





# Bronopol, Resorcinol, m-Cresol and Triclosan in the Nordic Environment

## **Bronopol, Resorcinol, m-Cresol and Triclosan in the Nordic Environment**

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*Nordic cooperation* is one of the world's most extensive forms of regional collaboration, involving Denmark, Finland, Iceland, Norway, Sweden, and three autonomous areas: the Faroe Islands, Greenland, and Åland.

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*Nordic cooperation* seeks to safeguard Nordic and regional interests and principles in the global community. Common Nordic values help the region solidify its position as one of the world's most innovative and competitive.

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# Preface

Since 2001 the Nordic countries have systematically been screening the environment for potentially hazardous substances. The aim of the Nordic environmental screening of substances is to obtain a snapshot of the occurrence of potentially hazardous substances in the environment both in regions most likely to be polluted as well as in some background areas. The focus is on little known, anthropogenic substances and their derivatives, which are either used in high volumes or are likely to be persistent and hazardous to humans and other organisms. The substances of focus in this study, bronopol and resorcinol, are included due to their toxicity and volume, and not because they are very persistent or bioaccumulating. This was done to gain experience in environmental screening of this kind of substances. If the substances subjected for screening are found in significant amounts this may result in further investigations or monitoring on national level.

The Nordic screening project is run by a project group with representatives from the National Environmental Research Institute of Denmark, the Finnish Environment Institute, the Environment and Food Agency of Iceland, the Food-, Veterinary and Environmental Agency of the Faroe Islands, the Norwegian Pollution Control Authority and the Swedish Environmental Protection Agency. The selection of the substances was made by the project group.

The project is financed and supported by the Nordic Council of Ministers through the Nordic Chemicals Group and the Nordic Monitoring and Data Group as well as the participating institutions. The chemical analyses have been carried out jointly by the Norwegian Institute for Air Research (NILU) and the Swedish Environmental Research Institute (IVL).

The respective participating Nordic countries organised sample selection and collection and transport of samples based on a sample protocol and manuals provided by the analytical laboratories.





# Summary

The aim of this screening programme was to investigate the occurrence of bronopol and resorcinol in environmental samples from the Nordic countries. In order to allow for some comparisons two other chemicals, *m*-cresol and triclosan, both with known environmental distribution and earlier subjected to screening, were added to the programme.

The compounds selected for this screening are widely used. Because of their application they may end up in the environment through various waste streams. In the screening the concentrations of the chemicals were determined in a variety of media, collected at different geographical sites and representing different source characteristics e.g. point sources and dispersed use related to many activities and products. An additional aim was to highlight important transport pathways and to estimate current emissions.

Despite a high consumption of *bronopol* in the Nordic countries as well as in the EU, it was not found in any of the samples analyzed. Bronopol undergoes rapid hydrolysis as well as biodegradation, which may explain its absence in the environmental matrix samples.

*Resorcinol* is widely used and was also frequently found in the municipal influent water samples. Despite that it is significantly reduced in the Sewage Treatment Plants (STPs) along with a reported high biodegradability, it is still present in some effluent water samples. More importantly it was also detected in environmental samples of water and sediment. Concentrations of resorcinol are higher than the concentration of triclosan in environmental samples outside point sources but are lower or in the same range as triclosan in environmental samples outside municipal STPs. In contrast to triclosan resorcinol seem to be more present in water samples than in sediment samples.

*Triclosan* was found in high concentrations in STP sludge. This may be a reflection of the use of triclosan in health care products. Triclosan was also frequently detected in STP effluent water and in sediment outside STPs. The present study shows that, despite a stated decreased usage of the compound within the industry, triclosan is still detected in and around STPs and in some environmental samples.

The registered usage volume of *m*-cresol is only available for Sweden, however, the compound was found in several influent and effluent samples from municipal STPs in Denmark, Finland and Sweden and in leachate water samples in Finland and Sweden. In effluent water and in environmental samples it was however mainly found in the vicinity of an industrial pulp and paper STP in Finland. Earlier studies have shown the importance of emissions from biomass burning for this compound and

this along with the diffuse spreading of the substance from products not registered in SPIN seem to be of importance for municipal and industrial STP concentrations. A high removal efficiency of the substance seems however to limit its environmental distribution.

# Sammanfattning

Målsättningen med denna screening har varit att undersöka förekomst av bronopol och resorcinol i miljöprover från de nordiska länderna. För att möjliggöra jämförelse med andra ämnen har även två referenssubstanser, m-cresol och triclosan, med tidigare känd förekomst och fördelning i miljön inkluderats i studien.

De ingående ämnena har stor användning inom flera olika områden och kan därför nå miljön via flera olika vägar. Ämnena har därför analyserats i flera i olika matriser. Prover har dessutom valts ut med syfte att få en geografisk spridning samt för att representera olika typer av spridningskällor t.ex. punktkällor respektive diffus användning relaterad till produkter.

Trots en hög konsumtion av *bronopol* så väl i de nordiska länderna som inom EU, kunde ämnet inte detekteras i något av de analyserade proverna. Bronopol hydrolyseras snabbt samt har en snabb nedbrytningsprocess vilket kan förklara varför man inte kan återfinna ämnet i miljön.

*Resorcinol* används brett och detekterades också frekvent i det inkommande vattnet till reningsverken. Trots att det reduceras avsevärt under reningsverksprocesserna och trots att ämnet har en hög nedbrytningspotential går det fortfarande att återfinna resorcinol i utgående vatten från reningsverken och i vissa ytvatten och sedimentprover. Koncentrationen av resorcinol var högre än triclosan utanför vissa punktkällor men lägre eller i samma nivå i miljöprover utanför reningsverk. Till skillnad från triclosan förekommer resorcinol oftare i vattenprover än i sedimentprover.

*Triclosan* detekterades i höga koncentrationer i reningsverksslam. Triclosan återfanns också frekvent i utgående vatten från och i sedimentprover utanför reningsverk. Föreliggande studie visar att det trots en påstådd minskad användning av industrin kan triclosan fortfarande detekteras i reningsverksprover och i miljön.

Registrerade användningsvolumer för *m-cresol* finns bara för Sverige, trots detta kunde ämnet detekteras i flera inkommande vattenprover till reningsverk i Danmark, Finland och Sverige. I utgående vatten och i miljöprover återfanns det däremot enbart i närheten av en papersmassfabrik i Finland. Tidigare studier av emissioner från förbränning av biomassa har påvisat denna källas betydelse för halter av m-cresol i miljön. Detta tillsammans med diffus spridning från produkter som inte registreras i spin-databasen tycks vara av stor betydelse för detta ämne avseende reningsverkshalterna. En hög reningseffekt i reningsverken tycks däremot begränsa dess spridning i miljön.



# 1. Frame of the study

The aim of this screening programme was to investigate the occurrence of bronopol and resorcinol in environmental samples from the Nordic countries. In order to allow for some comparisons two other chemicals, m-cresol and triclosan, both with known environmental occurrence and distribution and earlier subjected to screening, were added to the programme.

The compounds selected for this study are widely used chemicals. Because of their application they may end up in the environment through various waste streams. In the screening, the concentrations of the chemicals were determined in a variety of media, collected at different geographical sites and representing different source characteristics e.g. point sources and dispersed use related to many activities and products. Background samples were also taken. An additional aim was to highlight important transport pathways and to estimate current emissions.

The results of this screening project will be part of scientific measures to estimate the environmental risk posed by these chemicals in the vulnerable Nordic ecosystems.

The structures of the chemicals are shown in Figure 1–4.

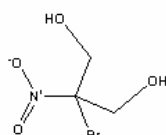


Figure 1. Bronopol  
(2-Bromo-2-nitropropane-1,3-diol, CAS 52-51-7)

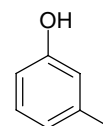


Figure 2. m-Cresol  
(3-methylphenol, CAS 108-39-4)

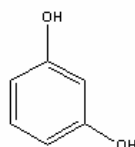


Figure 3. Resorcinol  
(1,3-Benzenediol, CAS 108-46-3)

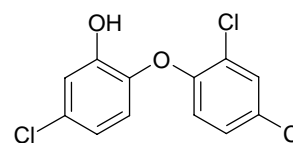


Figure 4. Triclosan  
(2-Hydroxy-2',4,4'-trichlorodiphenylether CAS 3380-34-5)



## 2. Background

### 2.1 Bronopol; Chemical properties, fate and toxicity

Bronopol (2-bromo-2-nitropropane-1,3-diol, CAS 52-51-7) forms white to slightly yellow crystals. Due to its application in many consumer available products, bronopol is released to the environment through various waste streams (see below). The substance is highly soluble in water and not expected to bioaccumulate. Literature references on the issue of the persistence of bronopol are ambiguous. Some state that it is stable to hydrolysis under normal conditions but at increased temperatures and/or higher pH rapid hydrolysis may occur (EPA, 2006 a). Others state that in aqueous solution bronopol is easily transformed to formaldehyde. Other transformation products consist of bromonitroethane, bromoethanol, and bromonitroethanol (Wang, 2002).

When released to water bronopol is not expected to adsorb to suspended solids and sediments nor is vaporisation from the water phase expected to be an important transport pathway (HSDB, 2006). Bronopol is easily biodegradable (EPA, 2006).

**Table 1. Chemical and physical data**

Substance	MW (g/mol)	$W_{sol}$ (g/l)	$V_p$	H (Atm m <sup>3</sup> /mol)	Log $K_{ow}$	BCF (L/kg)	$K_{oc}$	Ref
Bronopol	199.9		$1.3 \cdot 10^{-5}$ mmHg (25°)	$1.3 \cdot 10^{-11}$			5	1
m-Cresol	108.1	22.7 (25°)	0.11 mm Hg (25°)	$8.56 \cdot 10^{-7}$ (25°)	1.96			2
Resorcinol	110.1	1400 (20°)	$2.7 \cdot 10^{-4}$ hPa (25°)	$8.1 \cdot 10^{-11}$ (25°)	0.8-0.97	2.4	65	3
Triclosan	289.6	0.012	$7 \cdot 10^{-4}$		4.8			4

Ref 1: HSDB, 2006; 2: SRC 2006; 3: ESIS, 2006; 4: Samsö-Petersen et al, 2003

When released to air, the vapour pressure indicates that bronopol may exist both in the particulate phase and in the vapour phase where it may react with hydroxyl radicals with an estimated half-life of 97 hours (HSDB, 2006).

For ecological risks, bronopol is practically non-toxic to slightly toxic to birds; slightly to moderately toxic to freshwater fish and terrestrial invertebrates; moderately to highly toxic to estuarine/marine invertebrates; and slightly toxic to estuarine/marine fish (EPA, 2006). Bronopol is classified as harmful if swallowed or in contact with skin; irritating to eyes, respiratory system and skin; risk of serious damage to eyes and very toxic to aquatic organisms (Xn; R21/22 - Xi; R37/38-41 - N; R50; ESIS, 2006).

The ecotoxicological data found in literature for bronopol is only for acute toxic effects. A predicted no effect concentration (PNEC) for water, derived from the most sensitive acute test for bronopol with a safety factor of 1000 (TGD, 2003), is estimated to 0.78 µg/l (Table 2).

**Table 2. Aquatic toxicity for bronopol (US EPA, 2006)**

Scientific name, Common name	Endpoint	Effect Measurement	Duration	Conc (mg/L)
<b>Americamysis bahia</b> Opossum shrimp	LC50	Mortality	96 h	5.9
<b>Crassostrea gigas</b> Pacific oyster	EC50	Immobilization	48 h	0.78
<b>Cyprinodon variegatus</b> Sheepshead minnow	LC50	Mortality	96 h	59

## 2.2 m-Cresol; chemical properties fate and toxicity

Cresol (methyl phenol) occurs as three isomers: o-cresol, m-cresol and p-cresol (also known as 2-methylphenol, 3-methylphenol and 4-methylphenol respectively). The isomer selected for this screening is m-cresol (3-methylphenol, CAS 108-39-4). A bioaccumulation factor of 20 in fish indicates no major bioaccumulation potential in higher trophic levels. A bioaccumulation factor of 4900 in algae indicates however a risk for bioaccumulation in lower organisms (ESIS, 2006).

m-Cresol is classified as toxic through skin contact and toxic if swallowed (R24, 25, ESIS, 2006). A PNEC for water, derived from the most sensitive acute toxicity found in literature with a safety factor of 1000 (TGD, 2003), is here estimated to 6 µg/l (Table 3).

