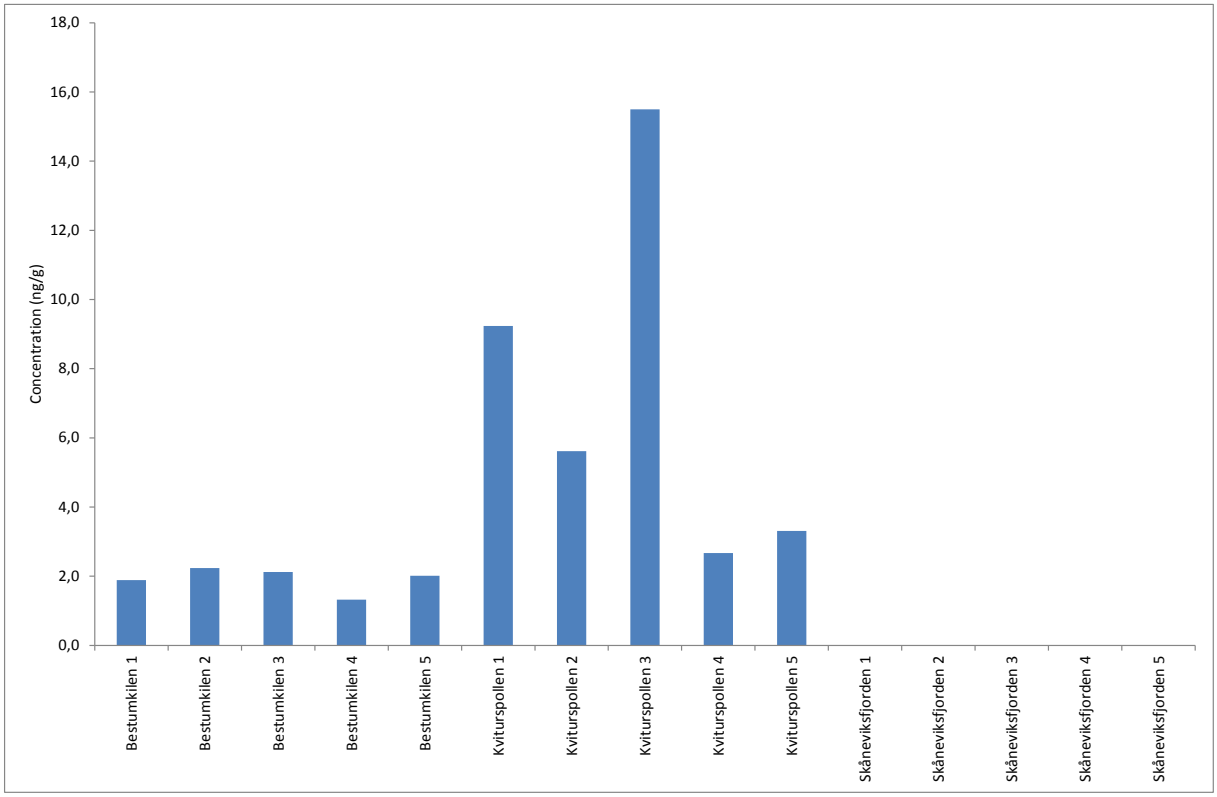

Figure 14. Concentrations of ethylenethiourea in particulates (ng/L)

6.3 *N,N*-dimethylsulfamide

N,N-dimethylsulfamide (DMS) was detected at aqueous concentrations of between 456 and 774 ng/L in the Østensjø samples and 104 and 540 ng/L in the Holmendammen samples (Figure 15, Table 32). The levels in the control lake of Skodøltjenn were below the limit of detection of 5 ng/L. Sediment concentrations of DMS were below 5 ng/g in all of the sediments samples collected (Table 33).

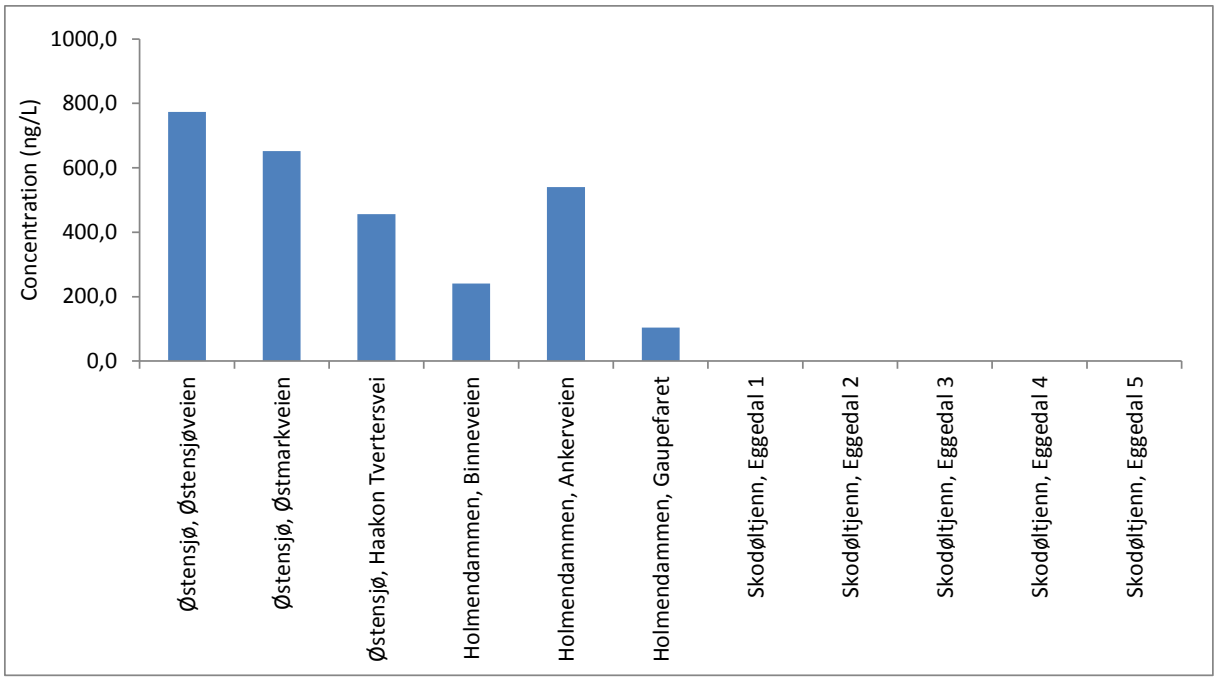

Figure 15. Concentrations of *N*,*N*-dimethylsulfamide (DMS) in water (ng/L)

6.4 Second generation anticoagulant rodenticides (SGARs)

The SGARs were only detected in livers of the golden eagle and eagle owl samples provided by the Norwegian School of Veterinary Science (Figure 16). None of the selected rodenticides were detected above the limit of detection (<2-5 ng/g) in the osprey, perergrine falcon and gyrfalcon livers (Table 34). Four of the five selected rodenticides were detected; brodifacoum, bromadiolone, difenacoum and flocoumafen. Brodifacoum was determined in 7 of the 16 golden eagle livers with detectable concentrations of between 22 and 154 ng/g present. Bromadiolone was detected in both golden eagle (7/16 samples) and eagle owl livers (4/8 samples) with measureable concentrations of between 11 and 110 ng/g and 74 and 158 ng/g respectively. Difenacoum was only detected in 2 of the eagle owl livers at a level of 39 and 181 ng/g. Flocoumafen was detected in 2 golden eagle livers (15 and 117 ng/g) and a single eagle owl sample.

