Organophosphorous flame retardants in Arctic biota

Statlig program for forurensningsovervåking
Rapportnr. 1092/2011

Utført av
Organophosphorous flame retardants in Arctic biota.
Fosfororganiske flammehemmere i Arktis biota

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1. Preface

Organophosphorous flame retardants (PFRs) are frequently utilized as flame retardants, plastic softeners and anti-foaming agents. They are further used in hydraulic fluids, paints, lacquers, glues and floor finisher waxes. The broad use of PFRs may result in diffusive spread to the environment. Some of the PFRs might be stable and thereby resist decomposition when released to the environment. PFRs contain phosphor which is an important component in the proteins of every living organism. PFRs might therefore be taken up by living organism and disturb the organism’s life functions.

The information on PFRs in biota in Norway and the Norwegian Arctic area is scarce. To increase the knowledge of PFRs in the Norwegian Arctic a total of 14 PFRs were analyzed in eight animal species from Svalbard.

The Norwegian Polar Institute (NPI) and Institute for Environmental Studies (IVM) of VU University Amsterdam performed this study on behalf of the Climate and Pollution Agency (Klif).

We would like to thank all who have participated in projects that delivered samples for this screening. We would further thank Jon Fuglestad, the project manager at Klif, for his contribution on the project.

Tromsø, April 2011.

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Summary

Organophosphorous flame retardants (PFR) are currently used for various industrial purposes. The knowledge of their distribution in the environment is scarce. Screening PFRs in the Arctic biota may give valuable information about the behaviour of these compounds in the environment. A detection of these contaminants in Arctic biota indicates that PFRs undergo long-range transport since there are no known local sources in the Arctic. Further, their distribution in the food web could give an indication whether PFRs accumulate in the food web or not. As part of the work related to international conventions, harmful chemicals should be removed from the market if they fulfil the following criteria; persistency, volatility (potential for long-range transport), biomagnification (accumulate in the food web) and toxicity (show toxic effects). Therefore, contaminants that are detected in the Arctic environment, accumulate in the food web and may cause toxic effects are of high importance to be phased out and substituted by less harmful compounds.

The main goal of the present study was to screen for PFRs in the Arctic biota. A total of eight animal species from Svalbard were included in the screening: one fish species; capelin (Mallotus villosus), three seabird species; Brünnich’s guillemot (Uria lomvia), black-legged kittiwake (Rissa tridactyla) and glaucous gull (Larus hyperboreus) and four mammalian species; ringed seal (Phoca hispida), harbour seal (Phoca vitulina), arctic fox (Vulpes lagopus) and polar bear (Ursus maritimus). The samples from these species were collected in collaboration with other research projects at the Norwegian Polar Institute (NPI).

A total of 14 different phosphorous compounds were analyzed in 94 samples from the species listed above. Four of these compounds (TBP, ToCrP, DBPhP and V6) were not found in any of the samples. Except for tris(2-butoxyethyl)phosphate (TBEP) that was found at elevated levels in 50 % of the arctic fox samples, all compounds were found at low levels. The concentrations were often close to the detection level. Seven of the 14 compounds were present in ≥30 % of the samples within at least one species. In capelin, five of the compounds were detected in 60 % of the samples and three compounds (TCPP, TPhP and EHDPP) were present in all samples.

These results indicate that seven (TCEP, TCPP, TBEP, TEHP, TPhP, EHDPP and TCrP) of the 14 analyzed phosphorous compounds undergo long-range transport to the Arctic. None of the compounds seems to biomagnify from fish to seabirds or mammals in the Arctic marine food chain. The TBEP was found in 10 % of the capelins and 50 % of arctic fox. This could indicate that the TBEP is more related to the terrestrial food chain, but a conclusion cannot be drawn as the arctic fox was the only terrestrial animal examined in this study.

It is recommended that PFRs will be further investigated in (arctic) terrestrial ecosystem.
Norsk sammendrag

Hovedmålet med dette studiet var å undersøke om fosfororganiske flammehemmere (PFR) var å finne i arktiske dyr. PFR har kun en gang tidligere vært analysert i prøver fra Arktis, og da i fisk og to arter sjøfugl. Kunnskapen om PFR i norsk og arktisk natur er svært begrenset. Det ble analysert prøver fra åtte ulike arter av fisk, fugl og pattedyr fra Svalbard. Bakgrunnen for å gjøre slike undersøkelser er todelt. Hvis disse stoffene blir funnet i Arktis er det et bevis på at de transporteres fra områder hvor de har blitt brukt. Det er ingen kjente punktutslipp på Svalbard. Videre kan distribusjon av stoffene i næringskjeden gi indikasjoner på hvorvidt de oppkonsentrerer (biomagnifiserer) i næringskjeden. Kriteriene for å regulere et stoff gjennom internasjonale konvensjoner er om stoffet brytes sakte ned (persistent), om det er flyktig (potensial for langtransport), om det oppkonsentrerer (biomagnifiserer) i næringskjeden og om det er giftig. Det er derfor slik, at stoffer som gjenfinnes i arktiske dyr, spesielt om dyret oppholder seg hele året i arktiske områder, er et bevis på at stoffet kan langtransporteres. Hvis stoffet i tillegg er giftig og akkumulerer i næringskjeden, så vil dette stoffet havne langt opp på listen over kjemikalier som trengs å reguleres.

Denne rapporten har analysert prøver fra en fiskeart; lodde (Mallotus villosus), tre sjøfuglarter; polarlomvi (Uria lomvia), krykkje (Rissa tridactyla) og polarmåke (Larus hyperboreus) og fire arter pattedyr; ringsel (Phoca hispida), steinkobbe (Phoca vitulina), fjellrev (Vulpes lagopus) og isbjørn (Ursus maritimus). Alle prøvene ble samlet inn fra pågående forsknings- og overvåkningsprosjekt ved Norsk Polarinstitutt (NPI) for å optimalisere resursbruk.

Det ble analysert 14 fosfororganiske flammehemmere i 94 prøver fra dyrene beskrevet over. Fire av 14 stoffer (TBP, ToCrP, DBPhP and V6) ble ikke funnet i noen av prøvene. Sju av 14 stoffer ble funnet i minst 30 % av prøvene i en art. I lodde ble fem av de 14 stoffene funnet i ≥ 60 % av prøvene og tre av stoffene (TCP, TPhP and EHDPP) ble funnet i alle prøvene. Med unntak av tri(2-butoxyethyl)fosfat (TBEP), som ble funnet i forhøyet konsentrasjon i halvparten av fjellrevprøvene, var konsentrasjonen av de påviste stoffene lav.

Resultatene fra denne undersøkelsen indikerer at sju (TCEP, TCP, TBP, TEHP, TPhP, EHDPP and TCrP) av de 14 undersøkte fosfororganiske flammehemmerne kan langtransporteres til arktiske områder. Ingen av de sju påviste stoffene ser ut til å oppkonsentrerer (biomagnifiserer) i næringskjeden. TBEP ble funnet i 10 % av prøvene fra lodde og 50 % av prøvene fra fjellrev. Dette kan bety at TBEP er sterkere relatert til det terrestriske miljøet, men noen sikre konklusjoner kan ikke trekkes siden fjellrev var den eneste undersøkte arten fra det terrestriske naturmiljøet.