**Table 3. Aquatic toxicity for m-cresol (ESIS, 2006)**

Species	Duration	LC50 (mg/l)	Remarks
<b>Leuciscus Idus</b> (fish)	48	6	
<b>Brachydanio rerio</b> (fresh water fish)	96	15.9	
<b>Salmo gairdneri</b> (estuary, fresh water fish)	96	8.6	
<b>Daphnia magna</b> (crustacea)	48	18.8	
<b>Daphnia magna</b> (crustacea)*	24	8.9	Effect endpoint; immobilisation

## 2.3 Resorcinol; chemical properties, fate and toxicity

Resorcinol (1,3-benzenediol, CAS 108-46-3) is a crystalline, aromatic chemical that is water soluble. Due to its applications the substance may be released to air and water. No hydrolysis is expected to occur due to



lack of hydrolysable functional groups. In the water phase adsorption to suspended solids is expected to be limited due to a low K<sub>oc</sub> (Table 1). The vapour pressure (Table 1) indicates that resorcinol in ambient air mainly will exist in the vapour phase where it may be degraded by photochemically produced hydroxyl radicals with a half-life of 1.9 hours. Assays based on the OECD ready biodegradability and inherent biodegradability protocol indicate that resorcinol is biodegradable (INDSPEC, 2004). The BCF-value (2.4) and Log K<sub>ow</sub>-value (0.8–0.97) indicate no major bioaccumulation.

Resorcinol is classified as harmful if swallowed, irritating to eyes and skin and very toxic to aquatic organisms (X<sub>n</sub>; R22 Xi; R36/38 N; R50; ESIS, 2006). Most ecotoxicological data found in literature within this study were based on acute toxicity tests. However, one test on chronic toxicity to *Daphnia* was found. A PNEC<sub>water</sub>, derived from this study with a safety factor of 100 (TGD, 2003) is estimated to 1.72 µg/l.

An EQS (=NOEC) was set by the Finnish Environment Institute. The EQS was derived from a short term test on *daphnia* with an EC<sub>50</sub> of 0.25 mg/l and a safety factor of 1000 resulting in an EQS of 0.25 µg/l (Londensborough, 2005).

**Table 4. Aquatic toxicity for resorcinol (ESIS, 2006) mg/l**

Species	Duration	LC50	EC50	NOEC	Remarks
<b>Leuciscus Idus</b> (fish)	96 h	34.7		25	
<b>Brachydanio rerio</b> (fresh water fish)	7d (early life stages)	262 (embryo)	54.8 (malformations)		
<b>Daphnia Magna</b> (crustacea)	48 h	0.25			method not according to guideline
<b>Daphnia magna</b> (crustacea)*	21 days		>0.172	0.172	
<b>Palaemonetes pugio</b> (Grasgarnelle, aquatic arthropod)	96 h	42.2			
<b>Chlorella Pyrenoides</b> (algae)	72 h	1.1			method not according to guideline
<b>Scenedesmus</b> (algae)	4 days	60			method not according to guideline

## 2.4 Triclosan; chemical properties, fate and toxicity

Triclosan (2-hydroxy-2',4,4'-trichlorodiphenylether, CAS 3380-34-5) is a relatively lipophilic compound (log K<sub>ow</sub> 4.8) and an ionising molecule with a pK<sub>a</sub> of 8.14 resulting in an even higher lipophilicity when pH is low and the molecule is not ionised. The non-ionised molecule is stable against photochemical reduction in contrast to the ionised molecule (Tixier et al. 2002). Because of its antimicrobial properties triclosan does not meet the criteria set for the standard biodegradability test (OECD 301C, 302C and 303A). Activated sludge tests and continuous activated sludge tests with radiolabelled test material contradicted this and showed instead

an almost complete removal of triclosan from waste water with an absence of persistent degradation materials (Ciba, 1998).

Triclosan is classified as “irritating to skin and eyes”, “very toxic to aquatic organisms” and “may cause long term adverse effects in the environment” (R 36/38; R50/53). A PNEC<sub>water</sub> derived from the most sensitive species for triclosan (Algae; *Scenedesmus*) with a safety factor of 10 is estimated to 0.05 µg/l (Ciba 1998, Reiss et al., 2002)

**Table 5. Ecotoxicity for triclosan (Ciba, 1998)**

Species	End Point	Duration	Concentration
Zebra fish ( <i>Brachidanio rerio</i> )	LC50	96h	0.54mg/l
Rainbow Trout ( <i>Oncorhynchus mykiss</i> )	LC50	96h	0.35 mg/l
Golden Orfe (fish; <i>Leuciscus idus</i> )	LC50	96h	0.56 mg/l
Fathead Minnow (fish; <i>Pimephales promelas</i> )	LC50	96h	0.26 mg/l
Daphnia Magna	EC50	48h	0.39 mg/l
Ceriodaphnia dubia	EC50	48 h	0.13 mg/l
Rainbow trout (fish)	NOEC (Early life stage toxicity (ELS))		24 µg/l
Daphnia magna (crustacea)	NOEC	21 days (OECD 202)	40 µg/l
Ceriodaphnia dubia (crustacea)	NOEC	7days	6 µg/l
<i>Scenedesmus subspicatus</i> (algae)	EC50	96h	1.4 µg/l
<i>Scenedesmus subspicatus</i> (algae)	NOEC	96h	0.5 µg/l

## 2.5 Summary of estimated PNECs

The suggested PNECs for bronopol, m-cresol, resorcinol and triclosan are summarized in Table 6. The PNECs are derived from the most sensitive species for respective compound (see sections above).

**Table 6. Estimated PNEC<sub>water</sub> (µg/l)**

Substance	PNEC	Safety factor
Bronopol	0.78	1000
m-Cresol	6	1000
Resorcinol	1.72	100
Triclosan	0.05	10

## 3. Application, use and previous measurements

### 3.1 Application and use

*Bronopol* is used as a preservative in cosmetics, hygiene products and in chem-technical products such as glue and paint. The substance can also be used as a slimicide and bactericide in industrial processes eg. paper mills, cooling water and air conditioning/humidifying systems (US EPA 2006 a). Products that may contain bronopol include dish detergents, disinfectants, hygienic biocides for veterinary use, pharmaceutical products for skin application, shampoo and bathing products (Wang et al, 2002). The concentration within products range between 0.01 and 0.1% (the maximum level allowed by EC directive 76/768; Weyland 1994).

As a biocide, bronopol is included in the European directive on biocides EG 2032/2003. All usage of the substance should be reported to national agencies.

Bronopol is a low production volume chemical in the European Union (produced or imported in EU with a tonnage > 10t/y but never more than 1000 t/y) with production registered in UK, Germany, Italy and the Netherlands. Because of the many areas of use bronopol is expected to be imported with both industrial and consumer products to the nordic countries. Finland has been the largest consumer of bronopol among the Nordic countries during the last years (Figure 5).

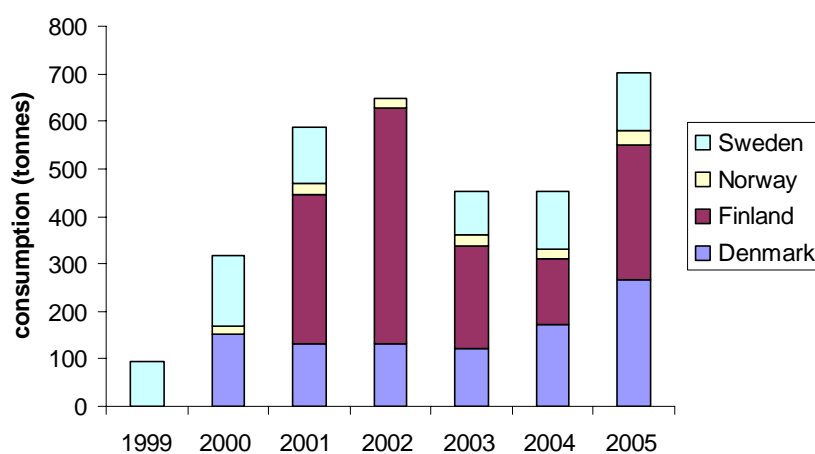


Figure 5. Consumption of bronopol in Nordic countries between 1999–2005

The major use of *m-cresol* is as an intermediate in the manufacture of chemicals and chemical products (SPIN, 2006). *m-Cresol* is also a component in coal tar creosote which is mainly used as a wood preservative, but also in pharmaceutical industry and in smoke flavouring (KEMI, 1994). Furthermore *m-cresol* is a constituent in crude oil (Ioppolo-Armanios, 1996), and as such, added to soap as disinfectant due to its activity as bactericide and fungicide. There have also been reports of its use as antioxidant in some commercial jet fuels to prevent the formation of deposits in aircraft engine fuel systems (ATSDR, 2001). *m-Cresol*, either pure or mixed with *p-cresol*, has also been important in the production of herbicides (ATSDR, 1992).

*m-Cresol* is a high production volume chemical (produced or imported in quantity of at least 1000 tonnes per year in EU by at least one industry) with registered production in Germany, Netherlands, France and UK (ESIS, 2006).

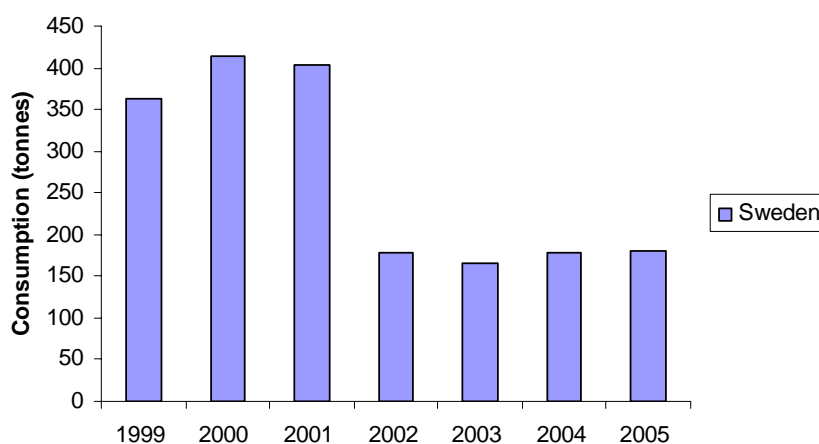


Figure 6. Consumption of *m-cresol* in Sweden 1999–2005

During the last 6 years there has been registered use of *m-cresol* in all Nordic countries. With the exception of Sweden, the use is confidential in the Nordic countries, due to too few registrants. In Sweden the use of *m-cresol* has decreased during these years (Figure 6).

The major intentional use of *m-cresol* is as intermediate in chemical synthesis. Thus, insignificant emissions are expected from use of chemical products. An exception is creosote, which is however only permitted for professional applications. Creosote is used outdoors in sleepers, power lines, enclosures, ports and waterways, where it might be released to the surrounding environment (KEMI, 2004). *m-Cresol* may also be subject to secondary formation in the atmosphere, through reactions between hydroxyl radicals and toluene or xylene respectively (e.g. Klotz et al., 1998). *m-Cresol* may also be formed through biodegradation of toluene

(Neilson, 2000). Thus all sources of toluene and xylene, e.g. gasoline, are potential sources.