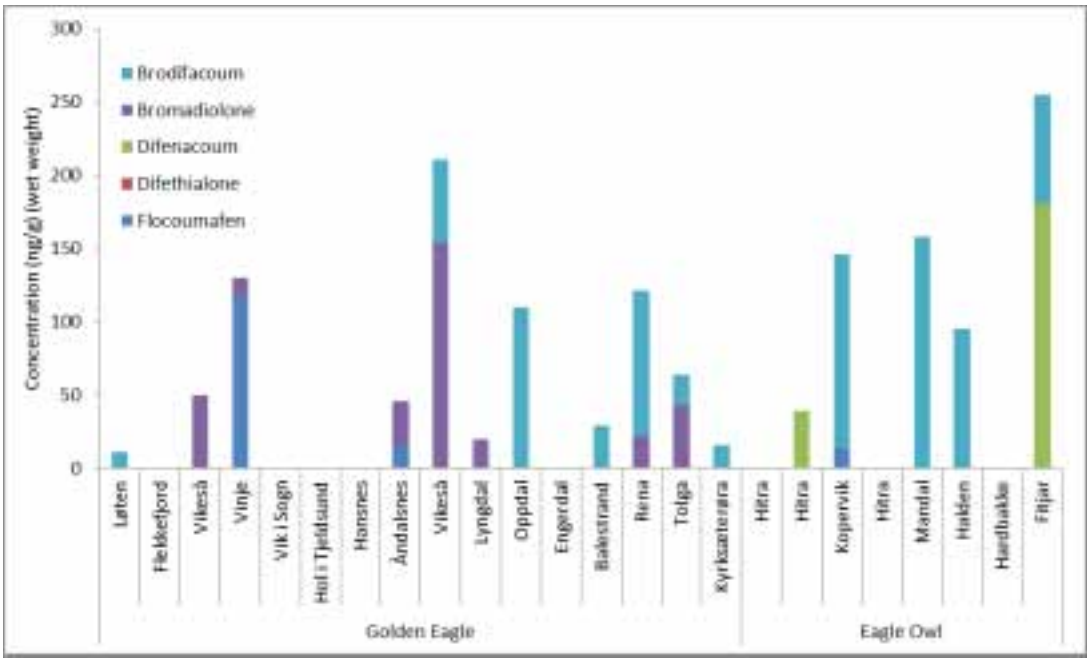

Figure 16. Concentrations of second-generation anticoagulant rodenticides (SGARs) in the livers of golden eagles (*Aquila chrysaetos*) and eagle owls (*Bubo bubo*)

6.5 Pesticides in the Arctic

The samples of whole polar cod and capelin, seal blubber, kittiwake and glaucous gull blood/plasma and eider duck and common guillemot eggs, collected from the Arctic, typically contained levels of chlorpyrifos, dacthal, methoxychlor, pentachlorophenol, trifluralin, that were below the limits of detection for each analyte. A single sample of ringed seal blubber contained 1.4 ng/g of chlorpyrifos (Tables 40-44).

7 DISCUSSION

7.1 Alkylphenolic compounds and bisphenol A

It is widely known that wastewater treatment works effluent is a significant point source of alkylphenols to the aquatic environment. Puddefjorden off Bergen has many potential sources of alkylphenolic compounds as it is a sewage impacted urban environment. Another study in a similar sewage impacted urban estuary in New York detected short chain APEOs and APs in sediment and water samples and effluent discharge was identified as a significant point source (Ferguson *et al.* 2001). NP and NP1EO concentrations were higher than OP and OP1EO which is a distribution pattern seen in many other studies and reflects surfactant usage patterns.

The majority of studies do not differentiate between the soluble and sorbed fraction when analysing water samples, whereas in this study we have differentiated between the two phases. There is a clear difference in the distribution patterns between the particulate and dissolved fractions in the locations sampled in Norway.

Sorption of NP onto marine sediments increases with increasing salinity and organic carbon content (Yang *et al.* 2011). Temperature is also a determining factor and as the hydrophobicity of NP decreases with an increasing temperature, sorption will therefore reduce. In this study the effects of TOC were clear in the freshwater sediments with total alkylphenolic compounds concentration clearly correlating with TOC concentrations (Figure 17). There was little correlation in the marine sediments indicating that there are other parameters, such as salinity effecting sorption processes.

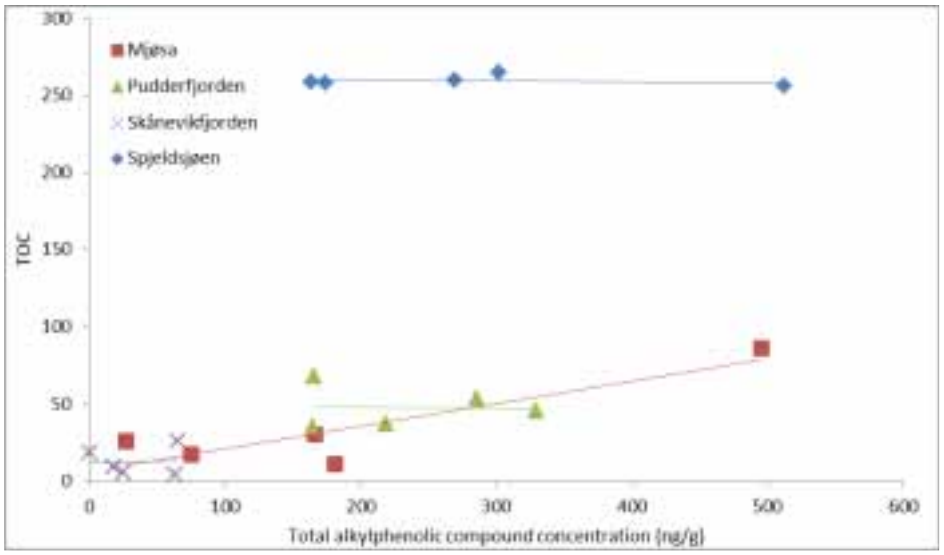

Figure 17. Correlation of total alkylphenolic compound concentration with total organic carbon (TOC) content in sediments

Degradation occurs more rapidly in the water column than in sediments (Ying *et al.* 2002), which may explain the presence of OP and NP in Spjeldsjøen where NP1EO and OP1EO were also detected.

The $PNEC_{sediment}$ value for tNP is 18 ng/g (Klif, 2011; Table 17) and this was exceeded in all but 2 of the sediment samples analysed. In the freshwater sediment samples the $PNEC_{sediment}$ was exceed by almost an order of magnitude in 7 out of 10 samples. The sediments from Skånevikfjord reference location contained no other alkylphenolic compounds but exceeded the $PNEC_{sediment}$ value in 3 out of the 5 samples. Where the $PNEC_{sediment}$ is exceeded it is possible that the presence of tNP in these samples is sufficiently high to pose a risk to sediment dwelling organisms. The $PNEC_{sediment}$ value for OP was not exceeded but if the same value is applied to OP1EO where no value is available, it was exceed 9 out of 10 samples in the freshwater sediment samples. All sediment samples from Puddefjorden contained levels of OP1EO were above the OP PNEC of 3.3 ng/g (Klif, 2011). The greater dilution factor in the marine environment than the lakes, in particular Spjeldsjøen with a surface area of only 0.32 km^2, and a maximum depth of 9 m could explain some of the differences in the measured concentrations in the two different environments.

Table 20. Predicted no effect concentrations (PNEC) values for nonylphenol, octylphenol and bisphenol A (Klif, 2011)

	NP	**OP**	**BPA**
$PNEC_{water}$ (µg/L)	0.33	0.12	1.6
$PNEC_{sediment}$ (µg/kg)	18	3.3	11

The effects of NP from produced water on Atlantic Cod have been investigated (Meier *et al.* 2007). Exposure to APs had a major effect on the levels of natural steroids in the fish although the cod were exposed to higher concentrations than those measured in this study. Other studies have investigated the effects of NP and BPA on Cod and concluded that BPA has a similar mode of action and similar endocrine disrupting effects as NP (Larsen *et al.* 2006; Olsvik *et al.* 2009).

The trout from Mjøsa and Spjeldsøen showed no contamination, with the exception of 3 trout form Mjøsa with low ng/L concentrations of tNP. Carp from the Great Lakes in Canada had concentrations of NP over 1000 times higher than these 3 trout samples (Schmitz-Afonso *et al.* 2003). Concentrations of NP1EO were up to 2500 ng/g in the Great Lakes, compared to <3 ng/g in Mjøsa.

The concentrations of 4NP and tNP in blue mussels samples from Puddefjorden and Skånevikfjorden were all less than detection limits whereas concentrations in Korea were up to 300 ng/g. The Korean sediment concentrations were comparable to the freshwater sediment concentrations in this study but significantly higher than in the marine samples (Wang *et al.* 2007). The results from this study support the less than detection limit results from previous studies in cod liver and blue mussel (Green *et al*, 2009)

In German surface waters BPA was measured at very low concentrations, 0.5-229 ng/L, and at higher concentrations in sediments, 10-190 µg/kg (Fromme *et al.* 2002).

The samples of whole polar cod and capelin, seal blubber, kittiwake and glaucous gull blood/plasma, and eider and common guillemot eggs collected from the Arctic contained levels of 4nOP, tOP, OP1EO, nNP, tNP and NP1EO below the limits of detection for each analyte (Tables 31-35).