På bakgrunn av funn fra denne undersøkelsen anbefaler vi at det gjøres undersøkelser av fosfororganiske flammehemmere i det terrestriske økosystemet.
2. Background

Persistent organic pollutants (POPs) are chemicals resistant to environmental degradation and can therefore remain intact for exceptionally long periods of time. They can further become widely distributed throughout the environment and accumulate in tissues of living organisms. POPs can bioaccumulate in the food web and are often found at higher concentrations upwards the food chain. Many of the POPs are found to be toxic to both humans and wildlife. Their levels and distribution have been reviewed in several publications and reports, as for example in (MacDonald et al., 2000; AMAP, 2004). However, new compounds having similar properties as the traditional POPs have not been studied much with respect to their environmental fate and distribution in Arctic areas. These include compounds that are being used in large amounts today, like brominated flame retardants (BFRs), perfluorinated compounds (PFCs) and organophosphorous flame retardants (PFRs) that are the focus in the present report.

The general conclusion from recent reviews is that the levels of many “legacy” POPs have declined due to past regulations and bans (AMAP, 1998; 2004; 2009). The newer POPs, like some of the BFRs and PFCs, are still increasing, while some of the first regulated BFRs, the penta- and octa-brominated diphenyl ethers (BDEs), has levelled off or started to decrease (AMAP, 2009; Helgason et al., 2009). The PFRs have not been studied in a temporal manner, but recently, their presence in sewage plants and biota close to settlements in Scandinavia (Marklund et al., 2005a; b; Green et al., 2008; Sundkvist et al., 2010) and in the Arctic (Evenset et al., 2009) has been reported.

The main goal of the present project was to study the occurrence of 14 different PFRs in biological samples from the Norwegian remote Arctic area, Svalbard.

2.1 Organophosphorous flame retardants (PFR)

PFRs are frequently utilized as flame retardants and plastic softeners. They are also used as antifoaming agents and additives in products such as lubricants, hydraulic fluids, floor polishes and glues (see details below). The broad use of PFRs may result in diffusive spread to the environment via leaching, abrasion, volatilisation and improper waste handling. Sources for PFRs could therefore be any parts of populated area including household, factories, construction sites, traffic and sewage and waste handling plants. The total use of flame retardants was 465.000 metric tonnes within the European Union (EU) in 2006, while the world wide use was 1.480.000 tonnes in 2004 (SRIConsulting, 2008). The PFRs volume of the total world use of flame retardants was 14 % (207,000 tonnes). The total use of PFRs within EU was 40,000 tonnes in 2006 (SRIConsulting, 2008). The import and use in the Scandinavian countries (Sweden, Denmark, Norway and Finland) was about 4000 tonnes per year in the period 2000-2008 (Figure 1) (SPIN2000, 2011).
The PFRs have previously been found to be ubiquitous in the environment. These substances have been found indoors and in various substances such as in snow, deposition samples, groundwater and surface waters (Marklund et al., 2005a; b; Green et al., 2008; Sundkvist et al., 2010). To our knowledge, there are few reports of PFRs in the Arctic areas. These reports indicated that five PFR-compounds were found in the Austfonna ice cap (Hermanson et al., 2005) and eight of 13 PFRs were found in fish samples from Svalbard (Evenset et al., 2009). Further were 5 of these 13 PFRs found in black-legged kittiwake (Rissa tridactyla) liver samples from Kongsfjorden in Svalbard (Evenset et al., 2009).

The acute oral toxicity (LC50rats) for the chlorinated and non-chlorinated flame retardants and plasticisers is moderate. It ranges from 1 to 6 g/kg bodyweight depending on the compound in question. For aquatic organism the 96h-LC50 values for non-chlorinated flame retardants in rainbow trout (Oncorhynchus mykiss) ranged from 0.26 mg/L to 250 mg/l (WHO, 1990; 1991a; b; 1998; 2000).

The octanol-water partition coefficient (Kow) of a substance can be used to predict its potential to accumulate in biota. A high log Kow value indicates ability to accumulate. Log Kow values for the present PFRs range from 2.83 to 5.73, indicating a difference in the bioaccumulation potential.

In the Norwegian environment PFRs have been found in indoor air, outdoor air in traffic areas, sewage sludge and freshwater and marine sediment collected close to urbanized areas (Green et al., 2008). These compounds have also been found in a cod (Gadus morhua) liver sample from the inner Oslo fjord. However, but the authors question the result from that particular sample (Green et al. 2008).
Table 1: Names, CAS-numbers, abbreviations and formulas of the included organophosphorous flame retardants (PFRs) included in this screening.

<table>
<thead>
<tr>
<th>Name</th>
<th>CAS-number</th>
<th>Abbreviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Triisobutyl phosphate</td>
<td>126-71-6</td>
<td>TiBP</td>
</tr>
<tr>
<td>Tributyl phosphate</td>
<td>126-73-8</td>
<td>TBP</td>
</tr>
<tr>
<td>Tris(2-chloroethyl)phosphate</td>
<td>115-96-8</td>
<td>TCEP</td>
</tr>
<tr>
<td>Tris(2-chloroisopropyl)phosphate</td>
<td>13674-84-5</td>
<td>TCPP</td>
</tr>
<tr>
<td>Tris(1,3-dichloro-2-propyl)phosphate</td>
<td>13674-87-8</td>
<td>TDCPP</td>
</tr>
<tr>
<td>Tris(2-butoxyethyl)phosphate</td>
<td>78-51-3</td>
<td>TBEP</td>
</tr>
<tr>
<td>Tris(2-ethylhexyl)phosphate</td>
<td>78-42-2</td>
<td>TEHP</td>
</tr>
<tr>
<td>Triphenyl phosphate</td>
<td>115-86-6</td>
<td>TPhP</td>
</tr>
<tr>
<td>2-Ethylhexyl diphenyl phosphate</td>
<td>1241-94-7</td>
<td>EHDPP</td>
</tr>
<tr>
<td>Tri-o-cresyl phosphate</td>
<td>78-30-8</td>
<td>ToCrP</td>
</tr>
<tr>
<td>Tritolyl phosphate</td>
<td>1330-78-5</td>
<td>TCrP</td>
</tr>
<tr>
<td>Dibutyl phenyl phosphate</td>
<td>2528-36-1</td>
<td>DBPhP</td>
</tr>
<tr>
<td>Butyl diphenyl phosphate</td>
<td>2752-95-6</td>
<td>DPhBP</td>
</tr>
<tr>
<td>2,2-bis(chloromethyl)trimethylene bis[2-chloroethyl]phosphate</td>
<td>38051-10-4</td>
<td>V6</td>
</tr>
</tbody>
</table>

2.1.1 Current knowledge on selected PFRs

Schematic overviews of the 14 PFRs included in this report are presented below. For some of these substances the classification (hazard symbols) can differ depended on which references are used. In this report we have used the classification from European Chemical Bureau.

2.1.1.1 Triisobutyl phosphate

![Triisobutyl phosphate structure](image)

Characteristics of the compound:

- **CAS No.**: 126-71-6
- **Molecular Formula**: C12H27O4P
- **Molecular Weight (g/mol)**: 266.31
- **Melting Point/range**: 205°C
- **Boiling Point/range**: 205°C
- **Partition Coefficient (log Kow)**: 3.1
- **Hazard Symbols**:

Sources:
- [http://www.chemicalbook.com/ProductIndex_EN.aspx](http://www.chemicalbook.com/ProductIndex_EN.aspx)
Synonyms:
- Phosphoric acid triisobutylester
- TIBP
- Tri-iso-butylphosphate
- Triisobutylphosphate

Applications:
- Antifoam textile
- Catalysis & Chemicals Processing
- Chemical synthesis
- Concrete additives
- Construction
- Construction material
- Glues and adhesives
- Manufacturing of glues and adhesives
- Paper & Board
- Paper Manufacture
- Textile auxiliaries
- Textiles and fibres

Figure 2: The total import of the Triisobutyl phosphate (TiBP) to Norway (red line) and to the Scandinavian countries (Norway, Denmark, Sweden and Finland) (green line). Tonnes per years for 2000 to 2008 (SPIN2000, 2011).
2.1.1.2 Tributyl phosphate