Unintentional emissions from biomass burning may also be of importance for environmental concentrations of m-cresol (Johansson et al., 2004a)

*Resorcinol* is an essential component of adhesive systems and an important chemical intermediate in the manufacture of speciality chemicals such as light screening agents for the protection of plastics from exposure to ultraviolet light. Other uses include the manufacture of dyestuffs, pharmaceuticals, flame retardants, agriculture chemicals, fungicidal creams and lotions, explosive primers, antioxidants, chain extender for urethane elastomers and treatment to improve mechanical and chemical resistance of paper machine fabrics (INDSPEC, 2004).

Resorcinol is a high production volume chemical (produced or imported in quantities of at least 1000 tonnes per year in EU by at least one industry) with registered production in Germany and Finland (ESIS, 2006). Because of the many areas of use, resorcinol is expected to be imported with both industrial and consumer products to the Nordic countries.

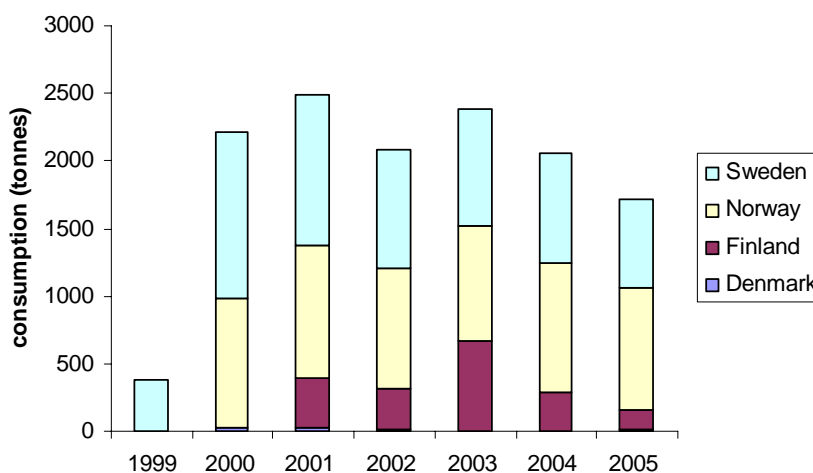


Figure 7. Consumption of resorcinol in Nordic countries 1999–2005

*Triclosan* is a chlorinated phenolic compound that for many years has been used as a biocide in several consumer products. Amongst others it has been used in toothpaste to prevent loosening of teeth, due to bacterial infections, as well as in fibre- and plastic material because of its ability to be incorporated into the material and then slowly leak out during a long period of time. During recent years a lot of attention has been drawn to triclosan and its feared negative impacts on the environment. As a result, many producers have stopped their use of the compound.

Triclosan is a low production volume chemical in the European Union (produced or imported in EU with a tonnage >10 t/y but never more than

1000 t/y) with production registered in Switzerland and Italy. There has been registered use in all Nordic countries during the last years with Sweden as the largest consumer (Figure 8).

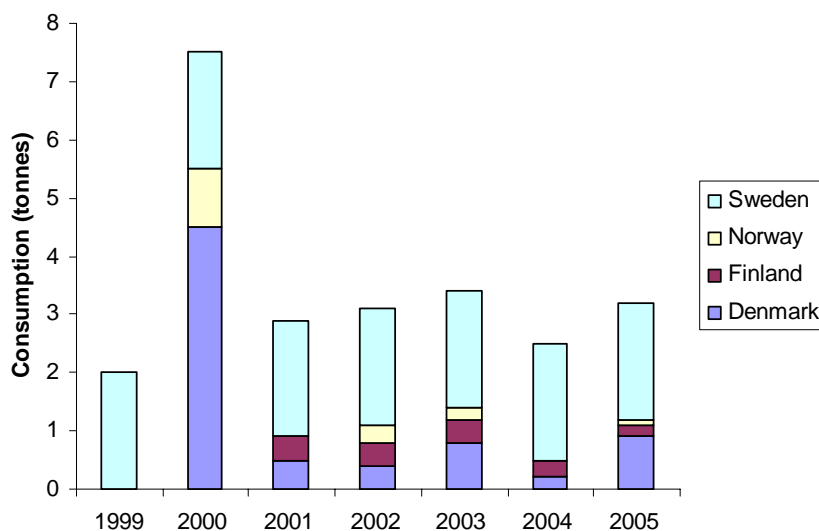


Figure 8. Consumption of triclosan in Nordic countries 1999–2005

### 3.2 Environmental levels and exposure

No literature references on detectable *bronopol* concentrations in the environment were found (Remberger et al., 2006). One literature reference of *resorcinol* measured in industrial sewage water in concentrations of 7–22 mg/l was found (ESIS, 2006). In the Swedish screening 2005 *resorcinol* was occasionally found in sewage water and sludge from municipal STPs in concentrations of 0.78 µg/l and 22 to 380 µg/kg DW respectively (Remberger et al., 2006)

*m-Cresol* was measured in air in two Swedish cities during winter-time, Lycksele in the north and Växjö in the south (Johansson et al., 2004 b, c). The average concentrations for the sampling periods are given in Table 7.

Table 7. Atmospheric levels of *m-Cresol* in two Swedish towns

	Average ng/m <sup>3</sup>	median ng/m <sup>3</sup>	min ng/m <sup>3</sup>	max ng/m <sup>3</sup>	Number
<b>Lycksele Jan–Mar 2002</b>					
Sampling station strongly influenced by domestic biomass burning	4.2	2.7	0.5	18	15
Sampling station influenced by domestic biomass burning	5.6	5.6	1.3	12	15
<b>Växjö Jan–Mar 2002</b>					
City Centre	4.2	2.5	<1	16	14
Sampling station influenced by domestic biomass burning	4.6	3.4	0.6	12	14

Emissions of cresols from small-scale biomass burning have recently been estimated (Johansson et al., 2004a). The emission factors of cresol varied significantly between the 20 domestic combustion devices tested. Emissions occurred regardless of the fuel type (pellets or wood), and their magnitude was mainly influenced by combustion conditions together with the type of combustion device. A study in the US also identified emissions of methyl phenols from small-scale combustion of wood and these were higher than those of benzene (Schauer et al, 2001). Two orders of magnitude lower emissions were obtained during studies of residential wood combustion (McDonald et al., 2000).

**Table 8. Previous measurements of Triclosan in the environment**

Matrix	Site information	Country	Concentrations	Reference
Air	Urban	Sweden	0.01–0.17ng/m <sup>3</sup>	Remberger et al., 2002
Air	Ambient	Sweden	0.003–0.005 ng/ m <sup>3</sup>	Remberger et al., 2002
Deposition	Urban	Sweden	9.7–20 ng/m <sup>2</sup> /d	Remberger et al., 2002
Deposition	Ambient	Sweden	0.2–0.41 ng/m <sup>2</sup> /d	Remberger et al., 2002
Deposition	Ambient	Sweden	1.6–3.5 ng/m <sup>2</sup> /d	Remberger et al., 2006
Fish	Recipient STP	Switzerland	35 ng/g ww	Balmer et al. 2004
Fish	Muscle	Sweden	0.8–13 µg/kg ww	Remberger et al., 2002
Fish	muscle	Norway	0.9–13 ng/g lipid	Fjeld et al., 2004
Fish	Liver (cod)	Norway	1– 96 ng/g lipid	Fjeld et al., 2004
Fish	Muscle	Sweden	0.56 µg/kg ww	Remberger et al., 2006
Sewage water	STP	Switzerland	0.600–1.300 µg/l	Lindström et al., 2002
Sewage water	Influent STP	Sweden	0.1–1.5 µg/L	Reference cited in Samsö-Petersen et al., 2003
Sewage water	Effluent STP	Sweden	0.2 µg/L	Reference cited in Samsö-Petersen et al., 2003
Sewage water	Influent STP	Denmark	2.5–1.7 µg/L	Reference cited in Samsö-Petersen et al., 2003
Sewage water	Effluent STP	Denmark	<1	Reference cited in Samsö-Petersen et al., 2003
Sewage water	Effluent STP	Switzerland	0,042 – 0,213 µg/l	Singer et al.,2002
Sewage water	Influent STP	Norway	0.22– 1.8 µg/l	Möskeland 2006
Sewage water	STP	Sweden	0.02–0.09 µg/l	Remberger et al., 2006
Sludge	STP	Sweden	0.028–6.4 µg/g d.w	Svensson et al.2002
Sludge	STP	Sweden	2800–4400 µg/kg d.w	Remberger et al., 2002
Sludge	STP	Sweden	2000–4300µg/kg d.w	Remberger et al., 2006
Soil	Industrial influence	Sweden	3–15 µg/kg DW	Remberger et al., 2002
Soil and Sediment	landfill	Norway	0.9 –105 ng/g dw	Fjeld et al., 2004
Sediment	Marine, Influence from chemical industries	USA	<d.l.–100mg/kg	Lopez-Avila and Hites, 1980
Sediment	Industrial influence	Sweden	2–25 µg/kg DW	Remberger et al., 2002
Sediment	Fresh water	Norway	0.3–2.8 ng/g	Fjeld et al., 2004
Sediment	marine	Norway	0,02–14 ng/g dw	Fjeld et al., 2004
Sediment	Recipient STP	Norway	20–36 µg/kg TS	Möskeland 2006
Sediment	Recipient landfill	Norway	17 µg/kg TS	Möskeland 2006
Sediment	Urban, ambient	Sweden	3.6–29 µg/kg DW	Remberger et al., 2006
Surface water	River, downstream population centers	USA	Ave= 140 ng/l Max 2300 ng/L	Kolpin et al.,2002
Surface water	Marine, influenced by STPs	Switzerland	1.4–74 ng/l	Lindström et al., 2002
Surface water	Sea & river, Influence from chemical industries	USA	600–40000ng/l	Lopez-Avila and Hites, 1980
Surface water	River, Receiving water from STP	Switzerland	11–98 ng/l	Singer et al.,2002
Surface water	Industrial influence	Sweden	<2–160 ng/l	Remberger et al. 2002

*Triclosan* has previously been measured at several sites in the environment (Table 8). *Triclosan* has also been detected in human breast milk, where three out of five randomly selected sample

s showed levels that were considered as high (60–300 µg/kg lipid weight; Adolfsson-Erici et al. 2002). Triclosan was also detected in different food stuff, such as meat (fat), egg, milk and fish in concentrations between 1.4–8.4 µg/kg ww while it could not be detected in vegetables nor cereals in a Swedish screening study (Remberger et al., 2002).



# 4. Methodology

## 4.1 Sample selection

All samples collected in this screening study are listed in Appendix 1, where also the sampling characteristics are given in detail. An overview of the sampling sites and their spatial distribution in the Nordic countries are shown for biota, sediment, soil, sludge, water and air in figure 9–13.

Each country made a selection of samples based on the knowledge of use and the expected occurrence of bronopol and resorcinol for that particular country. In all cases, samples were chosen to represent point sources as well as diffuse sources and in some cases also background areas. The goal was also to cover all the matrices air, water, sediment, sludge and biota. The strategies and samples selected for the different countries are outlined in the following sections.

### *4.1.1. Denmark*

#### Abiotic samples

Two sewage treatment plants (STP) were selected for screening. Lynetten is located in Copenhagen and receives mixed sewage water from many communities. Bjergmarken STP at Roskilde also receives mixed sewage water from households and industry with a capacity of 125000 PE. Effluent and influent water were sampled over one week.

Surface water was collected from recipients influenced by the STPs i.e. Øresund at Lynetten and the Roskilde Fjord at Roskilde. Samples of surface water and sediment were also collected in two lakes (Slåen sø and Silkeborg Langsø) in the Guden stream system. The Guden stream system is influenced by fish farming. A marine background sample was collected in Kattegat.

#### Biotic samples

Fish samples were collected in the Roskilde fjord and Øresund in the same areas as the surface water samples.

### *4.1.2. Faroe Islands*

#### Abiotic samples

Samples of leachate water from landfill and landfill soil were taken in the vicinity of Tórshavn from the Húsahaga landfill.