7.2 Zineb and ethylenethiourea (ETU)

Zineb has been used as an antifouling biocide since the development of alternative organic antifouling booster biocides to replace tributyltin (Thomas 2001); however, there are very few studies available on the environmental occurrence of zineb following its release from antifouling paints. It is, however, known that upon release from a painted surface zineb is rapidly hydrolyzed (< 96h) to ETU, 5,6-dihydro-3H-imidazo (2,1-c)-1,2,4-dithiazole-3-thione (DIDT) and ethelyne diisothiocyanate (EDI), which clearly explains the absence of zineb in the collected samples (Thomas 2001). The removal of ETU from natural waters is rapid with photolysis in the presence of photosensitizers (i.e. dissolved organic matter) resulting in reported half-lives of between 24 and 96 hrs (Xu 2000), although photolysis is not always a relevant removal pathway as it requires clear water for sunlight to penetrate. The presence of ETU in the particulate phase is less clear since it is a very polar compound with a reported log K_{OW} of -0.66 (Vershueren 1996). Its presence may possibly be explained by the high affinity of zineb for particulate material which is thought to facilitate its rapid transformation to ETU. It is likely that due to the speed of the transformation of zineb to ETU that the ETU levels measured on the particulates reflect the levels of zineb in the particulate phase at the point of particle isolation. It is therefore recommended that in future the monitoring of zineb be performed through determining the concentration of ETU. Values in the draft risk assessment for zineb under the EU biocides directive indicate that the levels of ETU detected on suspended particulate matter may indicate a risk to the marine environment.

7.3 *N,N*-dimethylsulfamide (DMS)

Release of the fungicides tolyfluanid and dichlofluanid and subsequent rapid microbial transformation leads to the polar transformation product DMS. Elevated levels of DMS in surface waters impacted by these fungicides is therefore not surprising and the levels determined in the surface waters of Østensjø and Holmendammen are similar to those that have been reported in Germany (up to 2,500 ng/L in urban surface waters; Schmidt et al. 2008). DMS has a negative octanol-water partition coefficient (-0.8) and is therefore unlikely to bioaccumulate or bind to sediments.

The risk associated with the presence of DMS in surface waters has been evaluated by the EC (EC 2006). The drinking water quality standard for DMS is 100 ng/L and all samples from Østensjø and Holmendammen exceed this. However, since these waters are not used for drinking water the levels do not pose an unacceptable risk to humans. DMS transforms to N-nitrosodimethylamine (NDMA) when drinking water is ozonated and this is of particular concern for drinking water quality. It is therefore recommended that future screening of DMS include lakes used for supplying drinking water.

DMS has a very low aquatic toxicity with a $PNEC_{Water}$ of 10 mg/L (EU 2009). It can therefore be assumed that there is no risk to the aquatic environment from the levels of DMS detected.

7.4 Second generation anticoagulant rodenticides (SGARs)

Brodifacoum and bromadiolone were the most frequently detected SGARs in this screening study as has previously been reported in studies on the occurrence of SGARs in raptor livers (Thomas *et al.* 2011; Sanchez-Barbudo *et al.* 2012). The residues measured in the Norwegian species are in the same orders of magnitude as those reported in the livers of European and North American species (Peregrine falcon, eagle owl and golden eagle) (Stone *et al.* 2003; Albert *et al.* 2010; Sanchez-Barbudo *et al.* 2012). The frequency of exposure is also similar to those reported for other raptor species with SGARs being detected in approximately 70% of the livers of golden eagles and 50% of the eagle owls examined in this study. These limited data would therefore suggest that the level of SGAR exposure in Norway is similar to other countries and that the golden eagle and eagle owl food chain is contaminated with SGARs. Little can be concluded from the absence of SGARs in the other raptor species (osprey, gyrfalcon and peregrine falcon) due the few samples analysed. A spatial evaluation of the data suggests that there is no clear pattern with regards to the location where the birds were collected and whether their livers contained SGARs (Figure 18), although a simple visual examination of Figure 18 suggests that the majority of the raptors analysed were from the more heavily populated areas of Norway.

The liver concentrations of SGARs associated with adverse effects and/or mortality has not been defined for most raptor species and establishing liver toxicity threshold values for SGARs is not a simple task (Stone *et al.* 2003; Walker *et al.* 2008). The main problems are associated with the uncertainties linked to analysis and the difference in species sensitivities to SGARs. The only residue toxicity data that have to date been reported for SGARs in raptor livers is a >100 to 200 ng/g "potential lethal range" for barn owls (Newton *et al*. 1998; 1999). This value is reported to provide a range of concern for potential toxicity and does not provide an indication or likelihood for effects. A probabilistic characterization of toxic liver concentrations in selected raptors (barred owl, barn owl, eagle owl and red-tailed hawk) suggests significant differences in species sensitivities and that there is a significant likelihood of poisoning in below 100 ng/g (Thomas *et al*. 2011). The EU Biocides Directive 98/8/EC (http://ec.europa.eu/comm/environment/biocides/index.htm) presents toxicity data

and PNEC values (Table 21) but the majority of the data is based on non-raptor species and the different between species sensitivities make it difficult to relate to the raptor species used in this study. In the absence of specific golden eagle and eagle owl toxicity thresholds for SGARs then any risk assessment is difficult. If >100 ng/g is assumed as a potential lethal range, accepting that there may be significant likelihood of poisoning below this level, 7 of the 24 (30 %) golden eagle and eagle owl liver samples analyzed contain total SGAR residues above this level. Further estimation of the potential mortality impact on the sampled raptor populations is not possible.

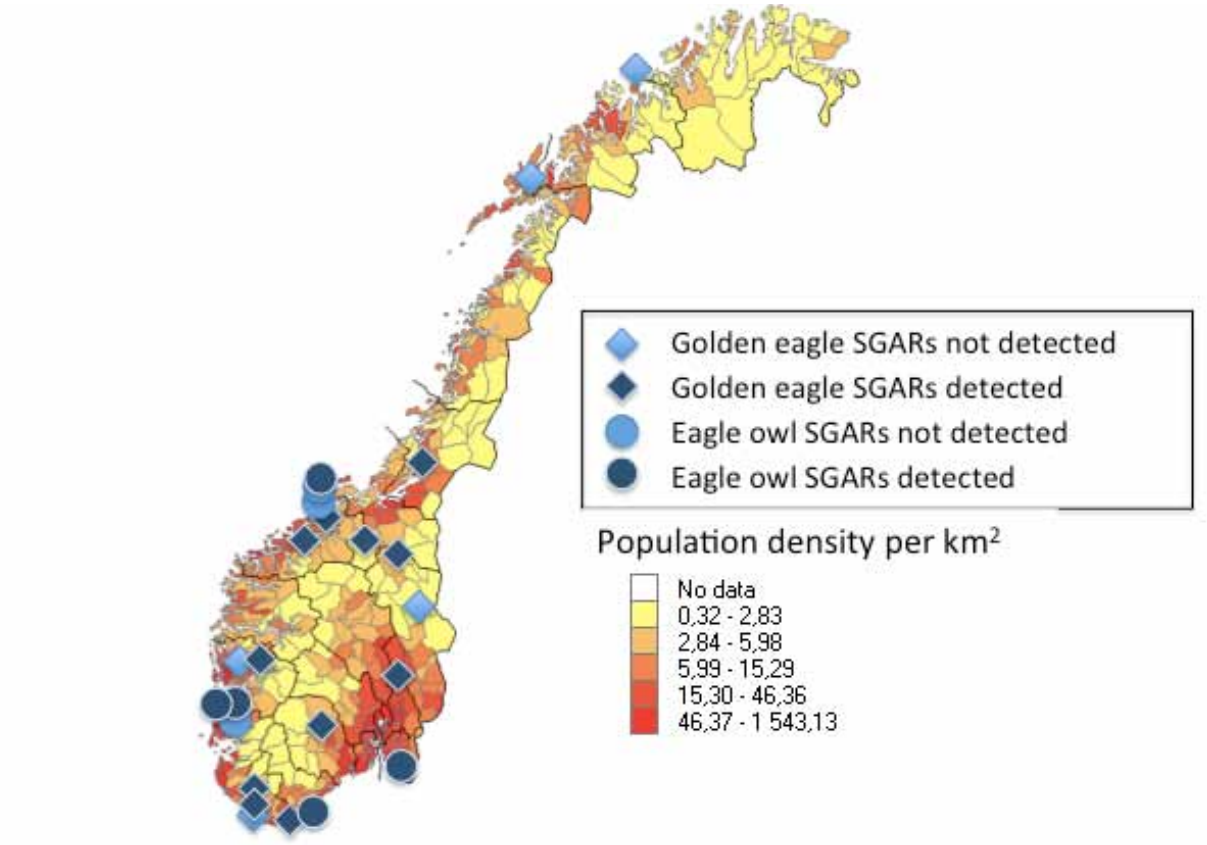

Figure 18. Collection locations for the golden eagles (*Aquila chrysaetos*) and eagle owls (*Bubo bubo*) analysed in this study along with human population density data (www.ssb.no)