Characteristics of the compound:

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>CAS No.</td>
<td>126-73-8</td>
</tr>
<tr>
<td>Molecular Formula</td>
<td>C12H27O4P</td>
</tr>
<tr>
<td>Molecular Weight (g/mol)</td>
<td>266.4</td>
</tr>
<tr>
<td>Melting Point/range</td>
<td>-70°C</td>
</tr>
<tr>
<td>Boiling Point/range</td>
<td>130°C</td>
</tr>
<tr>
<td>Partition Coefficient (log Kow)</td>
<td>3.5</td>
</tr>
<tr>
<td>Hazard Symbols</td>
<td>Xn – Harmful</td>
</tr>
<tr>
<td></td>
<td>Xi – Irritant</td>
</tr>
</tbody>
</table>

Source:
- [http://www.miljostatus.no/tema/Kjemikalier/Kjemikalielister/Stofflisten/](http://www.miljostatus.no/tema/Kjemikalier/Kjemikalielister/Stofflisten/)
- [http://www.chemicalbook.com/ProductIndex_EN.aspx](http://www.chemicalbook.com/ProductIndex_EN.aspx)

(OECD, 2001)

Synonyms:
- Tributylphosphate
- Tributilfosfato
- Celluphos 4
- Butyl phosphate
- Phosphoric acid tributyl ester
- Tributyle (phosphate de)
- tributyl ester
- Tributylfosfaat
- Disflamoll TB
- Tributoxyphosphine oxide
- Tri-n-butyl phosphate

Applications:
- Catalysis & Chemicals Processing
- Chemical synthesis
- Construction
- Dyestuffs, pigments and optical brighteners
- Hydraulic oils
- Manufacturing of plastics
- Manufacturing of rubber, latex
- Pigments
- Plastic & Polymers
- Rubber, latex
- Specialities
Figure 3: The total import of the Tributyl phosphate (TBP) to Norway (red line) and to the Scandinavian countries (Norway, Denmark, Sweden and Finland) (green line). Tonnes per years for 2000 to 2008 (SPIN2000, 2011).

2.1.1.3 Tris(2-chloroethyl)phosphate

<table>
<thead>
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<th>Characteristics of the compound:</th>
</tr>
</thead>
<tbody>
<tr>
<td>CAS No.:</td>
</tr>
<tr>
<td>Molecular Formula:</td>
</tr>
<tr>
<td>Molecular Weight (g/mol):</td>
</tr>
<tr>
<td>Melting Point/range</td>
</tr>
<tr>
<td>Boiling Point/range</td>
</tr>
<tr>
<td>Partition Coefficient (log Kow):</td>
</tr>
<tr>
<td>Hazard Symbols:</td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>

Source:
- [http://www.miljostatus.no/tema/Kjemikalier/Kjemikalielister/Stofflisten/](http://www.miljostatus.no/tema/Kjemikalier/Kjemikalielister/Stofflisten/)

Synonyms:
- 3CF
- AmgardTCEP
- CEF
- CLP
• Celluflex CEF
• Disflamoll TCA
• Fyrol CEF
• Fyrol CF
• Genomoll P
• NSC 3213
• Niax 3CF
• Niax Flame Retardant 3CF
• Tri(2-chloroethyl)phosphate
• Tri(chloroethyl)phosphate
• Tri(b-chloroethyl)phosphate
• Tris(2-chloroethyl)orthophosphate

Applications:
• Flame retardant
• Additive plasticiser and viscosity regulator

Figure 4: The total import of the Tris(2-chloroethyl)phosphate (TCEP) to Norway (red line) and to the Scandinavian countries (Norway, Denmark, Sweden and Finland) (green line). Tonnes per year for 2000 to 2008 (SPIN2000, 2011).
2.1.1.4 Tris(2-chloroisopropyl)phosphate

**Characteristics of the compound:**

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
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</thead>
<tbody>
<tr>
<td>CAS No.</td>
<td>13674-84-5</td>
</tr>
<tr>
<td>Molecular Formula:</td>
<td>C₉H₁₈Cl₃O₄P</td>
</tr>
<tr>
<td>Molecular Weight (g/mol)</td>
<td>327.6</td>
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<tr>
<td>Melting Point/range</td>
<td>358.5°C</td>
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<tr>
<td>Boiling Point/range</td>
<td></td>
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<tr>
<td>Partition Coefficient (log Kow)</td>
<td>2.59</td>
</tr>
</tbody>
</table>

**Hazard Symbols:**

Source:
- http://www.chemicalbook.com/ProductIndex_EN.aspx
- (Fisk et al., 2003)

**Synonyms:**
- 2-Propanol, 1-chloro-, phosphate (3:1)
- Amgard TMCP
- Antiblaze 80
- AntiblazeTMCP
- Daltoguard F
- Fyrol PCF
- Hostaflam OP 820
- Levagard PP
- TolgardTMCP
- Tris(1-chloro-2-propyl)phosphate
- Tris(1-methyl-2-chloroethyl)phosphate
- Tris(2-chloro-1-methylethyl)phosphate

**Applications:**
- Flame retardants
- Paints, laquers and varnishes
- Adhesives, binding agents
Figure 5: The total import of the Tris(2-chloroisopropyl)phosphate (TCPP) to Norway (red line) and to the Scandinavian countries (Norway, Denmark, Sweden and Finland) (green line). Tonnes per years for 2000 to 2008 (SPIN2000, 2011).

2.1.1.5 Tris(1,3-dichloro-2-propyl)phosphate

Characteristics of the compound:
CAS No.: 13674-87-8
Molecular Formula: C9H15Cl6O4P
Molecular Weight (g/mol): 430.9
Melting Point/range: -64°C
Boiling Point/range: 457.4°C
Partition Coefficient (log Kow): 3.65
Hazard Symbols:

Source:
Synonyms:
- 3PC-R
- Antiblaze 195
- CRP (fireproofing agent)
- FR 10
- FR 10 (phosphate)
- FyrolFR 2
- PF 38
- PF 38/3
- Tris(1,3-dichloroisopropyl)phosphate
- Tris(1-chloromethyl-2-chloroethyl)phosphate
- Tris[2-chloro-1-(chloromethyl)ethyl] phosphate

Applications:
- Flame retardant

There is no official information on import to Scandinavia as the import data are classified as “confidential” in the Spin2000 database. This means that the chemical are used in less than four products (SPIN2000, 2011).

2.1.1.6 Tris(2-butoxyethyl)phosphate

![Chemical structure of Tris(2-butoxyethyl)phosphate]

Characteristics of the compound:
- CAS No.: 78-51-3
- Molecular Formula: C18-H39-O7-P
- Molecular Weight (g/mol): 398.5
- Melting Point/range: -70°C
- Boiling Point/range: 413.9°C
- Partition Coefficient (log Kow): 3.75
- Hazard Symbols:

Source:

Synonyms:
- Ethanol, 2-butoxy-, phosphate (3:1) (7CI,8Cl,9CI)
- Phosphoric acid, tris(2-butoxyethyl)ester (6Cl)
- 2-Butoxyethanol phosphate
- Amgard TBEP
- Hostaphat B310
- Ethanol, 2-butoxy-,1,1',1''-phosphate
- Phosflex T-BEP
- Tri(2-butoxyethyl)phosphate
- Tris(2-n-butoxyethyl)phosphate
- tris-2-Butoxyethyl phosphate
- Tri(butoxyethyl)phosphate

Applications:
- Primary plasticizer for most resins and elastomers
- Floor finishes and waxes
- Flame-retarding agent.