Two sewage treatment plants in Tórshavn were sampled; the treatment site of the hospital, and the Sersjantvíkin treatment plant.

Effluent samples were taken from a paint factory in Strendur. The production at the site is seasonal, with spring and summer being the primary production period. The sampling was done at a time of none/limited production.

Sampling of coastal water (seawater) was done in the Tórshavn inner harbour, at the site Vágsbotn. This site, abbreviated BÁT, was used for cod sampling for studying the environmental state of the harbour in 2002 (Havnarvág 2002, [www.hfs.fo](http://www.hfs.fo)) and is one of marked contamination.

#### Biotic samples

Fish of various kinds were sampled in harbour areas. In Tórshavn harbour cod and saithe was sampled; the saithe outside the fish fillet factory, a site also close to the shipyard, and the cod at Bursatanga were the ferries dock. Dab were taken in the marina at Skála and sculpin near the paint factory in Strendur, a site also close to a salmon slaughterery. Samples of liver from 20 fledging fulmars were taken 3/9/05. The birds were caught off the Vestmannacliffs.

#### 4.1.3. Finland

##### Abiotic samples

Two forest industry locations, one chemical industry location and two municipal waste deposits connected to sewage treatment plants were selected for this screening. Site selection was based on possible use of bronopol, since no indication of sites with a risk of resorcinol emissions to the water environment could be identified. Influent and effluent wastewater and sludge was collected from all five sites twice in November 2005. Both forest industry enterprises and chemical industry enterprises were located in South East Finland. Bronopol has been used as slimicide in latex production at Anjalankoski and as slimicide and preservative in pulp/paper production at Kuusankoski and Imatra. The municipal sewage treatment plants of Espoo (pe 240 000) and Lappeenranta (pe 59 000) represent diffuse sources while Kuusankoski (pe 20 000) represents both small-scale industrial and municipal sources. Additionally, leachate water from waste deposits in Espoo and Lappeenranta directed to respective treatment plants were collected twice. One surface water sample (1m depth) and one sediment sample (0–10 cm depth) were taken from two sites, River Kymijoki two km below Kuusankoski sewage treatment plant and Lake Saimaa one km from the factory in Imatra.

#### Biotic samples

Roach (*Rutilus rutilus* L.) from River Kymijoki in Huruksela 20 km below Kuusankoski were caught with gillnets and pooled samples were prepared at IVL laboratory.

#### 4.1.4. Iceland

All Icelandic sites were located inside or close to the capital city Reykjavík.

#### Abiotic samples

Samples of non-dehydrated and non-processed sludge were collected at two sewage treatment plants (STP) in Reykjavík, Klettagarðar (K11 and K12) and Ánanaust (An1 and An2), which handle all sewage from Reykjavík and several neighbouring towns.

Three point source samples were taken to measure output from Álfsnes which is Reykjavík's waste landfill site. River recipient water samples (ALF1 and ALF2) were taken from a runoff stream coming from the Álfsnes site. A coastal recipient water sample (Alf4) was taken close to where the runoff stream enters the sea.

Two point source coastal recipient water samples (GUF1 and GUF2) were taken to measure possible output from an old landfill site at Gufunes in Reykjavík. The site is located near the coast and was used to store landfill waste from Reykjavík and neighbouring towns until 1991. The chemicals in the landfill site are not well known since control on waste dumping was not as strict in those years. Previous measurements have confirmed some seeping of material from the landfill to the sea.

One point source coastal recipient water sample (SNA1) was taken from Snarfari, a small harbour of yachts and motorboats in Reykjavík.

#### Biotic samples

No biotic samples were taken in Iceland.

#### 4.1.5. Norway

The Norwegian samples represent two case studies:

1. Inner part of Oslo fjord, representing pollution from diffuse sources of an urban area with more than 600.000 inhabitants.
2. Northern part of Lake Øyern, representing diffuse sources from a smaller urban population and an industry area where resorcinol is used in the production of glue for wood processing (closed process with a treatment plant for phenolic substances) and water from upstreams rivers including one with effluents from a municipal sewage treatment plant 100 000 p.e feeding into Svellet. This case

was supplemented by samples from a river in East Agder County, close to a factory using resorcinol based glue in wood processing.

#### Abiotic samples

In the inner Oslo fjord area water (untreated and treated) and sludge samples were taken from the two main sewage treatment plants. Water and sediment samples were also taken in the fjord not very far from these plants.

In the Øyern area treated and untreated water samples were taken from an industrial treatment plant in the factory using resorcinol in the production. Water and sludge samples were taken at different distances from the industrial area to the delta and the nearest part of the lake.

#### Biotic samples

A marine fish sample was taken near the fjord water sample site closest to urban Oslo. A lake fish sample was taken in the recipient of the industrial site near Lake Øyern.

#### 4.1.6. Sweden

The Swedish samples were chosen in order to highlight potential releases of the substances into the environment i.e. via point sources or diffuse spreading. Sampling sites were also spread over the country in order to get a geographical distribution of the compounds.

Water effluent samples as well as air samples were taken from a pulp industry in Grums with known use of resorcinol. Water, sediment and biota samples were taken outside the factory in order to highlight the environmental load outside the potential point source.

Seven water effluent samples and seven sludge samples from municipal sewage treatment plants were provided for the Swedish sampling programme. In addition to this, one sample from a wetland adjacent to one of the STPs was included. To further investigate diffuse spreading patterns, two water effluent samples from landfill leachates in Helsingborg and Eskilstuna were added to the Swedish sampling programme.

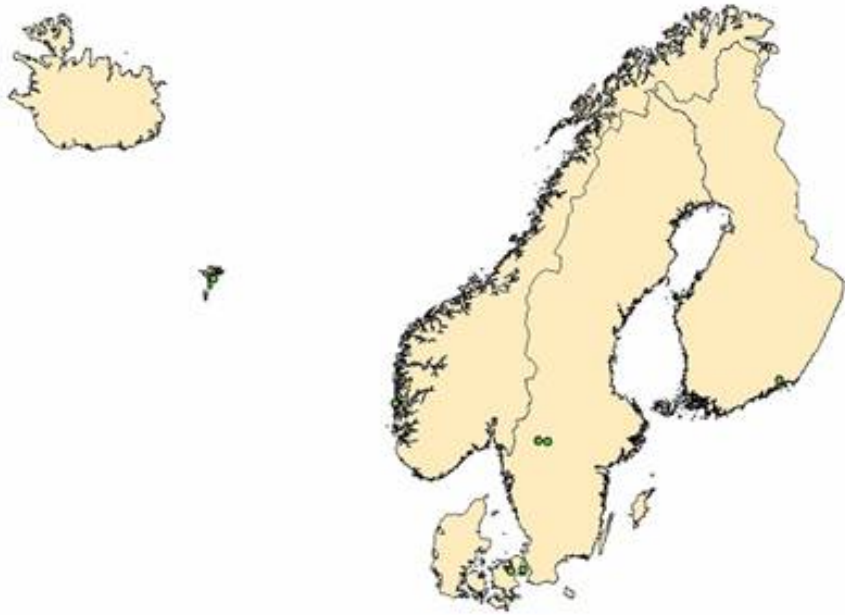


Figure 9. Nordic sampling sites for biota.

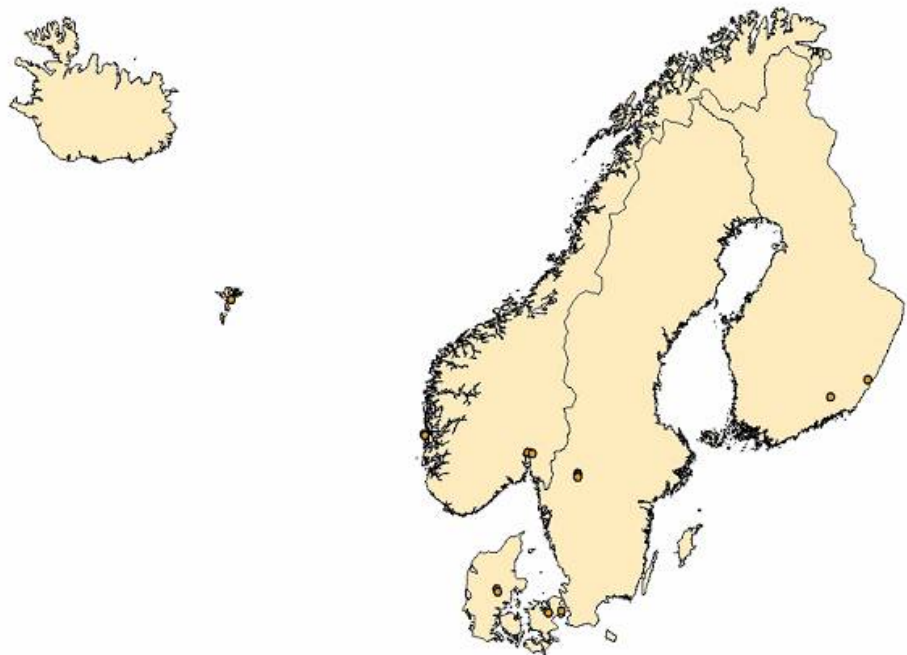
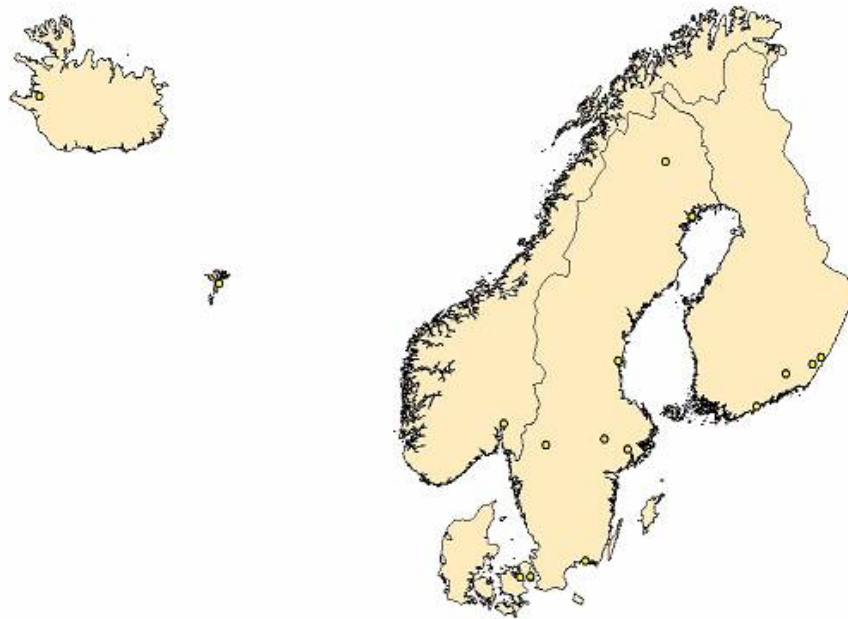
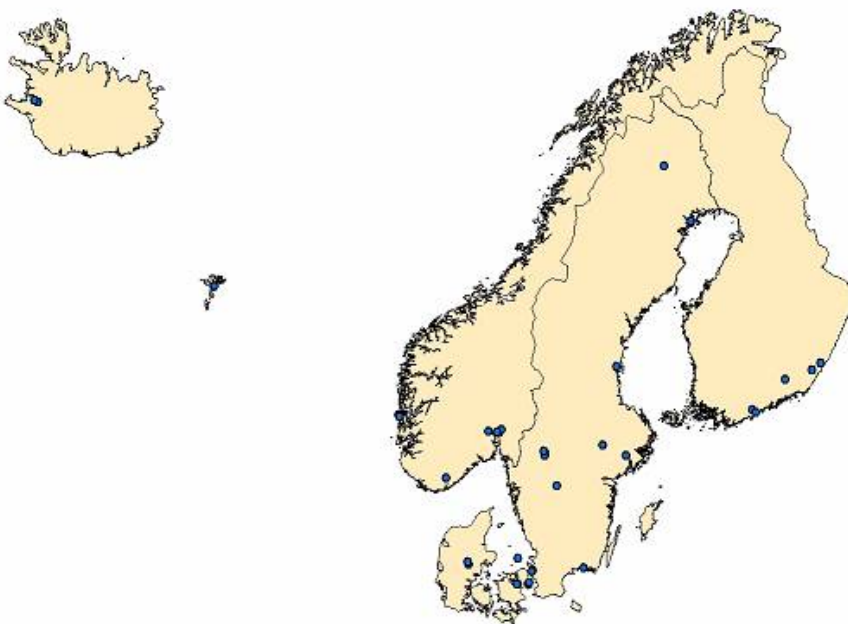


Figure 10. Nordic sampling sites for sediment & soil.