Table 21. Toxicity data summary from the EU Biocides Directive 98/8/EC (http://ec.europa.eu/comm/environment/biocides/index.htm)

Rodenticide	Toxicity threshold	$PNEC_{oral}$
Brodifacoum	LC_{50} < 0.72 mg/kg food Laughing gull (Leucophaeus atricilla)	0.013 µg/kg diet 0.0128 µg/kg bw/d*
Bromadiolone	LD_{100} 0.056 mg/kg bw/d Great horned owl (*Bubo virginianus)*	0.75 µg/kg diet 0.19 µg/kg bw/d
Difenacoum	-	0.5 µg/kg diet 0.1 µg/kg bw/d
Difethialone	LC_{50} 18.9 mg/kg food Mallard duck (*Anas platyrhynchos)*	0.33 µg/kg diet 0.04 µg/kg bw/d
Flocoumafen	LD_{50} 24 mg/kg bw Mallard duck +(*Anas platyrhynchos)*	> 2.1 µg/kg diet > 0.25 µg/kg bw/d

*µg/kg bw/d is defined as µg/kg body weight/ day

7.5 Pesticides in the arctic

There is concern over the occurrence of the selected CUPs in Artic biota since their presence shows that they can be transported over long distances and accumulate in the food web (Muir and de Wit 2010). In the current study, even though sensitive analytical methods were used the levels of CUPs in all of the samples analysed was below detection limits. It is acknowledged that there are very limited data for CUPs in Arctic biota and that there is widespread concern over their occurrence (Muir and de Wit 2010). A recent review of the available data on the occurrence of CUPs in the Arctic reported that methoxychlor has been previously detected at the highest levels in snowcrab muscle tissue and liver (29 and 12 ng/g lw respectively) and that the median levels of methoxychlor in the other fish and invertebrate studies was typically <5 ng/g lw (Vorkamp *et al*. 2004). As in this screening study, methoxychlor was not detected in capelin. Although Vorkamp *et al*. (2004) studied different sea bird and sample matrices, the levels of methoxychlor were also below the limits of detection in the seabirds analysed. The limited data available for dacthal and chlorpyrifos in Arctic fish show levels of less than 1 and 10 ng/g lw detected in samples collected from Alaska (Landers *et al*. 2008). This study has clearly added to the limited data available on CUPs in Arctic biota, however as recently highlighted by Muir and de Wit there is a need for a more systematic approach to assess whether CUPs might be accumulating in the Arctic since the limited body of data that are currently available is inconclusive.

8. CONCLUSIONS

Concentrations of OP, NP1EO and BPA measured in the samples were all lower than reported threshold values. The tNP $PNEC_{sediment}$ value was exceeded for in all but 4 of the sediment samples collected suggesting that there is a potential risk to sediment dwelling organisms in these areas. OP1EO was also detected in sediments at concentrations high enough to cause concern. The concentrations of tNP and OP1EO detected in biota species and water samples were all of less significance than those in sediment.

The antifouling paint biocide zineb was not detected above the limit of detection in any of the dissolved, particulate or sediment samples. Values in the draft risk assessment for zineb under the EU biocides directive indicate that the levels of ETU detected on suspended particulate matter may indicate a risk to the marine environment.

The fungicide transformation product DMS was detected in two urban lakes. The levels detected represent no risk to aquatic organisms based upon the low aquatic toxicity of DMS. All samples contained levels above the drinking water quality standard or 100 ng/L, although there is no risk to humans from these particular lakes since they are not used as drinking water sources.

The SGARs brodifacoum, bromadiolone, difenacoum and flocoumafen were detected in golden eagle and eagle owl livers. Thirty percent (7/24) of the golden eagle and eagle owl livers contained total SGAR residue levels above a toxicity threshold of 100 ng/g while it is not possible to rule out poisoning below this threshold and the further estimation of the potential mortality impact on the sampled raptor populations is not possible.

Selected current use pesticides (CUPs) and alkylphenolic compounds were not detected in Arctic biota.

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10. APPENDICES

Table 22. Alkylphenolic compounds and bisphenol A in water (ng/L)

	Lat	Long	pH	DOC (mgC/L)	Salinity	4-t-OP	t-NP	4-n-OP	4-n-NP	OP1EO	NP1EO	BPA
Spjeldsjøen1	67,77640	28,97700	5.46	16.0		2	3	<0.1	<10	<0.01	<0.08	<0.05
Spjeldsjøen2	67,77640	28,97700				8,5	8,8	<0.1	<2	<0.1	<0.3	<0.05
Spjeldsjøen3	67,77640	28,97700				12	17	<0.2	<30	<0.02	<0.1	<0.05
Spjeldsjøen4	67,77640	28,97700				7,9	5,9	<01	<2	<0.04	<0.2	<0.05
Spjeldsjøen5	67,77640	28,97700				4,8	4	<0.1	<18	<0.02	<0.1	<0.05
Mjøsa1	67,53600	2,67020				30	17	<0.2	<3	0,04	<0.1	<0.05
Mjøsa2	67,45850	2,66660				71	42	<0.6	<15	0,04	<0.1	<0.05
Mjøsa3	67,47390	2,66400				26	20	<0.2	<10	0,02	<0.03	<0.05
Mjøsa4	67,45070	2,69350				40	34	0,1	<3	0,02	0,07	<0.05
Mjøsa5	67,48960	2,66830				15	26	<0.1	<4	0,02	0,16	<0.05
Puddefjorden1	60,38604	5,31094	8.01	1.3	30.2	323	303	<0.3	88	0,96	4	330
Puddefjorden2	60,38778	5,30789				15	11	<0.1	5,8	0,39	<0.3	13
Puddefjorden3	60,39044	5,30225				396	421	1,8	111	0,42	1,6	70
Puddefjorden4	60,39323	5,29318				53	82	0,9	18	0,36	<2	<3
Puddefjorden5	60,39542	5,28465				23	23	<0.2	9,9	0,37	<1	<3
Skånevikfjorden1	59,71893	5,85603	7.25	1100	27.1	1,9	18	<0.2	<4	0,45	<3	<3
Skånevikfjorden2	59,71288	5,85425				1,5	1,5	<0.1	<0.7	0,88	<2	<2
Skånevikfjorden3	59,73490	5,90027				2,6	2,6	<0.3	<3	0,66	<0.6	<0.6
Skånevikfjorden4	59,74207	5,99042				6,8	6,8	0,18	7,1	0,42	<0.7	<0.7
Skånevikfjorden5	59,73670	5,98375				15	15	0,13	9,8	0,58	<0.9	<0.9

Table 23. Alkylphenolic compounds and bisphenol A in particulates (pg/L)

	Lat	Long	4-t-OP	t-NP	4-n-OP	4-n-NP	OP1EO	NP1EO	BPA
Spjeldsjøen1	67,77640	28,97700	193,8	394,66	<1	<2	10,63	84,87	194,52
Spjeldsjøen2	67,77640	28,97700	314,3	474,5	<3	<30	15,26	115,118	103,87
Spjeldsjøen3	67,77640	28,97700	242,05	518,8	<2	<30	22,31	146,24	120,4
Spjeldsjøen4	67,77640	28,97700	274,46	881,06	<1.5	<30	16,9	108,5	60,43
Spjeldsjøen5	67,77640	28,97700	472,15	506,46	<2.5	<41	15,79	85,07	228,6
Mjøsa1	67,53600	2,67020	327,89	721,28	<2	<60	24,36	<115	138,62
Mjøsa2	67,45850	2,66660	255,48	681,45	<6	<35	15,8	<99	63,4
Mjøsa3	67,47390	2,66400	282,16	817,41	<2	<78	32,18	<258	996,32
Mjøsa4	67,45070	2,69350	256,06	528,4	<3	<48	18,03	<220	203,67
Mjøsa5	67,48960	2,66830	363,53	1309,04	<2.2	<29	16,08	<95	138,11
Puddefjorden1	60,38604	5,31094	2,69	4,42	<0.1	<1.23	0,13	0,61	0,86
Puddefjorden2	60,38778	5,30789	3,75	<4.3	<0.11	<1.34	0,15	0,64	1,58
Puddefjorden3	60,39044	5,30225	1,31	1,68	<0.03	<0.42	0,1	0,93	0,45
Puddefjorden4	60,39323	5,29318	1,58	2,37	<0.03	<0.48	0,13	0,58	0,35
Puddefjorden5	60,39542	5,28465	1,17	1,61	0,16	1,73	0,39	0,87	0,1
Skånevikfjorden1	59,71893	5,85603	2,14	<1.91	0,22	<0.34	1,57	1,6	0,06
Skånevikfjorden2	59,71288	5,85425	3,39	<2.21	0,4	<0.33	1,63	5,55	0,08
Skånevikfjorden3	59,73490	5,90027	2,73	<2.35	0,08	<0.24	1,31	6,14	0,04
Skånevikfjorden4	59,74207	5,99042	1,52	<2.1	<0.02	<0.54	1,15	5,31	0,04
Skånevikfjorden5	59,73670	5,98375	1,97	<2.3	<0.01	<0.5	0,95	5,99	0,04