Figure 6: The total import of the Tri(2-butoxyethanol)phosphate (TBEP) to Norway (red line) and to the Scandinavian countries (Norway, Denmark, Sweden and Finland) (green line). Tonnes per years for 2000 to 2008 (SPIN2000, 2011).
2.1.1.7 Tris(2-ethylhexyl)phosphate

Characteristics of the compound:

<table>
<thead>
<tr>
<th>Property</th>
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<td>CAS No.</td>
<td>78-42-2</td>
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<tr>
<td>Molecular Formula:</td>
<td>C24H51 O4 P</td>
</tr>
<tr>
<td>Molecular Weight (g/mol)</td>
<td>434.7</td>
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<tr>
<td>Melting Point/range</td>
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<tr>
<td>Boiling Point/range</td>
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<tr>
<td>Partition Coefficient (log Kow)</td>
<td>4.22</td>
</tr>
</tbody>
</table>

Hazard Symbols:

Source:
- (Fisk et al., 2003)

Synonyms:
- Disflamoll TOF
- TOF
- Trioctylphosphate
- Tri-iso-octylphosphate

Applications:
- Catalysis & Chemicals Processing
- Chemical synthesis
- Flame retardants for polymers
- Manufacturing of peroxides
- Oxidation agents
- Plastic & Polymers
- Plasticizers for polymers
- Polymer auxiliaries
Figure 7: The total import of the Tris(2-ethylhexyl)phosphate (TEHP) to Norway (red line) and to the Scandinavian countries (Norway, Denmark, Sweden and Finland) (green line). Tonnes per years for 2000 to 2008 (SPIN2000, 2011).

2.1.1.8 Triphenyl phosphate

![Triphenyl phosphate structure](image)

**Characteristics of the compound:**

- **CAS No.:** 115-86-6
- **Molecular Formula:** C18H15O4P
- **Molecular Weight (g/mol):** 326.3
- **Melting Point/range:** 48-50°C
- **Boiling Point/range:** 412.4°C
- **Partition Coefficient (log Kow):** 4.59
- **Hazard Symbols:**

Source:
- [http://www.chemicalbook.com/ProductIndex_EN.aspx](http://www.chemicalbook.com/ProductIndex_EN.aspx)
- (Fisk et al., 2003)
Synonyms:
- Phenyl phosphate, (PhO)3PO
- Phosflex TPP
- Disflamoll TP
- Phosphoric acid, esters, triphenyl ester
- Phosphoric acid, triphenyl ester
- Phenyl phosphate ((PhO)3PO)
- Triphenyl phosphate ester
- Triphenyl phosphate (TPP)
- Triphenyl phosphate (C18H15O4P)
- Triphenyl Phosphate (TPP)
- S 4 (phosphate)
- Triphenoxyphosphine oxide
- Celluflex TPP
- Reofos TPP

Application:
- Flame retardants for polymers
- Plastic & Polymers
- Plasticizers for polymers
- Polymer auxiliaries

Figure 8: The total import of the Triphenyl phosphate (TPhP) to Norway (red line) and to the Scandinavian countries (Norway, Denmark, Sweden and Finland) (green line). Tonnes per year for 2000 to 2008 (SPIN2000, 2011).
2.1.1.9  2-Ethylhexyl diphenyl phosphate

![Chemical Structure]

**Characteristics of the compound:**

- **CAS No.:** 1241-94-7
- **Molecular Formula:** C20 H27 O4 P
- **Molecular Weight (g/mol):** 362.4
- **Melting Point/range:** -54°C
- **Boiling Point/range:** 421.2°C
- **Partition Coefficient (log Kow):** 5.73

**Hazard Symbols:**

**Source:**
- (Fisk et al., 2003)

**Synonyms:**
- 2-Ethylhexyldiphenyl phosphate
- Diphenyl 2-ethylhexyl phosphate
- Disflamoll DPO
- Octicizer
- Phosflex 342
- Phosflex 362
- Santicizer 141

**Applications:**
- Flame retardants for polymers
- Plastic & Polymers
- Plasticizers for polymers
- Polymer auxiliaries
Figure 9: The total import of the 2-Ethylhexyl diphenyl phosphate (EHDPP) to Norway (red line) and to the Scandinavian countries (Norway, Denmark, Sweden and Finland) (green line). Tonnes per years for 2000 to 2008 (SPIN2000, 2011).

2.1.1.10 Tri-o-cresyl phosphate

![Diagram of Tri-o-cresyl phosphate]

**Characteristics of the compound:**

- **CAS No.:** 78-30-8
- **Molecular Formula:** C21H21O4-P
- **Molecular Weight (g/mol):** 368.4
- **Melting Point/range:** -25°C
- **Boiling Point/range:** 410°C
- **Partition Coefficient (log Kow):** 5.11
- **Hazard Symbols:** T – Toxic, N – Dangerous for the environment

**Source:**

- [http://www.miljostatus.no/tema/Kjemikalier/Kjemikalielister/Stofflisten/](http://www.miljostatus.no/tema/Kjemikalier/Kjemikalielister/Stofflisten/)
Synonyms:
- Phosphoric acid, tri-o-tolyl ester (8CI)
- NSC 438
- Phosflex 179C
- TOCP
- TOKF
- TOTP
- Tri-o-tolyl phosphate
- Tris(2-tolyl)phosphate
- Tris(o-cresyl)phosphate
- Tris(o-methylphenyl)phosphate
- Tris(o-tolyl)phosphate
- o-Cresyl phosphate

Applications:
- Plasticizer in lacquers and varnishes
- Flame retardants

There is almost no official information on import to Scandinavia as most of the yearly data are classified as “confidential” in the Spin2000 database. This means that the chemical are used in less than four products (SPIN2000, 2011). However, for four years the use in Denmark was from 10 to 100 kg (SPIN2000, 2011).

2.1.1.11 Tritolyl phosphate

![Tritolyl phosphate structure]

**Characteristics of the compound:**
- CAS No.: 1330-78-5
- Molecular Formula: C21H21O4P
- Molecular Weight (g/mol): 368.4
- Melting Point/range: -30°C
- Boiling Point/range: 420°C
- Partition Coefficient (log Kow): 5.11

Hazard Symbols:
- http://www.lookchem.com/cas-133/1330-78-5.html
Synonyms:
- Phosphoric acid, tritolyl ester (8CI)
- Celluflex 179C
- Cresyl phosphate
- Disflamoll TKP
- Durad 124
- Durad 125
- Fyrquel 150
- Imol S 140
- Kronitex
- Kronitex R
- Kronitex TCP
- Lindol
- Lindol XP Plus
- Nissan Unflame TCP
- Phosflex TCP
- Phosphlex 179A
- Phosphoric acid tricresyl ester
- RB 6
- Reomol TCP
- Sansocizer TCP
- SumiguardTBT
- Syn-O-Ad 8484
- T 306
- T 306 (ester)
- TCP
- Tricresol phosphate
- Tricresylorthophosphate
- Tricresyl phosphate
- Tris(methylphenyl)phosphate
- Tris(tolyloxy)phosphine oxide
- Tritolyl phosphate

Applications:
- Flame retardants for polymers
- Plastic & Polymers
- Plasticizers for polymers
- Polymer auxiliaries
- Additive in lubricating and hydraulic oils
- Air filter medium
- Waterproofing
- Additive to heat exchange medium
Figure 10: The total import of the Tritolyl phosphate (TCrP) to Norway (red line) and to the Scandinavian countries (Norway, Denmark, Sweden and Finland) (green line). Tonnes per years for 2000 to 2008 (SPIN2000, 2011).