*Figure 11. Nordic sampling sites for sludge.*



*Figure 12. Nordic sampling sites for water.*

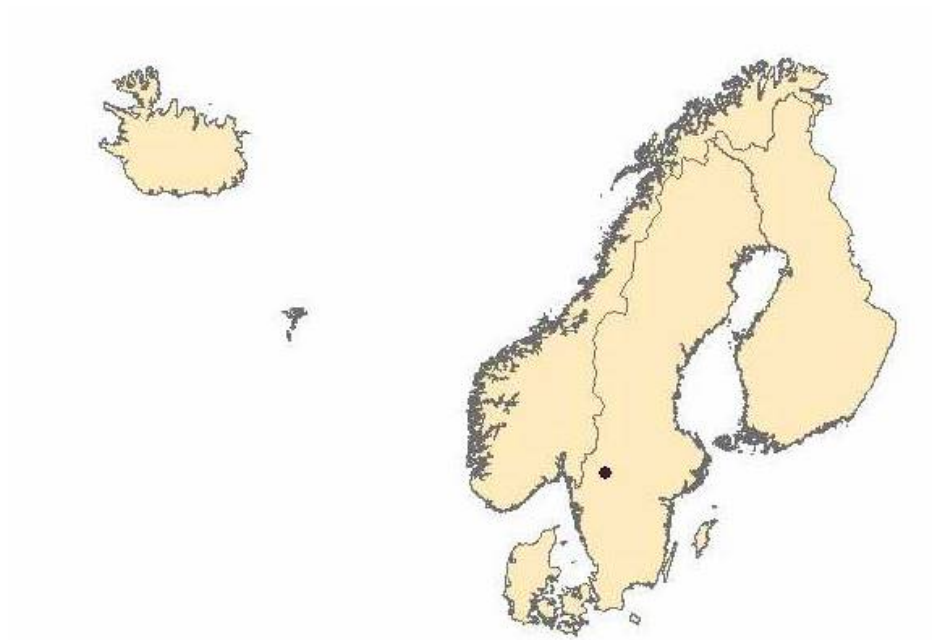


Figure 13. Nordic sampling sites for air.

## 4.2. Sampling

### Water, sediment, sludge and biota samples

As a guideline for adequate and consequent sampling, the laboratories in charge provided a manual (Appendix 2) for the sampling personnel in the Nordic countries participating in the screening. Detailed instructions for sampling, storing and transport were given. Sampling protocols for all sample types were included in the manual. The aim of the sampling protocol was to

- Guide the personnel responsible for sampling on how to avoid contamination of the samples.
- Ensure documentation of the sampling procedure, quality of the sample and environmental and physical circumstances during the sampling.

All samples were sent directly to the analytical laboratories by the national institutions responsible for sampling.

### Air samples

The Swedish air samples were collected using a low volume air sampler (LVS) with a flow of approximately 1 m<sup>3</sup>/h. The air was passed through a glass fiber filter (MG160, Munktell) where particles were collected and

then through a glass column packed with the adsorbents XAD-2 (Amberlite) and polyurethane foam (PUF). Sampling duration was four weeks. Filter and adsorbents were changed every week. Prior to sampling, glass fiber filters were heated to 400°C, and the adsorbent columns were cleaned by Soxhlet extraction with acetone. After sampling, the filter and columns were wrapped in aluminum foil and sent to the laboratory, where they were stored in a freezer (-18°C) until analysis. Additional filters and columns used as field blanks were sent back to the laboratory unexposed.

### 4.3 Chemical analysis of the collected samples

Note: The samples have been analysed at the IVL laboratory and the NILU laboratory. The IVL sample identity coding has the prefix MR- and the NILU sample coding has the prefix Bron-. Correspondingly, the analytical method description below is denoted as the IVL-method and the NILU-method. The samples from Norway, Iceland and the Faroe Islands were analysed with the NILU-method while the samples from Sweden, Denmark and Finland were analysed with the IVL-method.

#### 4.3.1 Analysis of water samples

IVL-method: In order to stabilize the analytes, acid was added prior to thawing the sample. An aliquot of the thawed water sample (200 ml) was spiked with recovery standard (3-fluor ethyl parabene), acidified (pH 2) and fortified with sodium chloride (20 g/l) before concentration on an SPE-column (ENV+; Isolut). The analytes were eluted with ethyl acetate and MTBE. The extract was concentrated by azeotropic evaporation and subsequently derivatised and cleaned up on a silica gel column prior to GC-MS analysis (see below).

NILU-method: The samples (400 ml) were pH-adjusted to 3 using hydrochloric acid (HCl). C13 labelled triclosan and C13 labelled bisphenol-A were added as recovery standards. The samples were enriched by solid phase extraction (HLB, Waters) after a pre-condition step using methanol, acetone and water. The flow rate during extraction was ~5 ml/min. The solid phase column was rinsed with water (10 ml), and then eluted using 5 ml of a methanol-acetone mixture. The extract volume was reduced to 0.5 ml prior to the analysis using high performance liquid chromatography combined with high resolution mass spectrometry (HPLC/HRMS).

#### 4.3.2 Analysis of biota samples

IVL-method: The sample was fortified with recovery standard (3-fluor ethyl parabene) and homogenized in acetonitrile. The extraction was per-



formed by gentle agitation on a shaking board. The sample was centrifuged and the organic extract was saved in a new test tube and diluted with sodium chloride solution and subsequently MTBE-extracted. The combined extract was concentrated and dried in the same manner as the water samples (see above). Subsequently the extract was derivatised and cleaned up on a silica gel column prior GC-MS analysis (see below).

NILU-method acidic extraction: The sample was acidified with hydrochloric acid (HCl) and spiked with C13 labelled triclosan and C13 labelled bisphenol-A as recovery standard. Water was added as extraction solvent and the sample was vigorously shaken. The extraction was performed twice and the combined extracts were further treated as water samples.

#### 4.3.3 Analysis of sludge, sediment and soil samples

IVL-method: The sample was fortified with recovery standard (3-fluor ethyl parabene) and extracted with acetonitrile. The sample was centrifuged and the organic extract was transferred to a test tube and diluted with sodium chloride solution and subsequently MTBE-extracted. The combined extract was concentrated and dried in the same manner as for the water samples (see above) and was thereafter derivatised and cleaned up on a silica gel column prior to GC-MS analysis (see below).

NILU-method acidic extraction: The sample was acidified with hydrochloric acid (HCl) and spiked with C13 labeled triclosan and C13 labeled bisphenol-A as recovery standard. Water was added as extraction solvent and the sample was vigorously shaken. The extraction was performed twice and the combined extracts were further treated as water samples.

NILU-method basic extraction: The sample was spiked with C13 labeled triclosan and C13 labeled bisphenol-A as recovery standard. 0.1 M NaOH was added as extraction solvent and the sample was vigorously shaken. The extraction was performed twice and the combined extracts were acidified and further treated as water samples. The basic extract was used for optimum recovery triclosan analysis.

#### 4.3.4 Analysis of Air

The filters, the XAD/PUF and the glass cartridges used were all subjected to Soxhlet extraction using acetone as solvent for 24 h. The sample extract was concentrated first on a RotoVap and further under a nitrogen gas stream. The extract was dried and derivatized as below.

#### 4.3.5 Derivatisation

The extract, dissolved in hexane/MTBE, was reacted with acetic acid anhydride in the presence of sodium acetate. The reaction was accom-

plished on a heating block at 85°C for two hours. The excess reagents were removed by shaking with di-potassium carbonate. The derivatised extract was dried over sodium sulfate and finally cleaned up on a silica gel column (see below).

#### 4.3.6 Silica gel column

A deactivated silica gel column was prepared in a Pasteur pipette. The derivatised analytes were transferred to the column and eluted by a mobile phase containing hexan: MTBE. The eluate was concentrated and internal standard (biphenyl) was added prior to analysis by GC-MS.

#### 4.3.7 GC/MS quantification of IVL- samples

The extracts were analysed on a 6890 N gas chromatograph coupled to a 5973N mass selective detector (Agilent). The injection, 1 µl, was done in splitless mode at 250°C. The fused silica capillary column (VF-5MS 30 m x 0.25 mm i.d. x 0.25 µm film thickness, Varian) was held at 50°C for 3 min., ramped 12°C/min to 280°C and held at 280°C for 10 min. Helium was used as carrier gas. The detector was used in selected ion monitoring mode (SIM). Resorcinol and 3-methylphenol was analysed in electron ionization mode at energy of 70 eV. Bronopol and triclosan was analysed in negative chemical ionization mode (NCI) with methane as reaction gas. The analytes were identified by their characteristic retention time and one target ion (Tgt-ion, used for quantification) and in most cases one or two qualifier ions (Q-ion) used to increase specificity was recorded. Quantification was based on comparison of peak abundance to the known response of the internal standard (biphenyl). The reported analyte concentrations were corrected according to the determined surrogate standard losses.

**Table 9. Target and qualifier ions**

Substance	Tgt-ion	Q-ion
Diphenyl (EI-mode)	154	-
3-Fluor ethyl parabene (EI-mode)	139	184
Resorcinol (EI-mode)	110	152
m-cresol (EI-mode)	108	107; 150
3-Fluor ethyl parabene (NCI-mode)	183	-
Triclosan (NCI-mode)	251	253
Bronopol (NCI-mode)	79	81

#### 4.3.8 HPLC/HRMS quantification of NILU- samples

Liquid chromatography was performed with an Agilent 1100 liquid chromatography system (Agilent Technologies, Waldbronn, Germany), equipped with an autosampler, a quaternary pump, an on-line degassing

system and a diode array detector (UV). The compound separation was performed with a reversed phase C<sub>18</sub> column (Atlantis dC18, 2.1 mm ID x 150 mm length, 3 µm, Waters, Milford USA). A stainless steel inlet filter (Supelco, 0.8 µm) was used in front of a pre-column with the same stationary phase as the separation columns. Gradient elution was performed with water as solvent A and acetonitrile as solvent B. The binary gradient had a flow rate of 0.2 ml min<sup>-1</sup> and started with 95% A. Solvent B was introduced linearly up to 90% at 10 minutes and kept isocratic until 16 minutes. The column was then washed and equilibrated and ended with a total runtime of 40 minutes. The analytical detector was a Micromass LCT orthogonal-acceleration time-of-flight (TOF) mass spectrometer (MS) equipped with a Z-spray electrospray ion source and a 4 GHz time to digital converter (TDC) (Micromass Ltd., Wythenshawe, Manchester, UK). The instrument was operated in negative mode and the electrospray source parameters were optimised to the following values: sample cone cycling 20/30 V, capillary voltage 2.8 kV, extraction cone 3 V, source temperature 135°C, desolvation temperature 350°C, cone gas flow 20 l h<sup>-1</sup> and desolvation gas flow 600 l h<sup>-1</sup>. The pusher frequency was operated in automatic mode. The data processing and instrument (HPLC/HRMS) control were performed by the MassLynx software, and quantitation was performed with signal extraction of a peak width of 90 amu (typical).