Table 24. Alkylphenolic compounds and bisphenol A in sediments (ng/g)(dry weight)

	Lat	Long	Korn	TOC (ugC/mgTS)	4-t-OP	t-NP	4-n-OP	4-n-NP	OP1EO	NP1EO	BPA
Spjeldsjøen1	67,77640	28,97700	13	260	<50	248,08	0,4	<8	21,02	<30	<40
Spjeldsjøen2	67,77640	28,97700	16	256	<50	489,08	0,19	<4	23,36	<35	<16
Spjeldsjøen3	67,77640	28,97700	18	259	<50	142,84	0,29	<10	20,5	<25	3,71
Spjeldsjøen4	67,77640	28,97700	19	258	<50	160,27	0,28	<10	13,4	<30	<10
Spjeldsjøen5	67,77640	28,97700	19	265	<50	244,8	0,24	<10	16,96	39,66	<10
Mjøsa1	67,53600	2,67020	36	26.6	<25	23,54	<0.1	<1	3,76	<5	<6
Mjøsa2	67,45850	2,66660	68	10.6	<25	158,12	<0.1	<0.5	3,74	19,27	<20
Mjøsa3	67,47390	2,66400	50	86.1	<25	264,86	0,67	17,6	13,58	198,33	8,72
Mjøsa4	67,45070	2,69350	51	30.3	<25	160,58	<0.1	<0.5	5,5	<10	<13
Mjøsa5	67,48960	2,66830	55	17.1	<25	73,2	<0.1	<0.5	1,9	<3	<6
Puddefjorden1	60,38604	5,31094	48	35.3	<15	160	0,18	<4	4,8	<40	<8
Puddefjorden2	60,38778	5,30789	47	46.5	42	192	0,46	<6	6,9	88	17
Puddefjorden3	60,39044	5,30225	41	54.3	45	154	0,78	19	9,6	57	20
Puddefjorden4	60,39323	5,29318	34	68.3	17	137	0,45	5,9	4,9	<20	8,2
Puddefjorden5	60,39542	5,28465	17	37.5	19	115	2,1	79	3,6	<25	3,8
Skånevikfjorden1	59,71893	5,85603	24	4,3	<15	63	<0.1	<0.5	1,6	<2	<5
Skånevikfjorden2	59,71288	5,85425	93	18,5	<15	<10	<0.1	<0.5	1,7	<1.5	<5
Skånevikfjorden3	59,73490	5,90027	18	5,7	<15	25	<0.1	<1	1,9	<3	<5
Skånevikfjorden4	59,74207	5,99042	20	8,9	<15	18	<0.1	<0.5	1,3	<2	<5
Skånevikfjorden5	59,73670	5,98375	76	25,7	<15	65	<0.1	<1.5	2,2	<3	<5

Table 25. Alkylphenolic compounds and bisphenol A in trout filet (ng/g) (wet weight)

	Lipid (%)	W% C/N	4-t-OP	t-NP	4-n-OP	4-n-NP	OP1EO	NP1EO	BPA
Spjeldsjøen1	3,47	3,83	<5	<4	<0,5	<1	<0,5	<3	<1,5
Spjeldsjøen2	2,33	3,52	<5	<4	<0,5	<1	<0,5	<3	<1,5
Spjeldsjøen3	2,67	3,52	<5	<4	<0,5	<1	<0,5	<3	<1,5
Spjeldsjøen4	1,92	3,38	<5	<4	<0,5	<2	<0,5	<3	<1,5
Spjeldsjøen5	2,52	3,58	<5	<4	<0,5	<1	<0,5	<3	<1,5
Spjeldsjøen6	2,04	3,38	<5	<4	<0,5	<2	<0,5	<3	<1,5
Spjeldsjøen7	2,79	3,65	<5	<4	<0,5	<1	<0,5	<3	<1,5
Spjeldsjøen8	2,73	3,58	<5	<4	<0,5	<1	<0,5	<3	<1,5
Spjeldsjøen9	2,14	3,43	<5	<4	<0,5	<1	<0,5	<3	<1,5
Spjeldsjøen10	2,47	3,57	<5	<4	<0,5	<2	<0,5	<3	<1,5
Spjeldsjøen11	2,1	3,41	<5	<4	<0,5	<1	<0,5	<3	<1,5
Spjeldsjøen12	1,49	3,27	<5	<4	<0,5	<3	<0,5	<3	<1,5
Spjeldsjøen13	1,99	3,39	<5	<4	<0,5	<1	<0,5	<3	<1,5
Spjeldsjøen14	2,85	3,61	<5	<4	<0,5	<5	<0,5	<3	<1,5
Spjeldsjøen15	2,01	3,43	<5	<4	<0,5	<2	<0,5	<3	<1,5
Mjøsa1	7,45	4,66	<5	6,13	<0,5	<1	<0,5	<3	<1,5
Mjøsa2	7,77	4,69	<6,5	9,79	<0,5	<3	<0,5	<3	<1,5
Mjøsa3	4,64	3,93	<6,5	<5	<0,5	<3	<0,5	<3	<1,5
Mjøsa4	4,27	3,87	<5	<4	<0,5	<1	<0,5	<3	<1,5
Mjøsa5	5,59	4,25	<5	5,73	<0,5	<2	<0,5	<3	<1,5
Mjøsa6	6,02	4,30	<5	<5	<0,5	<2	<0,5	<3	<1,5
Mjøsa7	2,94	3,54	<5	<4	<0,5	<2	<0,5	<3	<1,5
Mjøsa8	2,14	3,42	<5	<4	<0,5	<1	<0,5	<3	<1,5
Mjøsa9	4,03	3,90	<5	<4	<0,5	<2	<0,5	<3	<1,5
Mjøsa10	3,55	3,79	<5	<4	<0,5	<2	<0,5	<3	<1,5
Mjøsa11	3,08	3,61	<5	<4	<0,5	<2	<0,5	<3	<1,5
Mjøsa12	1,07	3,13	<5	<4	<0,5	<2	<0,5	<3	<1,5
Mjøsa13	2,7	3,59	<5	<4	<0,5	<2	<0,5	<3	<1,5
Mjøsa14	2,44	3,45	<5	<4	<0,5	<2	<0,5	<3	<1,5
Mjøsa15	2,01	3,39	<5	<4	<0,5	<2	<0,5	<3	<1,5
Mjøsa16	2,71	3,56	<5	<4	<0,5	<2	<0,5	<3	<1,5

Table 26. Alkylphenolic compounds and bisphenol A in cod liver (ng/g) (wet weight)

	Lipid (%)	W% C/N	4-t-OP	t-NP	4-n-OP	4-n-NP	OP1EO	NP1EO	BPA
Byfjord1	38,24	3,00	<10	<11	<1	3,09	<1	5,4	6,3
Byfjord2	39,22	2,99	<10	<30	<1	8,8	<1	8,5	4,5
Byfjord3	60,02	2,99	<10	76,9	<1	3,8	<1	44,47	46,3
Byfjord4	49,69	3,01	<10	85,7	<1	2,2	2,75	21,3	15,6
Byfjord5	63,39	3,00	10,6	36,8	<1	114	<1	<7	26,8
Byfjord6	62,22	3,06	18,73	135,2	<1	2,5	3,35	56,75	20,1
Byfjord7	62,08	3,02	29,97	302,9	<1	1,5	7,38	54,76	44,8
Byfjord8	55,01	3,01	<10	<10	<1	40,6	<1	16,43	8,5
Byfjord9	74,76	3,01	41,63	383,43	<1	5,4	10,1	120,92	<500
Byfjord10	62,47	3,04	<10	196,48	<1	5,3	3,24	92,39	<1000
Byfjord11	65,1	2,97	<10	56,86	<1	90,4	<2	30	4,5
Byfjord12	15,7	2,98	<10	<40	<1	14,47	<1	9	<4
Byfjord13	77,58	3,03	<10	74	2,34	29,37	<1	17,42	9,9
Byfjord14	51,54	3,04	28,14	117,5	<1	2,4	<2	18,07	<200
Byfjord15	58,09	3,02	<10	16,3	<1	2,3	<2	46,06	18,7
Skånevikfjorden1	37,86	3,01	<3	<20	<2	<5	<3	<15	<20
Skånevikfjorden2	43,88	2,99	<5	<20	<5	<5	<3	<15	<15
Skånevikfjorden3	45,74	3,00	<5	<25	<5	<10	<10	<10	<15
Skånevikfjorden4	12,92	3,00	<3	<20	<2	<5	<3	<10	<10
Skånevikfjorden5	56,20	2,99	<2	<25	<3	<20	<3	<15	<20
Skånevikfjorden6	38,78	2,95	<5	<25	<10	<20	<3	<15	<20
Skånevikfjorden8	67,30	3,00	<5	<20	<5	<5	<4	<25	<25