2.1.1.12 Dibutyl phenyl phosphate

Characteristics of the compound:
CAS No.: 2528-36-1
Molecular Formula: C14H23 O4 P
Molecular Weight (g/mol): 286.3
Melting Point/range
Boiling Point/range 333.3°C
Partition Coefficient (log Kow): 3.23
Hazard Symbols:

Source:

Synonyms:
- Butylphenyl phosphate
- Phosphoric acid, dibutyl phenyl ester
Applications:
- Hydraulic fluid in aircraft engines
- Plasticizer for polyvinyl chloride

There is no official information on import to Scandinavia as the import data are classified as “confidential” in the Spin2000 database. This means that the chemical are used in less than four products (SPIN2000, 2011).

2.1.1.13 Butyl diphenyl phosphate

![Chemical structure of Butyl diphenyl phosphate](image)

**Characteristics of the compound:**
- CAS No.: 2752-95-6
- Molecular Formula: C16H19 O4 P
- Molecular Weight (g/mol): 306.3
- Melting Point/range: 368.4°
- Boiling Point/range: 368.4°
- Partition Coefficient (log Kow):
- Hazard Symbols:

Source:

**Synonyms:**
- Phosphoric acid, butyl diphenyl ester
- Butyldiphenylphosphat

**Applications:**
- Hydraulic fluid and additives in aircraft engines

There is no official information on import to Scandinavia as the import data are classified as “confidential” in the Spin2000 database. This means that the chemical are used in less than four products (SPIN2000, 2011).
2.1.1.14 2,2-bis(chloromethyl)trimethylene bis[bis(2-chloroethyl)phosphate]

Characteristics of the compound:

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>CAS No.</td>
<td>38051-10-4</td>
</tr>
<tr>
<td>Molecular Formula</td>
<td>C13H24Cl6O8P2</td>
</tr>
<tr>
<td>Molecular Weight (g/mol)</td>
<td>583.0</td>
</tr>
<tr>
<td>Melting Point/range</td>
<td>-50.5°C</td>
</tr>
<tr>
<td>Boiling Point/range</td>
<td></td>
</tr>
<tr>
<td>Partition Coefficient (log Kow)</td>
<td>2.83</td>
</tr>
</tbody>
</table>

Source:
- [http://www.wernerblank.com/polyur/chemistry/additives/cas38051104.htm](http://www.wernerblank.com/polyur/chemistry/additives/cas38051104.htm)
- (SCHER, 2007)

Synonyms:
- Phosphoric acid 2,2-bis(chloromethyl)-1,3-propanediyl tetrakis(2-chloroethyl) ester (ECL)
- 2,2-Bis(chloromethyl)-1,3-propanediol 2-chloroethanol phosphate (1:4:2)
- 2,2-Bis(chloromethyl)-1,3-propanediyl bis[bis(2-chloroethyl)phosphate]
- 2,2-Bis(chloromethyl)-1,3-propanediyltetrakis(2-chloroethyl)phosphate
- Chloroalkyl phosphates 2,2-bis(chloromethyl)-1,3-propanediyltetrakis(2-chloroethyl)phosphate
- Amgard V6
- Antiblaze 100

Applications:
- Flame retardant for polyurethane foams

There is no official information on import to Scandinavia as the import data are classified as “confidential” in the Spin2000 database. This means that the chemical are used in less than four products (SPIN2000, 2011).
3. Materials and methods

The purpose of this project was to screen samples from remote area for PFRs. The samples were therefore collected from the Svalbard area by the NPI (Figure 11, page 35). The samples were delivered from other scientific projects to optimize the use of resources.

3.1 Selected species

Eight animal species from Svalbard were included in the present screening project. The collection details are given in Table 2. More information about the species is given below.

Table 2. Overview of species sampled investigated for PFRs.

<table>
<thead>
<tr>
<th>Species</th>
<th>Scientific name</th>
<th>Location</th>
<th>Sampling year</th>
<th>Age</th>
<th>Sex</th>
<th>N</th>
<th>Sample matrix</th>
</tr>
</thead>
<tbody>
<tr>
<td>Capelin</td>
<td>Mallotus villosus</td>
<td>Kongsfjorden</td>
<td>2009</td>
<td>–</td>
<td>–</td>
<td>10</td>
<td>Whole fish</td>
</tr>
<tr>
<td>Black-legged</td>
<td>Rissa tridactyla</td>
<td>Kongsfjorden</td>
<td>2009</td>
<td>Adult</td>
<td>F+M</td>
<td>12</td>
<td>Liver</td>
</tr>
<tr>
<td>kittiwake</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Brünnich’s guillemot</td>
<td>Uria lomvia</td>
<td>Kongsfjorden Bjørnøya</td>
<td>2007</td>
<td>–</td>
<td>–</td>
<td>5</td>
<td>Egg</td>
</tr>
<tr>
<td>Glaucous gull</td>
<td>Larus hyperboreus</td>
<td>Bjørnøya</td>
<td>2010</td>
<td>–</td>
<td>–</td>
<td>12</td>
<td>Egg</td>
</tr>
<tr>
<td>Ringed seal</td>
<td>Phoca hispida</td>
<td>Kongsfjorden</td>
<td>2010</td>
<td>Adult</td>
<td>F+M</td>
<td>10</td>
<td>Blubber</td>
</tr>
<tr>
<td>Harbour seal</td>
<td>Phoca vitulina</td>
<td>Prins Karls Forland</td>
<td>2009</td>
<td>Adult</td>
<td>F+M</td>
<td>10</td>
<td>Plasma</td>
</tr>
<tr>
<td>Arctic fox</td>
<td>Vulpes lagopus</td>
<td>Wijdefjorden-Austfjordnes</td>
<td>2010</td>
<td>Adult</td>
<td>F+M</td>
<td>10</td>
<td>Liver</td>
</tr>
<tr>
<td>Polar bear</td>
<td>Ursus maritimus</td>
<td>West Spitsbergen and Storfjorden</td>
<td>2008</td>
<td>Adult</td>
<td>M</td>
<td>20</td>
<td>Plasma</td>
</tr>
</tbody>
</table>

The Barents- and Arctic Seas fish fauna is characterized by relatively few species (Gjøsæter, 2009). Food web studies have identified capelin (*Mallotus villosus*) as important food for other fish, sea mammals and seabirds (Barrett, 2007; Gjøsæter, 2009). The capelin is a small forage fish of the smelt family found in the Atlantic and Arctic oceans. It forages along the Polar front and at the edge of the ice shelf. In autumn it migrates to the cost of Northern Norway and Kola Peninsula for spawning. Capelin spawns on sandy beaches and sandy bottom at the age of 2-6 years, and most capelins dies after spawning. The capelins wintering area are south of the ice-covered parts of the Barents Sea. Capelin is a fatty fish species that transfer energy from lower trophic to higher trophic level in the Barents Sea ecosystem.

The Barents Sea is a productive ecosystem (Sakshaug et al., 2009). The high primary production generates energy to the secondary production and the large fish and seabird stocks. The estimated numbers of breeding birds in the Barents Sea is about 4 million pairs (Gabrielsen, 2009). The three seabird species black-legged kittiwake, Brünnich’s guillemot (*Uria lomvia*) and glaucous gull (*Larus hyperboreus*) represent the avian predators. These three species were chosen because they forage in different levels of the marine food web. The black-legged kittiwake forages in the upper water masses. The kittiwake diet consists mainly of small fishes, such as capelin and polar cod (*Boreogadus saida*), and different pelagic amphipods. The Brünnich’s guillemot is a diver and eats mainly small fish and squids, while...
the glaucous gull is an omnivore predator. Its diet ranges from low food chain items such as crustaceans, through fish to high food chain items such as seabird eggs, chicks and adults and carrion (Barry and Barry, 1990; Erikstad, 1990).