**Table 10. Monitoring ions**

Substance	MW	Monitoring ion	Confirming ion
Bronopol	200	168	170
Resorcinol	110	109	
<i>m</i> -cresol	108	107	
Triclosan	289	287	289

#### 4.4 Quality control and method comparison

Adequate quality control measures and documentation were introduced covering the entire analytical procedure: sampling, storage, transport, sample preparation, analysis and quantification. To assure a correct sampling procedure and reduce the risk of contamination as well as to assure documentation of possible deviations during sampling and transport, a sampling protocol was developed.

All glass equipment, glass fiber filters, sodium sulfate and sodium chloride were cleaned by heating to 400°C for 4 h prior to use. All other equipment was washed with organic solvent before use.

##### 4.4.1 Recoveries

The recoveries of the different analytes were tested in separate experiments. Acceptable recovery of added surrogate standards and analytes

was generally >75%. Analytes were quantified using the internal standard approach: all reported values were recovery corrected. Method blanks and field blanks were run for each sample batch, to assess background interference and contamination.

#### 4.4.2 Limit of detection

The limit of detection (LOD) was defined as 3 times the background noise level for blanks but with consideration of the actual sample noise. For some of the analytes background levels were detected. Background levels were subtracted from measured sample values.

**Table 11a. Limit of Detection (IVL-samples)**

Matrix	Bronopol	Resorcinol	m-cresol	Triclosan
Water	0.1 µg/l	0.0056–0.4µg/l	0.01 µg/l	0.002 µg/l
Sediment	3–20 ng/g dw	1–10 ng/g dw	0.5–1.0 ng/g dw	2–5 ng/g dw
Fish	10 ng/g fw	5 ng/g fw	5 ng/g fw	0.1 ng/g fw
Sludge	5–24 ng/g dw	7–9 ng/g dw	2–7 ng/g dw	4–10 ng/g fw

**Table 11b. Limit of Detection (Nilu-samples)**

Matrix	Bronopol	Resorcinol	m-cresol	Triclosan
Water	0.1 µg/l	0.1 µg/l	0.1 µg/l	0.1 µg/l
Sediment	5 ng/g dw	2 ng/g dw	10 ng/g dw	1 ng/g dw
Fish	5 ng/g fw	1 ng/g fw	10 ng/g fw	1 ng/g fw
Sludge	5 ng/g dw	1 ng/g dw	10 ng/g dw	1 ng/g fw

#### 4.4.3 Identification

The following quality criteria were used to ensure correct identification and quantification of the target compound: (a) the retention time matched those of the standard compounds within 0.1 min, S/N was >3 (b) the ratio of the selected ions (Tgt- and Q-ions) was within  $\pm 20\%$  of expected / theoretical values. The recovery of all internal standards in the analyzed samples was within acceptable limits, 40–90%.

#### 4.4.4 Laboratory and field blanks

A minimum of one laboratory and one field blanks were analysed along with each batch of samples. Bronopol and resorcinol were generally below the limit of detection of the method used in the blank samples.

#### 4.4.5 Bronopol stability test

Bronopol is recognised as an unstable compound (Wang et al. 2002). In laboratory experiments we showed that the instability of bronopol was pronounced under neutral to alkaline pH but was fairly stable (> one

month) under acidic conditions ( $\text{pH} < 6$ ; figure 14). Bronopol was stable for at least 45 days when stored in freezer at neutral pH.

Considering this insight we added acid to the samples subsequent thawing and concentration (4.3.1).

The recovery rate and precision of the analytical method used for bronopol in sediment was investigated in spiking experiments. Sediment samples were spiked with bronopol and resorcinol at different pH. The samples were mixed well and left in the darkness. Samples were collected periodically and centrifuged. The pore water was used for analysis and in some cases the remaining sediment phase was extracted with acetonitrile to recover adsorbed analytes. In parallel experiments pore water, extracted from sediment, was spiked with bronopol and resorcinol at different pH. Samples were analysed periodically to examine the stability.

The following observations from these experiments were made. The stability of bronopol in sediment at natural pH (6) was poor ( $T_{1/2}$  less than one hour; table 12). The stability was clearly better when the pH was lowered to 2 (table 12) but after 24 h no bronopol could be detected (table 12). In contrast to bronopol, resorcinol was recovered quantitatively after 24 hours at pH between 2 and 6 (table 12).

Acetonitrile extraction of sediment clearly showed that the disappearance of bronopol could not be explained by adsorption (table 12).

In pore water extracted from sediment bronopol and resorcinol was quite stable at pH 2–6 (table 13).

It was stated that a method intercomparison between the two laboratories of the project group (NILU and IVL) should be carried out. As bronopol was apparently very unstable in sediment we concluded that spiking experiments with sediment was not possible.

These results also imply that it is not likely to find bronopol in sediment samples.

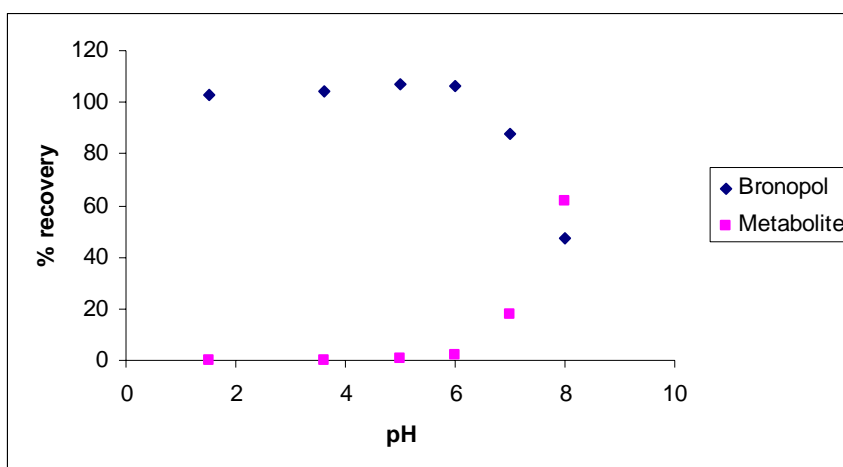


Figure 14: The stability of bronopol at different pH. Bronopol was dissolved in buffer at different pH and left in darkness for 48 hours subsequent analysis.

**Table 12. Stability of bronopol and resorcinol in sediment**

pH	Bronopol 1 h	Resorcinol 1 h	Bronopol 24 h	Resorcinol 24 h	Bronopol ACN 24 h	Resorcinol ACN 24 h
6	9	99	<1	85	<1	78
4	12	102	<1	nd	<1	51
2	60	104	<1	104	<1	20

Explanations: Recovery of bronopol and resorcinol after 1 and 24 hour exposure to sediment (pore water + sediment) at different pH. ACN 24 h: Acetonitrile extract of sediment after 24 hour. nd =not determined.

**Table 13. Stability in pore water (24 h)**

Compound	pH 2	pH 6
Bronopol	100	46
Resorcinol	100	85

Explanations: Recovery of bronopol and resorcinol after 24 hour exposure to pore water at different pH.

#### 4.4.5 Laboratory intercomparison

In table 14 the results from the intercomparison experiments are given. The biota sample is a spiked aliquot of the Bron28, and the sediment sample is a spiked aliquot of the MR4674. The analytical methods show adequate screening performance for both matrices.

**Table 14. Laboratory intercomparison of the chemical analysis**

Institute	Matrix	Resorcinol	Bronopol	m-Cresol	Triclosan
	Biota, theoretical (ng/g fw)	100	100	100	100
IVL	Biota (ng/g fw)	170	42	85	11
NILU*	Biota (ng/g fw)	55	49	53	52
	Sediment, theoretical, (ng/g dw)	No spike	2300	No spike	No spike
IVL*	Sediment (ng/g dw)	3.2	<4.2	2.8	2
NILU	Sediment (ng/g dw)	22	<5	<10	<1

Explanations: Theoretical values are based on spiked amounts of the specific analyte.

\*: Laboratory responsible for the sample spiking.

## 5. Results and discussion

Bronopol was not detected in any of the samples analysed. Limits of detection for the different matrices analysed are given in Table 11. An overview of the results for the other compounds is given in the following chapter. All results are presented in detail in Appendix 3. Name of the countries in figures are abbreviated according to Table 15.

**Table 15. Abbreviations for Nordic countries**

Abbreviation	Country
Dk	Denmark
Fi	Finland
Fo	Faroe Islands
Ic	Iceland
N	Norway
S	Sweden

### 5.1 Concentrations in samples from STPs and landfills

#### 5.1.1 Sewage water from municipal STPs

m-Cresol was frequently found in influent water to municipal STPs (Figure 15). Concentrations ranged between 0.24 µg/l to 3 µg/l (LOD <0.1µg/l). In effluent water m-Cresol occurred in levels between 0.014 µg/l and 0.03 µg/l.

Resorcinol was detected more frequently but in similar concentrations as m-cresol and ranged from 0.075 µg/l to 3.5 µg/l in influent water (LOD<0.0056 µg/l). In effluent water concentrations of resorcinol ranged between 0.007 µg/l and 0.24 µg/l. In the Swedish screening 2006, resorcinol was only detected in one effluent sample (0.078 µg/l) (Remberger et al., 2006). Levels of m-cresol as well as of resorcinol were lower in influent samples from the Faroe Islands and Norway compared with Finland and Denmark (no influent water samples were taken in Iceland and Sweden).

Triclosan was the most frequently found substance in influent water. It occurred however in lower concentrations than the other substances (0.057 µg/l – 0.15 µg/l). Levels in influent water were lower than what was reported in a Norwegian screening from 2005 and lower than what has earlier been reported from Denmark. The data was similar or lower than the results of a Swedish study (Table 8).