Table 27. Alkylphenolic compounds and bisphenol A in crab (ng/g) (wet weight)

	Lipid (%)	W% C/N	4-t-OP	t-NP	4-n-OP	4-n-NP	OP1EO	NP1EO	BPA
Puddefjorden 1	15,70	9,95	<10	7,9	<0,5	6,6	<1	<15	<1
Puddefjorden 2	8,70	5,74	<10	<5	<0,5	6	<1	<10	<2
Puddefjorden 3	19,29	11,62	<10	9,4	<0,5	5,5	<1	<10	<1
Puddefjorden 4	14,86	6,33	<10	7,9	<0,5	5,3	<1	<10	<3
Skånevikfjorden1	11,20	11,33	<10	<5	<0,5	4,8	<1	<10	<1
Skånevikfjorden2	9,67	7,11	<10	<5	<0,5	2,5	<1	<40	<1
Skånevikfjorden3	10,74	4,76	<10	<5	<0,5	5,3	<1	<30	<1
Skånevikfjorden4	6,86	6,11	<10	<5	<0,5	<2	<1	<15	<2

Table 28. Alkylphenolic compounds and bisphenol A in blue mussel (ng/g) (wet weight)

	Lat	Long	Lipid (%)	W% C/N	4-t-OP	t-NP	4-n-OP	4-n-NP	OP1EO	NP1EO	BPA
Puddefjorden 1	60,38142	5,34978	2,39	4,89	<2	<7	<0,5	<1	<1	<6	<1
Puddefjorden 2	60,38078	5,34413	1,96	4,60	<2	<7	<0,5	<1	<1	<3	<1
Puddefjorden 3	60,38706	5,30123	2,03	4,64	<2	<7	<0,5	<1	<1	<4	<1
Puddefjorden 4	60,39876	5,30241	2,51	4,64	<2	<7	<0,5	<1	<1	<3	<1
Puddefjorden 5	60,39230	5,28310	2,37	4,94	<3	<12	<2	<30	<12	<12	<1
Skånevikfjorden1	59,73143	5,87493	1,49	4,21	<4	<15	<0,5	<5	<1	<3	<1
Skånevikfjorden2	59,73437	5,93528	1,65	4,00	<4	<20	<0,5	<3	<1	<6	<1
Skånevikfjorden3	59,76593	5,93168	0,94	4,67	<2	<40	<0,5	<3	<1	<6	<1
Skånevikfjorden4	59,74810	5,96500	1,79	4,54	<2	<10	<0,5	<5	<1	<3	<1
Skånevikfjorden5	59,73833	5,99040	2,14	5,37	<4	<30	<0,5	<2	<5	<12	<1

Table 29. ETU and zineb concentrations in water (ng/L)

Water	Lat	Long	TOC (mgC/L)	Cu (mg/L)	pH	Salinity	ETU (ng/L)	Zineb (ng/L)
Bestumkilen 1	59,55133	10,30328	3.8	0.0094		15.2	<5	<5
Bestumkilen 2	59,55011	10,40063					<5	<5
Bestumkilen 3	59,55171	10,39573					<5	<5
Bestumkilen 4	59,54849	10,39343					<5	<5
Bestumkilen 5	60,26289	5,25371					<5	<5
Kviturspollen 1	60,26264	5,25259	1.6	0.002	1.6	28.9	<5	<5
Kviturspollen 2	60,26276	5,25082	1.5	<0.002	1.5	29.4	<5	<5
Kviturspollen 3	60,26329	5,24830	1.4	<0.002	1.4	29.0	<5	<5
Kviturspollen 4	60,26310	5,24164	1.5	<0.002	1.5	28.6	<5	<5
Kviturspollen 5	59,71893	5,85603	1.5	<0.002	1.5	29.2	<5	<5
Skånevikfjorden 1	59,71288	5,85425			7.5	27.1	<5	<5
Skånevikfjorden 2	59,7349	5,90026					<5	<5
Skånevikfjorden 3	59,74206	5,99041					<5	<5
Skånevikfjorden 4	59,73670	5,98375					<5	<5
Skånevikfjorden 5	59,73670	5,98375					<5	<5

Table 30. ETU and zineb concentrations in particulates (ng/L)

Particulate	Lat	Long	ETU (ng/L)	Zineb (ng/L)
Bestumkilen 1	59,55133	10,30328	1.9	<20
Bestumkilen 2	59,55011	10,40063	2.2	<20
Bestumkilen 3	59,55171	10,39573	2.1	<20
Bestumkilen 4	59,54849	10,39343	1.3	<20
Bestumkilen 5	60,26289	5,25371	2.0	<20
Kviturspollen 1	60,26289	5,25371	9.2	<20
Kviturspollen 2	60,26264	5,25259	5.6	<20
Kviturspollen 3	60,26276	5,25082	15.5	<20
Kviturspollen 4	60,26329	5,24830	2.7	<20
Kviturspollen 5	60,26310	5,24164	3.3	<20
Skånevikfjorden 1	59,71893	5,85603	<1	<20
Skånevikfjorden 2	59,71288	5,85425	<1	<20
Skånevikfjorden 3	59,73490	5,90026	<1	<20
Skånevikfjorden 4	59,74206	5,99041	<1	<20
Skånevikfjorden 5	59,73670	5,98375	<1	<20

Table 31. ETU and zineb concentrations in sediment (ng/g) (dry weight)

Sediment	Lat	Long	PSA <63um %tv	TOC (ugC/mgTS)	Cu (ng/g)	ETU (ng/g)	Zineb (ng/g)
Bestumkilen 1	59,55171	10,40403	77	18.6	130	<10	<20
Bestumkilen 2	59,55133	10,30328	79	42.9	186	<10	<20
Bestumkilen 3	59,55011	10,40063	65	42.9	158	<10	<20
Bestumkilen 4	59,55171	10,39573	68	44.5	237	<10	<20
Bestumkilen 5	59,54849	10,39343	68	35.2	138	<10	<20
Kviturspollen 1	60,26289	5,25371	51	143	109	<10	<20
Kviturspollen 2	60,26264	5,25259	67	92.7	79.0	<10	<20
Kviturspollen 3	60,26276	5,25082	61	144	202	<10	<20
Kviturspollen 4	60,26329	5,2483	17	61.7	31.5	<10	<20
Kviturspollen 5	60,2631	5,24164	14	37.9	37.4	<10	<20
Skånevikfjorden 1	59,71893	5,85603	24	4,3	4,7	<10	<20
Skånevikfjorden 2	59,71288	5,85425	93	18,5	15	<10	<20
Skånevikfjorden 3	59,7349	5,90026	18	5,7	4,2	<10	<20
Skånevikfjorden 4	59,74206	5,99041	20	8,9	7,08	<10	<20
Skånevikfjorden 5	59,7367	5,98375	76	25,7	23,7	<10	<20

Table 32. Concentrations of N-N-dimethylsulfamide in water (ng/L)

Water	Sample type	N,N-dimethylsulfamid
Østensjø, Østensjøveien	96 hr composite	773,9
Østensjø, Østmarkveien	96 hr composie	651,8
Østensjø, Haakon Tvertersvei	spot sample	456,1
Holmendammen, Binneveien	spot sample	241,0
Holmendammen, Ankerveien	spot sample	540,3
Holmendammen, Gaupefaret	96 hr composite	104,4
Skodøltjenn, Eggedal 1	spot sample	<5
Skodøltjenn, Eggedal 2	spot sample	5,0
Skodøltjenn, Eggedal 3	spot sample	<5
Skodøltjenn, Eggedal 4	spot sample	<5
Skodøltjenn, Eggedal 5	spot sample	<5

Table 33. Concentrations of N-N-dimethylsulfamide in sediment (ng/g)(dry weight)

Sediment	N,N-dimethylsulfamid
Skodøltjenn, Eggedal 1	<5
Skodøltjenn, Eggedal 2	<5
Skodøltjenn, Eggedal 3	<5
Skodøltjenn, Eggedal 4	<5
Skodøltjenn, Eggedal 5	<5
Holmendammen Tråkka	<5
Holmendammen Ankerveien	<5
Holmendammen Stasjonsveien	<5
Østensjø Østensjøterasse	<5
Østensjø Vilbergveien	<5