The black-legged kittiwake is a medium-sized gull that has a circumpolar distribution. It is a cliff breeder in the Arctic and boreal zone throughout much of the northern hemisphere. The kittiwake is the most numerous gull species, but is classified as vulnerable in Norway and close to endangered in Svalbard in the Norwegian Red list due to large reductions of the breeding populations around the northern hemisphere. Black-legged kittiwakes are not true migrants, but they disperse widely over most of the North-Atlantic Ocean outside the breeding season. The breeding population in Svalbard is estimated to be 270,000 pairs, of which ca. 90,000 pairs breed on Bjørnøya (Strøm, 2006).

The Brünnich’s guillemot is a stout built auk with a high latitude circumpolar distribution in Arctic and sub-Arctic seas. It is one of the most numerous seabirds in the northern hemisphere with an estimated breeding population in Svalbard of 850,000 pairs. The Brünnich’s guillemot breeds on narrow cliff ledges, lays only one egg that both parents breed for about 32 days (Strøm, 2006).

The glaucous gull is a large gull. It has a circumpolar, high arctic breeding distribution. The glaucous gull breeds throughout most of Svalbard, either as single pairs or in small colonies. Their breeding areas are most often situated close to colonies of other seabirds. Glaucous gulls can also breed on small islets together with colonies of common eiders and geese. The glaucous gull is the only numerous avian predator in Svalbard. It feeds on a wide variety of fish, molluscs, crustaceans, eggs, chicks and adults of other seabirds, insects, carrion, refuse and offal. Glaucous gulls usually nest close to seabird colonies, lays 1-3 eggs and both parents breed for 27–28 days. Little is known about the trend in the total Svalbard population, but the population on Bjørnøya has shown a decline since 1986.

Four mammalian species were included in this screening. The ringed seal (Phoca hispida), the harbour seal (Phoca vitulina), the polar bear (Ursus maritimus) and the arctic fox (Vulpes lagopus). The ringed seal was chosen since it links the marine lower tropic levels with the higher trophic level. It does this by foraging on fish and crustacean, and is itself the most important food for the polar bear. Ringed seal leftovers from polar bear hunts are also eaten by arctic foxes, glaucous gulls and ivory gulls (Pagophila eburnea). The polar bear was chosen as the top predator in the marine environment, while the arctic fox represent both the terrestrial and marine food chain in Svalbard.

The ringed seals are small phocid seals with a circumpolar distribution. They are the only northern seal that can maintain breathing holes in thick sea ice and this special ability allows them to have an extensive distribution in the Arctic and sub-Arctic. Ringed seals give birth in the early spring to their single pup inside a snow lair constructed over a breathing hole in the sea ice. Ringed seals are a long-lived species that can reach 45 years of age. Ringed seals are the most abundant arctic seal and the Svalbard-Barents Sea population is thought to number in the hundreds of thousands (Kovacs and Lydersen, 2006).

The harbour seal are midsized phocid seal that varies in size across the range of the species. In Svalbard adult males are an average of 1.5 metres long and weigh on average 104 kg, while females are somewhat smaller in both length (1.4 m) and weight (83 kg). Harbour seals have one of the broadest distributions among the pinnipeds, ranging from temperate areas as far south as southern California to arctic waters of the North Atlantic and the North Pacific Oceans. The Svalbard population is genetically distinct and by far the world’s northernmost population of this species. In the Archipelago most harbour seals occur on Prins Karls
Forland, an island on the west coast of Spitsbergen where they can be seen year-round. The global population size is probably close to half a million animals. In Svalbard, the population is comprised of about 1,000 individuals. Harbour seals can live to be 30-35 years old, but in Svalbard the oldest one registered thus far is 22 years of age. Harbour seal pups are born during early–mid June in Svalbard. Harbour seals are opportunistic feeders that eat a wide variety of fish species and some cephalopods and crustaceans. The Svalbard population of harbour seals is on the National Red List, and is totally protected from hunting.

The arctic fox is the only mammalian terrestrial predator in the Svalbard archipelago. It has a circumpolar distribution and lives in two main tundra habitats, inland and coast, depending on the availability of food resources. In Svalbard they belong to the coastal ecotype feeding from both the marine and terrestrial food web. In Svalbard arctic foxes mate from February until mid April and average litter size is five to six cubs born in a den in May or early June. Average longevity is about three to four years, but a 13 year old individual has been recorded from Svalbard (Fuglei, 2006).

The polar bears are the largest species of bear in the world. They are markedly sexually dimorphic, with males being larger than females. The polar bear has a circumpolar distribution. There are approximately 20 different populations of polar bears in the Arctic, estimated to together contain approximately 25,000 bears. The Barents Sea population of polar bears counts about 2,500 polar bears and includes animals that den in both Svalbard and Franz Josef Land. Only pregnant female polar bears den during the winter to give birth to 1-3 cubs. She leaves the den with its cubs in late March or early April. Survival of juveniles is only about 30 % to the age of two years. Adult survival is high and a bear typically live to be 15-25 years old (Aars, 2006).

Picture 1: Picture of capelin (*Mallotus villosus*), © NPI.
Picture 2: Picture of black-legged kittiwake (*Rissa tridactyla*), © Kjetil Sagerup NPI.

Picture 3: Picture of Brünnich’s guillemot (*Uria lomvia*), © Hallvard Strøm NPI.
Picture 4: Picture of glaucous gull (*Larus hyperboreus*), © Anja Johansen Haugerud NPI.

Picture 5: Picture of ringed seal (*Phoca hispida*), © NPI.
Picture 6: Picture of harbour seal (*Phoca vitulina*), © Kit Kovacs NPI.

Picture 7: Picture of arctic fox (*Vulpes lagopus*) in summer fur, © Eva Fuglei NPI.
3.2 Species, sampling procedures and areas

The samples were analyzed individually and the sample matrixes were whole fish, egg, liver, blubber or blood plasma (Table 2). The capelin were trawled, sorted into the two sexes and directly frozen (-20 °C) in ziplock bags. Black-legged kittiwake were killed by neck dislocation and brought to the laboratory for dissection. A liver sample from each individual was placed in aluminum foil and a ziplock bag and was frozen (-20 °C). Brünnich’s guillemot and glaucous gull eggs were collected, wrapped individually in aluminum foil and a ziplock bag and stored frozen (-20 °C). Ringed seals were shot on the sea ice and the samples were taken from the carcasses on the ice. The blubber sample was cut out and placed in a ziplock bag frozen (-20 °C) within five hours. The blood samples from harbour seal were collected in vacuum tubes and centrifuged in a field laboratory. Plasma was collected and the samples were frozen in Eppendorf tubes. The Arctic fox samples came from trapped foxes. The carcasses were frozen by the trappers and brought to the Governor of Svalbard. The carcasses were stored frozen from 6 to 9 months until they were brought to the laboratory for dissection. A liver sample was individually packed in aluminum foil and stored frozen (-20 °C) until analyzes. Blood from polar bears was centrifuged in the field. Plasma was transferred to cryogenic vials and immediately frozen (-20 °C). Only new and clean equipments and material were used to handle the samples. All samples were stored frozen at -20°C or lower until analyzes.

The geographical areas of sample collection are given in Figure 11, while the location of each sample is given in Table 2.
Figure 11: Map of Svalbard which shows the names of the location for sampling. Sampling information for each species is given in Table 2.

Permission to collect samples was attended to each of the project that collected samples for this screening. All sampling was done in accordance to current regulations of the Norwegian Animal Welfare Act and all sampling was approved by the Governor of Svalbard.
3.3 Chemical analysis

PFR and lipid analyses were performed at the Institute for Environmental Studies (IVM) of VU University. The lipid analyses in blood were performed by the VU medical centre (VUmc).