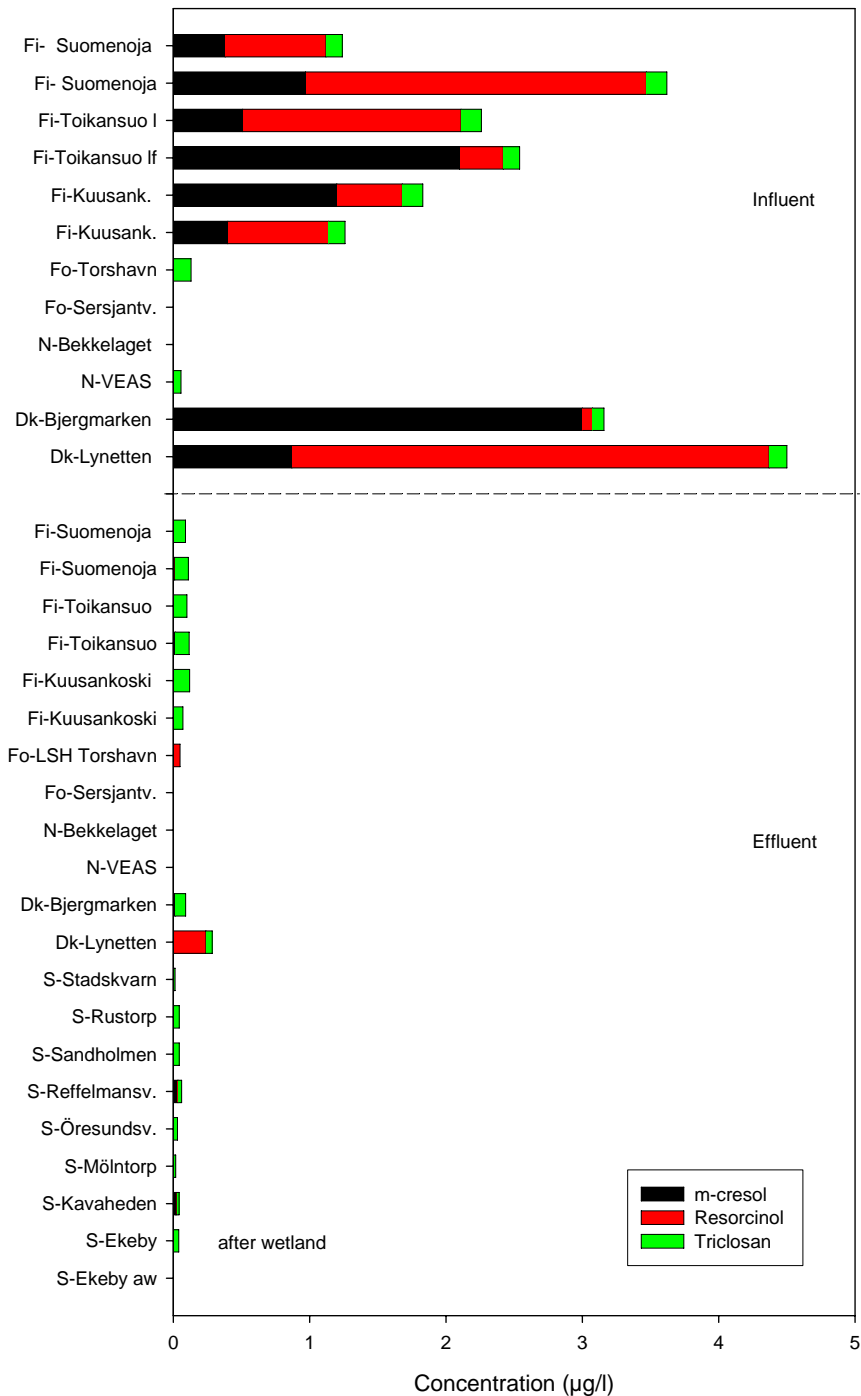


Figure 15. Concentrations in influent and effluent water from municipal STPs

Triclosan was also the most frequently detected substance in the effluent samples. Triclosan was also the substance that occurred in the highest levels in effluent water samples indicating a low removal rate in the STPs. Concentrations in effluent water ranged between 0.0051 µg/l (sample taken after a wetland) and 0.12 µg/l. The sample containing the highest concentration of triclosan in effluent water was collected in Kuusan-



koski, Finland (0.12 µg/l) and was ten times higher than what was detected in Sweden. The Kuusankoski STP sample corresponded with the results of one Swedish study (Samsö-Petersen, table 8) while the other samples with lower concentrations were in the same order of magnitude as the samples from the Swedish screening 2005 and other studies from Sweden and Denmark (Table 8). Effluent water samples from STPs from Norway and the Faroe Islands had the lowest detected levels also of triclosan.

5.1.2 Industrial STPs

m-Cresol and resorcinol were found in sewage water from pulp and paper factories (Figure 16). Concentrations in effluent water, ranging from 0.015 µg/l to 1.2 µg/l for m-cresol and 0.008 µg/l to 0.071 µg/l for resorcinol (Imatra, biological and chemical line respectively), were however in the same range or lower than for effluent water from municipal STPs. Resorcinol was also detected in influent (0.085 µg/l) but not in effluent sewage water from a closed glue production process with a treatment plant for phenolic substances.

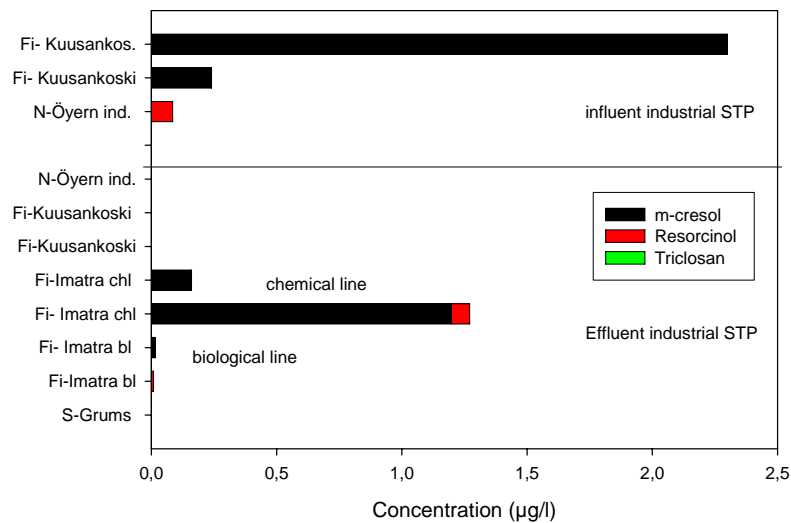


Figure 16. Concentrations in influent and effluent sewage water from industrial STPs

5.1.2 Sludge

m-Cresol was detected in 14 out of 19 municipal STP sludge samples (Figure 17) ranging between 4.5 ng/g dw and 110 ng/g dw (LOD < 2.3 ng/g dw – < 10 ng/g dw). The highest concentration was found in a sludge sample from Sersjantvikin on the Faroe Islands. Only one industrial sewage treatment plant from a pulp and paper factory in Finland contained higher concentration (110 ng/g dw) than the municipal STPs.

Resorcinol occurred in 15 out of the 19 analysed municipal STP sludge samples and in 2 of the 5 industrial STP sludge samples (Figure 17). Concentrations in municipal STP sludge ranged between 9 ng/g dw and 220 ng/g dw and in industrial STP sludge between 24 ng/g dw and 48 ng/g dw. Limit of detection were between <5.7 ng/g dw and < 8.7 ng/g dw. Levels in sludge were in the same range as in the Swedish screening (22ng/g to 380 ng/g) but the detection frequency was lower (Remberger et al., 2006).

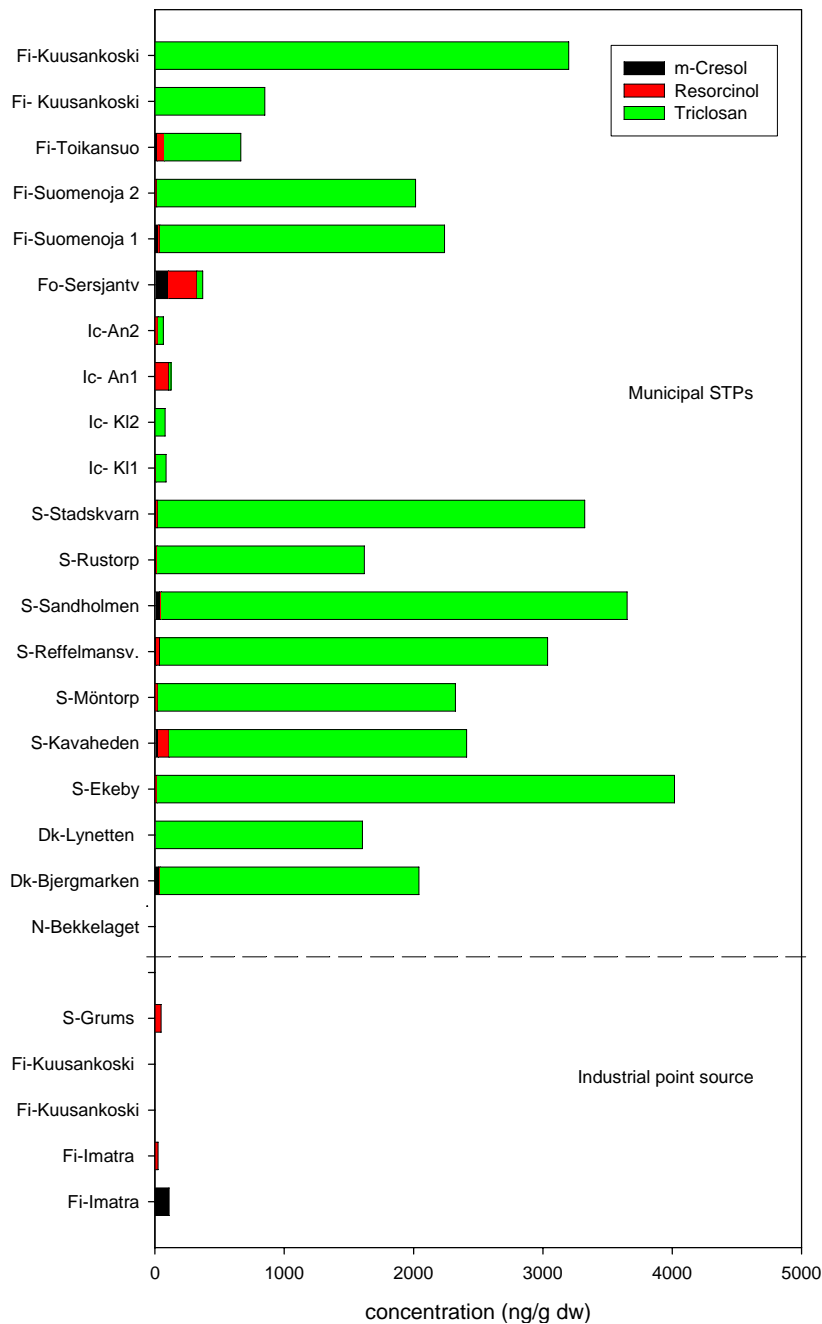


Figure 17. Concentrations in sludge from municipal and Industrial STPs

Triclosan was found in the highest concentration among the analysed substances in sludge (Figure 17). It occurred in all municipal STPs sludge samples. Concentrations in sludge ranged between 17 ng/g dw and 4000 ng/g dw which is lower or in the same level as for sludge samples from screening studies performed in Sweden 2006 and 2002 (Table 8). Levels of triclosan in sludge were generally higher in Finland, Sweden and Denmark compared to Iceland and the Faroe Islands. Triclosan was not detected in any of the industrial STP sludge samples.

5.1.3 Leachate water

m-Cresol was found in 7 out of 11 analysed leachate water samples in concentrations ranging from 0.043 µg/l (Ämmässuo, Finland) to 77 µg/l (Helsingborg, Sweden). Triclosan was detected in concentrations ranging from 0.0036 in Toikansuo in Finland to 0.046 at Torshavn, Faroe Islands. Resorcinol did not occur in any of the leachate water samples. Limits of detection were < 0.014 µg/l for m-Cresol, <0.0056µg/l to <0.8 µg/l for resorcinol and <0.0021µg/l to <0.07 µg/l for triclosan. Landfill leachate samples were collected at the landfill site and the water is in general further treated before it reaches the environment.

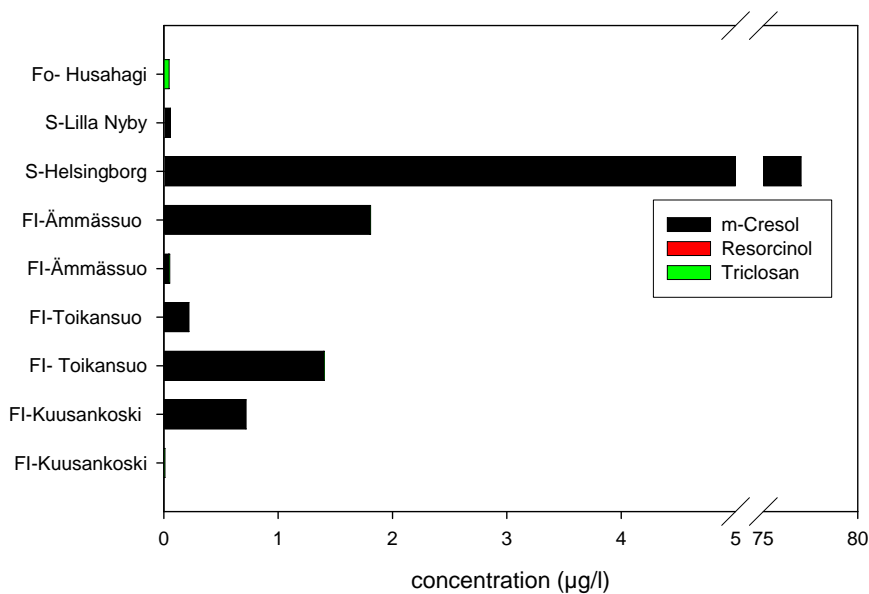


Figure 18. Concentrations in leachate water from landfills

## 5.2 Environmental concentrations

### 5.2.1 Air

m-cresol was detected in air samples collected at the Grums pulp industry in concentrations ranging between 3.1–5.2 ng/m<sup>3</sup>. All other substances were below limit of detection. Concentrations of m-cresol in air samples were in the same range as what has earlier been measured in cities in Sweden (Johanson et al, 2004b). The contribution of the factory is thus disputable.