Table 34. Concentrations of second generation anticoagulant rodenticides in liver (ng/g) (wet weight)

				Anticoagulant Rodenticde (ng/g liver)				
Species		**Location**	**Year**	**Flocoumafen**	**Difethialone**	**Difenacoum**	**Bromadiolone**	**Brodifacoum**
Osprey	*Pandion haliaetus*	Øvre Rendal	2010	<2	<5	<2	<5	<5
		Sør Trøndelag	2009	<2	<5	<2	<5	<5
		Valldal	2010	<2	<5	<2	<5	<5
Golden Eagle	*Aquila chrysaetos*	Løten	2010	<2	<5	<2	<5	11
		Flekkefjord	2009	<2	<5	<2	<5	<5
		Vikeså	2009	<2	<5	<2	50	<5
		Vinje	2010	117	<5	<2	13	<5
		Vik i Sogn	2010	<2	<5	<2	<5	<5
		Hol i Tjeldsund	2010	<2	<5	<2	<5	<5
		Hansnes	2011	<2	<5	<2	<5	<5
		Åndalsnes	2009	15	<5	<2	31	<5
		Vikeså	2010	<2	<5	<2	154	57
		Lyngdal	2011	<2	<5	<2	20	<5
		Oppdal	2011	<2	<5	<2	<5	110
		Engerdal	2009	<2	<5	<2	<5	<5
		Balestrand	2009	<2	<5	<2	<5	29
		Rena	2009	<2	<5	<2	22	99
		Tolga	2011	<2	<5	<2	43	21
		Kyrksæterøra	2009	<2	<5	<2	<5	16
Eagle Owl	*Bubo bubo*	Hitra	2011	<2	<5	<2	<5	<5
		Hitra	2011	<2	<5	39	<5	<5
		Kopervik	2011	13	<5	<2	<5	133
		Hitra	2010	<2	<5	<2	<5	<5
		Mandal	2011	<2	<5	<2	<5	158
		Halden	2010	<2	<5	<2	<5	95
		Hardbakke	2011	<2	<5	<2	<5	<5
		Fitjar	2009	<2	<5	181	<5	74
Gyrfalcon	*Falco rusticolus*	Berlevåg	2011	<2	<5	<2	<5	<5
Peregrine Falcon	*Falco peregrinus*	Roa	2010	<2	<5	<2	<5	<5
		Gaupne	2009	<2	<5	<2	<5	<5

Table 35. Alkylphenolic compounds in Polar cod (ng/g)

	% lipid	W% C/N	4-t-OP	t-NP	4-n-OP	4-n-NP	OP1EO	NP1EO
1	6,34	4,95	<1	<10	<1	<0.5	<0.5	<10
2	7,68	4,74	<1.9	<10	<1	<0.5	<0.5	<10
3	8,34	5,59	<1	<10	<1	<1.5	<0.5	<10
4	5,11	4,66	<1.3	<10	<1	<1	<0.5	<10
5	5,35	4,66	<1	<10	<3	<1.5	<0.5	<10
6	4,79	4,87	<2	<130	<1	<45	<0.5	<10
7	6,35	5,52	<2.6	<10	<1	<1	<0.5	<10
8	7,05	5,46	<2.1	<10	<1	<1	<0.5	<10
9	5,52	4,63	<1.3	<10	<1	<1.5	<0.5	<10
10	6,49	5,24	<1	<10	<1	<1	<0.5	<10

Table 36. Alkylphenolic compounds in capelin (ng/g)

	% lipd	W% C/N	4-t-OP	t-NP	4-n-OP	4-n-NP	OP1EO	NP1EO
1	7,46	5,42	<2	<10	<2	<2	<2	<10
2	4,72	4,52	<2	<10	<2	<2	<2	<10
3	6,15	5,26	<2	<10	<2	<2	<2	<10
4	8,13	5,71	<2	<10	<2	<2	<2	<10
5	6,40	5,37	<2	<10	<2	<2	<2	<10
6	7,49	4,71	<2	<10	<2	<2	<2	<10
7	5,59	4,84	<2	<10	<2	<2	<2	<10
8	9,97	5,47	<2	<10	<2	<2	<2	<10
9	8,27	6,39	<2	<10	<2	<2	<2	<10
10	14,40	7,28	<2	<10	<2	<2	<2	<10

Table 37. Alkylphenolic compounds in seal blubber (ng/g)

	Lat	Long	% lipid	W% C/N	4-t-OP	t-NP	4-n-OP	4-n-NP	OP1EO	NP1EO
BS3-2011 bearded seal	54,18000	87,32899	94,94	71,92	<10	<10	<15	<20	<10	<15
BS1-2011 bearded seal	54,18000	87,32899	109,30	73,78	<10	<10	<15	<20	<10	<15
RS1-2011 ringed seal	54,18000	87,32899	92,25	74,81	<10	<10	<15	<20	<10	<15
RS2 ringed seal	54,18000	87,32899	89,80	64,31	<10	<10	<15	<20	<10	<15
BS2 bearded seal	54,18000	87,32899	81,73	35,67	<10	<10	<15	<20	<10	<15

Table 38. Alkylphenolic compounds in Arctic sea bird egg (ng/g)

	% lipid	W% C/N	4-t-OP	t-NP	4-n-OP	4-n-NP	OP1EO	NP1EO
Common guilemot								
Egg 1 2010	14,04	8,96	<5	<10	<5	<10	<5	<10
Egg 1 2011	12,90	7,25	<5	<10	<5	<10	<5	<10
Egg 2 2010	11,67	8,24	<5	<10	<5	<10	<5	<10
Egg 2 2011	12,12	8,39	<5	<10	<5	<10	<5	<10
Egg 3 2010	13,33	8,29	<5	<10	<5	<10	<5	<10
Egg 3 2011	11,67	7,49	<5	<10	<5	<10	<5	<10
Egg 4 2010	11,29	8	<5	<10	<5	<10	<5	<10
Egg 4 2011	14,52	7,59	<5	<10	<5	<10	<5	<10
Egg 5 2010	11,48	7,52	<5	<10	<5	<10	<5	<10
Egg 5 2011	11,86	8,02	<5	<10	<5	<10	<5	<10
Eider duck								
Egg 2a	16,95	9,03	<5	<10	<5	<10	<5	<10
Egg 3a	16,39	9,4	<5	<10	<5	<10	<5	<10
Egg 5a	15,25	9,69	<5	<10	<5	<10	<5	<10
Egg 18a	16,95	9,14	<5	<10	<5	<10	<5	<10
Egg 26a	18,46	8,59	<5	<10	<5	<10	<5	<10
Egg 56	15,87	8,92	<5	<10	<5	<10	<5	<10
Egg 72a	16,13	9,33	<5	<10	<5	<10	<5	<10
Egg 82a	16,92	9,02	<5	<10	<5	<10	<5	<10
egg 90	17,54	9,53	<5	<10	<5	<10	<5	<10
Egg 128a	19,35	9,59	<5	<10	<5	<10	<5	<10

Table 39. Alkylphenolic compounds in arctic seabird whole blood and plasma (ng/ml)

	Lat	Long	W% C/N	4-t-OP	t-NP	4-n-OP	4-n-NP	OP1EO	NP1EO
Glaucous gull - serum									
1-2011	78,55670	11,57328	5,28	<20	<30	<10	<20	<10	<30
2-2011	78,55889	12,17847	5,41	<20	<30	<10	<20	<10	<30
3-2011	78,59153	11,57897	5,28	<20	<30	<10	<20	<10	<30
4-2011	78,59721	12,02255	5,33	<20	<30	<10	<20	<10	<30
5-2011	79,00184	12,04655	5,16	<20	<30	<10	<20	<10	<30
6-2011	78,55069	12,01082	5,18	<20	<30	<10	<20	<10	<30
7-2011	78,56662	12,15259	5,03	<20	<30	<10	<20	<10	<30
8-2011	78,55069	12,01082	4,99	<20	<30	<10	<20	<10	<30
9-2011	78,55889	12,17847	4,97	<20	<30	<10	<20	<10	<30
10-2011	78,56014	12,24820	4,97	<20	<30	<10	<20	<10	<30
Kittiwake - whole blood									
K56-07			3,31	<20	<30	<10	<20	<10	<30
K57-07			3,37	<20	<30	<10	<20	<10	<30
K71-07			3,30	<20	<30	<10	<20	<10	<30
K75-07			3,29	<20	<30	<10	<20	<10	<30
K97-07			3,28	<20	<30	<10	<20	<10	<30