3.3.1 PFR analysis biota

Samples of biota were homogenized with a blender and freeze dried, and further homogenized with a mortar. Based on the estimated lipid content an aliquot equal to 250 mg of lipids was extracted with accelerated solvent extraction (ASE350) after addition of internal standards (trimethyl-phosphate-d9, tris-n-butyl-phosphate-d27, triphenyl phosphate-d15, and tripentyl phosphate).

Dichloromethane:aceton (1:1, v/v) was used as extraction solvent. An aliquot of the extract was evaporated to dryness to determine the lipid content (extractable lipid content). The rest of the extract was evaporated to remove the acetone and transferred to isooctane followed by a silica gel clean-up to fractionate the PFRs from the matrix (lipids). A first fraction, containing the matrix was discarded using 15 % diethyl ether (DEE) in hexane. A second fraction, containing most of the cyclic PFRs, was collected with 15 % DEE in hexane. The aliphatic PFRs were collected in a third fraction with acetone. Fraction 2 was transferred to methanol/water and analysed by LC-MS/MS. Fraction 3 was further cleaned with a hydridSPE column (Supelco), and transferred to methanol/water for analysis by LC-MS/MS. All samples were evaporated to 200 µL.

3.3.2 PFR analysis blood

Samples of blood were taken, formic acid/2-propanol (4:1, v/v) and internal standards added. The samples were sonicated for 1 min. Then water/2-propanol (4:1, v/v) was added and mixed for 1 min. Finally, the precipitated plasma was diluted with water and mixed for 1 min. The precipitated sample was loaded on Oasis HLB SPE cartridges (Waters, Milford, USA), washed with 5 % methanol, and dried. PFRs were eluted from the cartridge with dichloromethane/isoctane (3:1). The sample was evaporated and transferred to methanol/water and analysed by LC-MS/MS.

3.3.3 LC-MS/MS and GC-MS analysis

LC separation was performed with a Luna (150 mm x 3 mm x 3 µm C18 column) using formic acid and a gradient of methanol/water at a flow rate of 0.25 ml/min using an Agilent 1200 HPLC system. The gradient from solvent A (methanol/water, 20:80) to solvent B (100 % methanol) was as followed: 0 min (55 % B), 0.5 min (70 % B), 27 min (100 % B), 44 min (55 % B). PFRs were detected with an Agilent 6410 triple quad MS system using electrospray ionization tandem mass spectrometry, in the multiple reaction monitoring (MRM) mode. Quantification was based on internal standards and an external calibration curve of the compounds of interest.

The GC-MS separation was performed on SGE BPX-5 column (25m x 0.22 mm x 0.25 µm) with a flow rate of 1.3 ml/min, helium was used as carrier gas. An Agilent 6890 gas chromatograph was coupled to an Agilent 5975 MS system. One µl of the extract was splitless injected at 250°C. The GC-MS was operated in the electron impact mode using selective ion monitoring (SIM). The oven temperature program was as follows: initial temperature 90°C, followed with 15 °C/min to 190°C, and finally with 10 °C/min to 310 °C with a hold time of 14 min. Quantification was based on deuterated internal standards and an external calibration curve of the PFR.
3.3.4  **Lipid**  
In plasma and serum samples triglycerides and cholesterol were determined by VUmc using an enzymatic colorimetric assay.

3.3.5  **Quality control**  
For the identification and quantification of PFRs the retention time, relative retention time to internal standards, the ratio of target and qualifier masses (GC-MS), and ratio of parent and daughter ions (LC-MS/MS) were used. A signal-to-noise ratio of 3:1 was used.

At each batch of samples the blank procedures was determined to determine background levels. If PFRs were present in the blank the long-term blank levels used for correction. In addition, in each batch of samples a sample was spiked to determine the recovery. It is estimated that the analytical method used in the current screening has and uncertainty about 20-40% which is acceptable for this type of studies.

Interlaboratory studies are not available for these compounds. In 2011 IVM together with the NORMAN network will organize a first interlab study on the analysis of PFR.

3.4  **Statistical analysis**  
Due to the fact that most of the analyzed samples had lower levels of PFRs than the detection limit (DL), the statistical procedure needed to be simple. The results are therefore presented as numbers of samples above DL and the mean are calculated only for results above DL. All data are further presented in the appendix.
4. Results and discussion

Fourteen PFRs (Table 1, page 9) were analyzed in eight species (Table 2, page 28) from the Svalbard area. Ten to 20 samples of each species were analyzed, resulting in a total of 94 different samples. Since these samples were all analyzed for the 14 PFRs a total of 1316 different measurements were made. Results from these 1316 measurements show that only 7\% (n = 98) of the samples were above the instruments detection limit for these PFRs (Table 3, page 40).

The TBP, ToCrP, DBPhP and V6 were not found in any of samples (Table 3). Further, TiBP, TDCCP and DPhBP were detected in less than 30\% of the samples of a single species (Table 3). These three components are therefore accounted as not detected in these species due to quality and security reasons. The components TCEP, TCPP, TBEP, TEHP, TPhP, EHDPP and TCrP were found in 30\% or more of the samples for at least one of the species investigated in the present study. This is in agreement with the result from the former screening of PFRs in Arctic fish and seabirds (Evenset et al., 2009).

EHDPP in capelin, with a mean value of 7.6 ng/g wet wt, and the TBEP in arctic fox, with a mean value of 132 ng/g wet wt, were quantified a step above the detection level. All the other PFR components were quantified just above the detection limit. The EHDPP and TBEP were the PFRs found at the highest concentrations in sewage sludge in Sweden (Haglund and Marklund, 2005; Marklund et al., 2005a). However, the EHDPP and TBEP did not dominate in samples of aquatic biota from Sweden (Sundkvist and Haglund, 2008). In these sample of aquatic biota (fish muscle) TCPP was detected at the highest concentrations, while EHDPP was found at one magnitude lower concentration (Sundkvist and Haglund, 2008; Sundkvist et al., 2010). In present study, both TCPP and TPhP were detected above detection limit in all the capelin samples, but at lower concentration than the EHDPP (Table 3). The composition and concentration of PFRs in the capelin are however in a good agreement with the former PFR screening study on fish and seabirds from Svalbard (Evenset et al., 2009). As in the present study, EHDPP was previously found at highest concentrations of all PFRs in fish from Svalbard (Evenset et al., 2009). Evenset et al. (2009) found EHDPP in all black-legged kittiwake and common eider samples, while the present study only found low levels of EHDPP in 25\% of the black-legged kittiwake samples. In the Brünnich’s guillemot and glaucous gull samples EHDPP was not detected.

The TBEP was found in 50\% of the arctic fox samples and the concentrations were the highest measured in this study. Arctic fox is part of both the marine and terrestrial food chain. The observation of high levels of TBEP in 50\% of the foxes may indicate that this compound is related to the terrestrial food chain, since it was not detected in the other marine species. Interestingly, the octanol-air partition coefficient (a measure for how well a chemical is retained in the body of air-breathing animals) for TBEP is relatively high (logKoa 13.06 EPI Suite™) suggesting that this compound has a slow respiratory elimination. Also TEHP, the only other compound detected in arctic fox, has a high logKoa (14.98), which suggests that the accumulation of both compounds is probably due to high gastrointestinal uptake and slow respiratory elimination. The TBEP were present in both females and males and in both fat and lean arctic foxes. Further, no correlation between body condition and the concentration of TBEP was found as the two highest concentrations were found in a lean female and a fat male, respectively. All arctic foxes were trapped at Austfjordnes (Figure 11, page 35). Austfjordnes is about 55 km from the nearest settlement (Pyramiden) and about 100 km away from Longyearbyen in Isfjorden. It is therefore unlikely that five of the arctic foxes accidentally had eaten something with TBEP from the settlements. Even though that the concentration of TBEP in arctic fox was about 10 times higher than in the only sample of capelin with TBEP and 10-100 times higher than for the other PFRs found in present study, TBEP was present in only 50\% of the arctic foxes. It is not possible to explain why half of the arctic foxes had high levels...
of TBEP, while it was not detected in the other half. However, this study has not included other species from the terrestrial food chain. As far as we know is the knowledge of PFRs in the terrestrial environment is scarce. The TBEP was further detected in one (10 %) of the capelin samples (Table 3). This compound was found in high concentration in sewage sludge in Sweden (Haglund and Marklund, 2005; Marklund et al., 2005a), but it was only detected in the Swedish fish samples close to known sources (Sundkvist and Haglund, 2008; Sundkvist et al., 2010). The TBEP was not found in the Arctic fish and seabird samples (Evenset et al., 2009).