Table 40. Current use pesticides in polar cod (ng/g)

	% fat	W% C/N	Pentachlorophenol	Trifluralin	Chlorpyrifos	Dacthal	Methoxychlor
1	6,34	4,95	<2	<0,1	<1	<0,3	<3
2	7,68	4,74	<2	<0,1	<1	<0,3	<3
3	8,34	5,59	<2	<0,1	<1	<0,4	<3
4	5,11	4,66	<2	<0,1	<1	<0,3	<3
5	5,35	4,66	<2	<0,1	<1	<0,4	<3
6	4,79	4,87	<2	<0,1	<2	<0,4	<3
7	6,35	5,52	<2	<0,1	<1	<0,3	<3
8	7,05	5,46	<2	<0,1	<1	<0,3	<3
9	5,52	4,63	<2	<0,1	<1	<0,3	<3
10	6,49	5,24	<2	<0,1	<1	<0,3	<4

Table 41. Current use pesticides in capelin (ng/g)

	% fat	W% C/N	Pentachlorophenol	Trifluralin	Chlorpyrifos	Dacthal	Methoxychlor
1	7,46	5,42	<5	<0,5	<0,1	<0,1	<24
2	4,72	4,52	<5	<0,3	<0,1	<0,1	<1
3	6,15	5,26	<5	<0,3	<0,1	<0,1	<2
4	8,13	5,71	<5	<0,3	<0,1	<0,1	<2
5	6,40	5,37	<5	<0,3	<0,1	<0,1	<2
6	7,49	4,71	<5	<0,3	<0,1	<0,1	<3
7	5,59	4,84	<5	<0,3	<0,1	<0,1	<2
8	9,97	5,47	<5	<0,3	<0,1	<0,1	<3
9	8,27	6,39	<5	<0,5	<0,2	<0,1	<2
10	14,40	7,28	<5	<0,3	<0,2	<0,2	<2

Table 42. Current use pesticides in seal blubber (ng/g)

	Lat	Long	% lipid	W% C/N	Pentachlorophenol	Trifluralin	Chlorpyrifos	Dacthal	Methoxychlor
BS3-2011 bearded seal	54,18000	87,32899	94,94	71,92	<5	<1	<1	<0.3	<2
BS1-2011 bearded seal	54,18000	87,32899	109,30	73,78	<5	<1	<1	<0.5	<5
RS1-2011 ringed seal	54,18000	87,32899	92,25	74,81	<5	<1	<1	<0.3	<5
RS2 ringed seal	54,18000	87,32899	89,80	64,31	<5	<1	**1,4**	<0.4	<4
BS2 bearded seal	54,18000	87,32899	81,73	35,67	<5	<1	<1	<0.4	<7

Table 43. Current use pesticides in arctic sea bird egg (ng/g)

	% lipid	W% C/N	Pentachlorophenol	Trifluralin	Chlorpyrifos	Dacthal	Methoxychlor
Common guilemot							
Egg 1 2010	14,04	8,96	<5	<0,3	<1	<1	<5
Egg 1 2011	12,90	7,25	<5	<0,3	<1	<1	<5
Egg 2 2010	11,67	8,24	<5	<0,5	<2	<2	<5
Egg 2 2011	12,12	8,39	<5	<0,3	<1	<1	<5
Egg 3 2010	13,33	8,29	<5	<0,3	<1	<1	<5
Egg 3 2011	11,67	7,49	<5	<0,3	<1	<1	<5
Egg 4 2010	11,29	8	<5	<0,3	<1	<1	<5
Egg 4 2011	14,52	7,59	<5	<0,4	<2	<1	<5
Egg 5 2010	11,48	7,52	<5	<0,3	<2	<1	<5
Egg 5 2011	11,86	8,02	<5	<0,5	<2	<2	<5
Eider duck							
Egg 2a	16,95	9,03	<5	<0,5	<2	<2	<40
Egg 3a	16,39	9,4	<5	<0,3	<2	<1	<15
Egg 5a	15,25	9,69	<5	<0,3	<1	<1	<15
Egg 18a	16,95	9,14	<5	<0,4	<2	<1	<10
Egg 26a	18,46	8,59	<5	<0,3	<1	<1	<6
Egg 56	15,87	8,92	<5	<0,5	<2	<2	<5
Egg 72a	16,13	9,33	<5	<0,3	<1	<1	<5
Egg 82a	16,92	9,02	<5	<0,3	<2	<1	<5
Egg 90	17,54	9,53	<5	<0,4	<2	<1	<10
Egg 128a	19,35	9,59	<5	<0,3	<1	<1	<5

Table 44. Current use pesticides in arctic seabird whole blood and plasma (ng/ml)

	Lat	Long	W% C/N	Pentachlorophenol	Trifluralin	Chlorpyrifos	Dacthal	Methoxychlor
Glaucous gull - serum								
1-2011	78,55670	11,57328	5,28	<5	<2	<5	<2	<10
2-2011	78,55889	12,17847	5,41	<5	<2	<5	<2	<10
3-2011	78,59153	11,57897	5,28	<5	<2	<5	<2	<10
4-2011	78,59721	12,02255	5,33	<5	<2	<5	<2	<10
5-2011	79,00184	12,04655	5,16	<5	<2	<5	<2	<10
6-2011	78,55069	12,01082	5,18	<5	<2	<5	<2	<10
7-2011	78,56662	12,15259	5,03	<5	<2	<5	<2	<10
8-2011	78,55069	12,01082	4,99	<5	<2	<5	<2	<10
9-2011	78,55889	12,17847	4,97	<5	<2	<5	<2	<10
10-2011	78,56014	12,24820	4,97	<5	<2	<5	<2	<10
Kittiwake - whole blood								
K56-07			3,31	<5	<2	<5	<2	<10
K57-07			3,37	<5	<2	<5	<2	<10
K71-07			3,30	<5	<2	<5	<2	<10
K75-07			3,29	<5	<2	<5	<2	<10
K97-07			3,28	<5	<2	<5	<2	<10

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Tittel- Title

Kartlegging av utvalgte alkylphenolic forbindelser, biocider, rottegift og nåværende bruk plantevernmidler.

Screening of selected alkylphenolic compounds, biocides, rodenticides and current use pesticides

Sammendrag

Denne screening prosjekt undersøkt forekomsten av nonylfenol og nonylfenol monoethoksylat, oktylfenol og oktylfenol monnoethoksylat, og bisfenol A i marin og ferskvanns akvatiske miljøer, bunnstoffet Zineb og dens nedbrytnings produkt ethylenethiourea i småbåthavner, det soppmiddel transformasjon produktet N,N-dimethylsulfamid, valgt annensgenerasjons antikoagulant rottegift i leveren hos utvalgte rovfugl, og nonylfenol og nonylfenol monoethoksylat, oktylfenol og oktylfenol monnoethoksylat og valgte nåværende bruk plantevernmidler i Arktis biota.

Summary

This screening survey investigated the occurrence of nonylphenol and nonylphenol monoethoxylate, octylphenol and octylphenol monnoethoxylate, and bisphenol A in the marine and freshwater aquatic environments, the antifouling biocide zineb and its transformation product ethylenethiourea in marinas, the fungicide transformation product *N,N*-dimethylsulfamide, selected second generation anticoagulant rodenticides in the livers of selected raptor species and nonylphenol and nonylphenol monoethoxylate, octylphenol and octylphenol monnoethoxylate and selected current use pesticides in Arctic biota.

4 emneord Alkyfenoler, Arktisk, Rottemidler, Biocider	4 subject words Alkylphenols, Arctic, Rodenticides, Biocides.

Statlig program for forurensningsovervåking

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Om Statlig program for forurensningsovervåking

Statlig program for forurensningsovervåking omfatter overvåking av forurensningsforholdene i luft og nedbør, skog, vassdrag, fjorder og havområder. Overvåkingsprogrammet dekker langsiktige undersøkelser av:

- overgjødsling
- forsuring (sur nedbør)
- ozon (ved bakken og i stratosfæren)
- klimagasser
- miljøgifter

Overvåkingsprogrammet skal gi informasjon om tilstanden og utviklingen av forurensningssituasjonen, og påvise eventuell uheldig utvikling på et tidlig tidspunkt. Programmet skal dekke myndighetenes informasjonsbehov om forurensningsforholdene, registrere virkningen av iverksatte tiltak for å redusere forurensningen, og danne grunnlag for vurdering av nye tiltak.
Klima- og forurensningsdirektoratet er ansvarlig for gjennomføringen av overvåkingsprogrammet.

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TA-2899/2012