Five of the seven PFRs, found in 30 % or more of the samples for at least one of the species, were found in the fish species capelin (Figure 12, at this page). The capelin is an important food item in the arctic marine food web. The birds and mammals are all above capelin in the food chain. They are therefore either directly or indirectly dependent of capelin as a nutrition source. A POP normally undergoes biomagnification, meaning that the levels of that POP increase upwards the food chain. PFRs have been observed to biomagnify up to fish in the aquatic food chain (Muir et al., 1985; Sundkvist et al., 2010). However, the present study indicates that further accumulation upwards the food chain of the five detected PFR (TCEP, TCPP, TPhP, EHDPP and TCrP) does not occur. It is expected that biotransformation of the studied PFRs occurs rapidly. For most PFRs, EPI Suite™ estimates that half-lifes in fish are less than days, suggesting fast biotransformation.

Figure 12: The organophosphorous flame retardants (PFRs) in eight Arctic species (ng/g wet wt). Only components found in ≥ 30 % of the samples were included. Species BLK – Black-legged kittiwake, C – capelin, HS – harbour seal and AF – arctic fox. PFRs were not present in ≥ 30 % of the samples in Brünnich’s guillemot, glaucous gull, ringed seal or polar bear.
Table 3: The concentration of organophosphorous flame retardants (ng/g wet wt) in Arctic biota. Only samples above the detection limit are included in the calculated mean level. The n state the number of samples included in the calculation and the total number of samples analyzed for this species. Empty cells means that all samples from this analyze were below the detection limit.

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<th>Glaucous gull</th>
<th>Ringed seal</th>
<th>Harbour seal</th>
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<td>Egg n mean</td>
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The SPIN2000 database (SPIN2000, 2011) includes data of annual import and use of regulated chemicals in Scandinavia (Norway, Sweden, Denmark and Finland). The import data (Figure 2-10) of PFR to the Scandinavian countries were plotted as mean annual import per year for the period 2000-2008 (Figure 13). Further, the PFR concentrations from capelin were included in the figure (Figure 13). The figure clearly shows that the five PFRs (TDCPP, ToCrP, DBPhP, DPhBP and V6) that are classified as “confidential” in the SPIN2000 database (SPIN2000, 2011) were not detected in Svalbard. Confidential means that the chemical is used in less than four products (SPIN2000, 2011). The TiBP and TBP were not detected in the present study, but were present in fish from Svalbard in an earlier study (Evenset et al., 2009). The TBEP and TEHP were only present in 10-20% of the capelin samples, but in 50% and 30% of the arctic fox samples, respectively.

The seven components TCEP, TCPP, TBEP, TEHP, TPhP, EHDPP and TCrP clearly undergo long range transport as local sources of PFRs probably doesn’t exists in Svalbard. In addition TIBP, TBP and DBPhP have also been detected in a previous study from Svalbard (Evenset et al., 2009).

Figure 13: The mean import of organophosphorous flame retardants (PFRs) to Scandinavia, tonnes pr year for the period 2000 – 2008. PFRs in black-legged kitiwake, capelin, harbour seal and arctic fox in Svalbard, mean concentration (ng/g + 1 wet weight). The PFR is only present if ≥ 30% of the samples were above detection limit.
5. Concluding remarks

The present study detected seven of the 14 examined organophosphorous flame retardants (PFRs) in animals from Svalbard. Five of these seven PFRs were found in capelin, two in arctic fox, one in black-legged kittiwake and one in harbour seal (Table 4, page 42). The presence of the seven components in biota from the remote Svalbard area indicates that these compounds undergo long range transport and are, at least to some degree, persistent and therefore of environmental concern. With the exception for TBEP in the arctic fox, all the present components were found at very low levels. TCEP, TCPP, TEHP, TPhP and TCrP were all found just above the detection limit, while EHDPP was found with a mean of 7.6 ng/g wet wt in capelin (Table 3).

The capelin is an important food item for higher trophic species in the arctic food chain. Five of the PFRs were present in the capelin, but not in the species higher up in the food chain. Only TCEP was detected in 40 % of the black-legged kittiwake samples. This observation supports an assumption that these five PFR (TCEP, TCPP, TPhP, EHDPP and TCrP) do not biomagnify above the level of fish in the food chain. However, a conclusion cannot be drawn since the number of observation above the detection limit was low.

The observation of high levels of TBEP in 50 % arctic foxes, and not in marine species, may indicate that this compound is related to the terrestrial food chain.

Table 4: The organophosphorous flame retardants examined was found in four of the eight species. Only components presented in ≥ 30 % of the samples within a species are presented.

<table>
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<th>Name</th>
<th>CAS-number</th>
<th>Abbreviation</th>
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<td>Capelin, kittiwake</td>
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<td>Capelin</td>
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6. References


7. Appendix 1 – Tabulated data

Appendix 1, page 1: Concentration of organophosphorous flame retardants (ng/g wet wt) in black-legged kittiwake (BLK) and capelin (C).

<table>
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<th>SPECIES</th>
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<th>TBP</th>
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Appendix 1, page 2: Concentration of organophosphorous flame retardants (ng/g wet wt) in Brünnich’s guillemot (BG) and glaucous gull (GG).

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Appendix 1, page 4: Concentration of organophosphorous flame retardants (ng/g wet wt) in arctic fox (AF) and polar bear (PB).

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Fosfororganiske flammehemmere i Arktis biota
Organophosphorous flame retardants in Arctic biota

Resultatene fra denne undersøkelsen indikerer at sju (TCEP, TCPP, TBEP, TEHP, TPhP, EHDPP and TCrP) av de 14 undersøkte fosfororganiske flammehemmerne (PFR) kan langtransporteres til arktiske områder. Fem av sju PFR ble funnet i fisk; lodde (Mallotus villosus), mens høyeste konsentrasjon (TBEP) ble funnet i fjellrev. Ingen av de sju påviste stoffene ser ut til å oppkonsentres (biomagnifiseres) i næringskjeden.

These results indicate that seven (TCEP, TCPP, TBEP, TEHP, TPhP, EHDPP and TCrP) of the 14 analyzed organophosphorous compounds (PFR) undergo long-range transport to the Arctic. Five of the seven PFRs were found in capelin (Mallotus villosus), while the highest concentration (TBEP) was measured in arctic fox (Vulpes lagopus). The compounds seem not to biomagnify from fish to seabirds or mammals in the Arctic marine food chain.
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
- foruring (sur nedbør)
- ozon (ved bakken og i stratosfæren)
- klimagasser
- miljøgifter

Overvåkingsprogrammet skal gi informasjon om tilstanden og utviklingen av forurensningssituationen, 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.

SPFO-rapport 1092/2011
TA-2791/2011