### 5.2.2 Surface water

In surface water m-cresol was only found outside a pulp and paper factory in Finland in a concentration of 0.022 µg/l. Resorcinol, on the other hand, was detected at several different locations. In Iceland it was found outside recipients for landfill runoff in concentrations between 0.008–0.055 µg/l and in a small harbour of yachts and motorboats in a concentration of 0.069 µg/l. In Norway it was found outside point sources and on the Faroe Islands in concentration ranging from 0.018 µg/l to 0.35 µg/l. The site at the Faroe Islands is one of earlier described marked contamination.

Triclosan was only found in surface water in the Roskilde fjord, a recipient for STP effluent water (0.0027 µg/l). This value is lower than what has previously been reported from the US but in the same range of what has been reported from Switzerland and what was reported in the Swedish screening study from 2002 (table 8).

### 5.2.3 Sediment and soil

m-Cresol was detected in sediment samples from a STP recipient in Denmark (0.41 ng/g dw). It was also found outside a pulp and paper factory in Sweden (1.4 ng/g dw) and in two lake sediment samples from Denmark (2.8 ng/g dw and 5.2 ng/g dw respectively; Figure 19). The only analysed soil sample from the Faroe Islands contained m-cresol in a concentration of 96 ng/g dw. All other substances were below limit of detection in this sample.

Resorcinol occurred in sediment samples outside two point sources in Norway (of 3 ng/g dw and 17 ng/g dw respectively), and at one lake in Denmark (3.2 ng/g dw).

Triclosan was detected outside four municipal sewage treatment plants in sediment samples in concentrations ranging between 0.6 ng/g dw outside Lynetten, Denmark to 180 ng/g dw outside VEAS, Norway. Triclosan was also found in Sweden outside a pulp and paper factory and in Denmark both in concentrations of 2 ng/g dw. The level of Triclosan in

the VEAS sample exceeds what has previously been reported in Swedish and Norwegian studies while the other samples are in the same range as previous studies (Table 8).

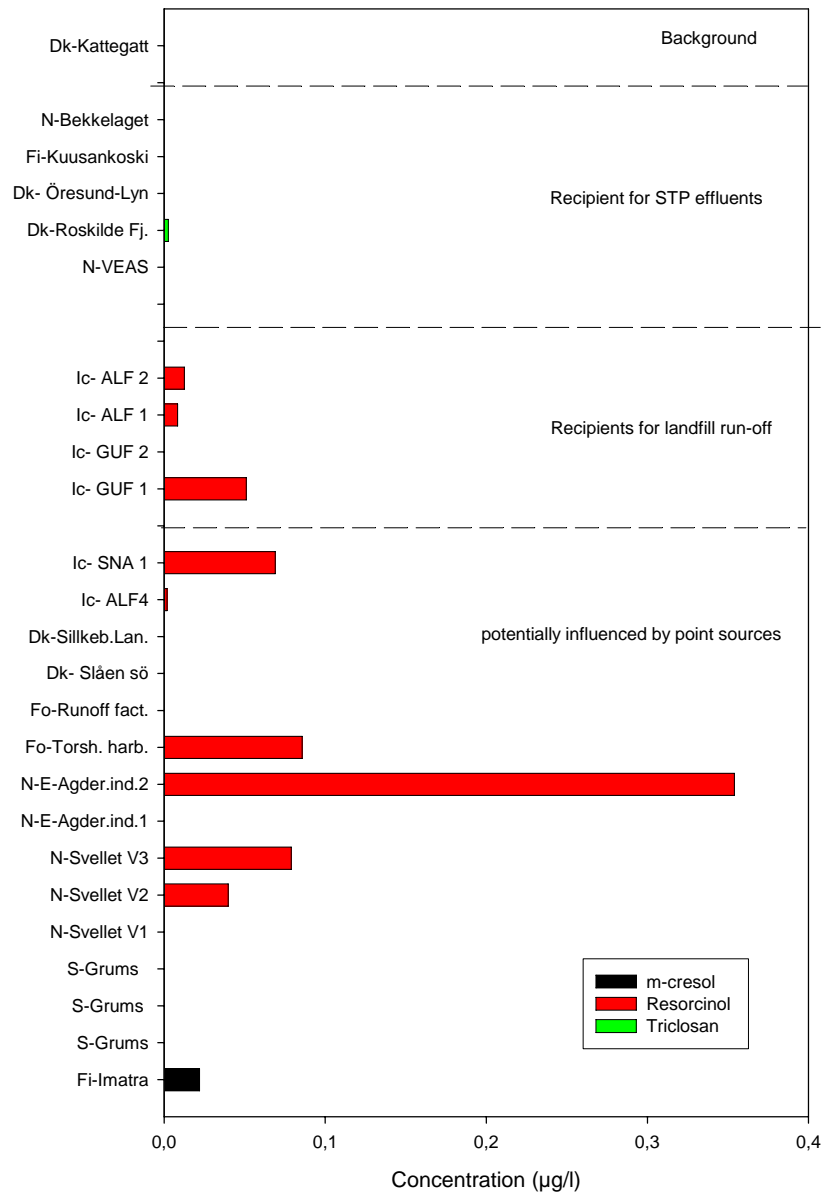


Figure 19. Concentrations in surface water (both fresh water and sea water)

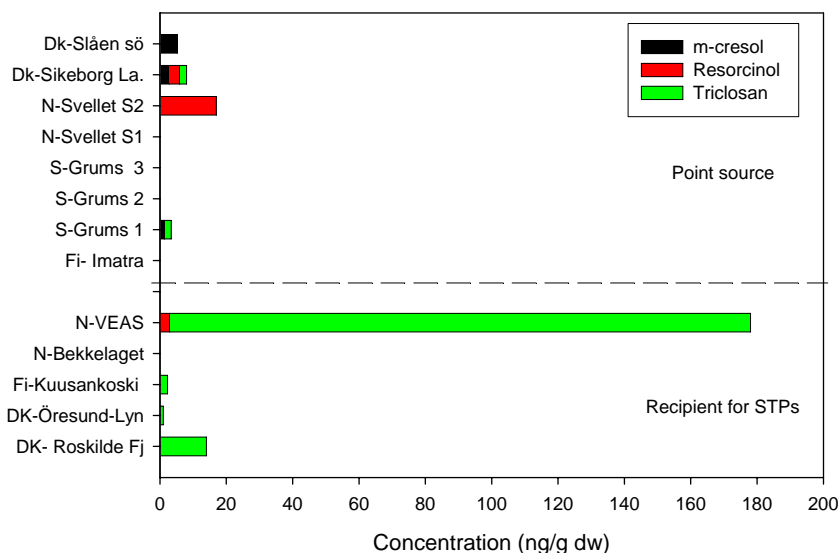


Figure 20. Concentrations in sediment

#### 5.2.4 Biota

Triclosan was detected in one fish sample (liver) from Roskilde fjord (0.13 ng/g ww) and in fish samples (liver and muscle) from Öresund Lynetten in (0.29 ng/g ww and 0.15 ng/g ww respectively). Both sites are recipients of municipal STP effluents. Levels were in the lower range of what has earlier been reported from Sweden and Norwegian studies (table 8). All other substances in biota samples were below the detection limit.

### 5.3 Concentration patterns at geographically related sites

Resorcinol and m-cresol were the only substances detected in sewage sludge and water from industrial STPs.

Resorcinol also occurred in surface water samples and sediment samples adjacent to two industrial activities in Norway, one near Svellet and one in East Agder. At the industrial site "Svellet" the concentrations were however lower in both water and sediment samples closer to the factory (V1, S1) than at the reference points (V2, V3, S2). m-Cresol was detected in water but not in sediment samples outside a pulp and paper factory in Imatra, Finland. m-Cresol also occurred in two sediment samples at two different sites in Denmark, lake Slåen and lake Silkeborg. Resorcinol and triclosan were found in the Silkeborg sediment sample. The origin of the compounds in these samples is still rather elusive as there is no known use of the compounds in the vicinity of the sampling sites.

Outside the pulp and paper factory in Sweden, triclosan and m-cresol were detected in a sediment sample but not in any of the water samples.

Triclosan was the dominating substance in the sludge samples from municipal STPs. Levels were higher in sludge from STPs in Denmark, Finland and Sweden than from STPs in Iceland and at the Faroe Islands. In the effluent samples triclosan and resorcinol were detected rather frequently while only recipient water samples from two countries (resorcinol in Iceland and triclosan in Denmark) contained concentration above LOD of any of the substances. In four of the STP recipient sediment samples concentrations were above LOD of triclosan. Outside VEAS STP in Norway the concentration was nearly an order of magnitude higher than the second highest concentration in sediment samples from Roskilde Fjord in Denmark.

m-Cresol was detected in several leachate water samples.

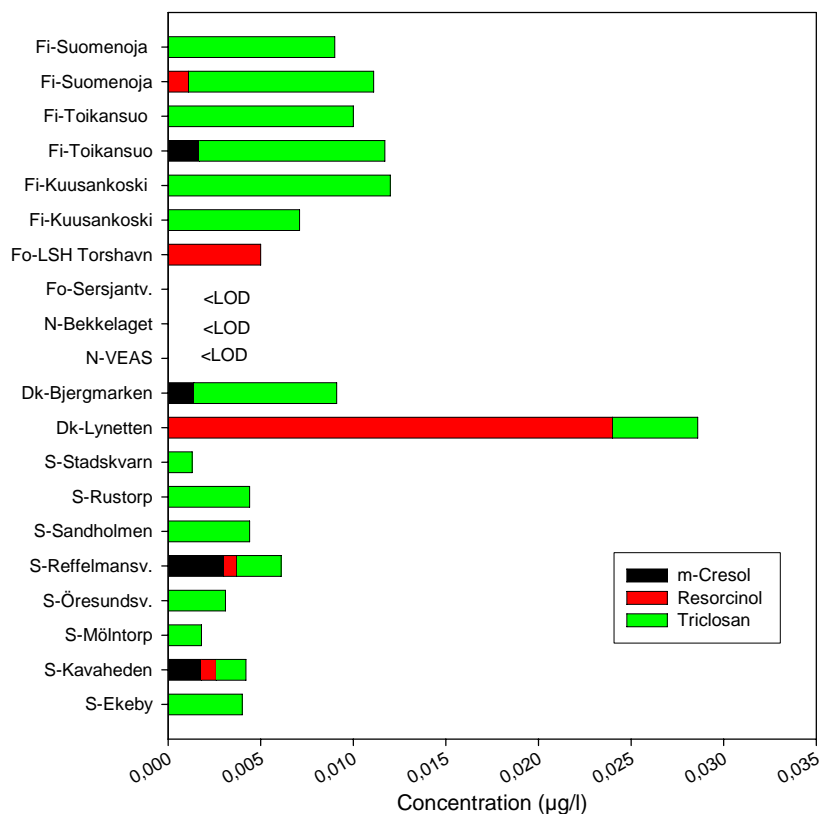


Figure 21. Concentration in effluent water (diluted with a factor of 10)

### 5.4 Ecotoxicological risk assessment

A way to relate the measured concentrations to known environmental effects is to perform a risk assessment and derive risk characterization quotients based on the measured concentrations (MEC) and the predicted

no effect concentrations (PNEC). When the  $MEC/PNEC \leq 1$  no negative effects of the substance are expected but when  $MEC/PNEC \geq 1$  the substance is considered to be problematic in the environment and further investigations are needed (TGD, 2003). PNECs for the risk assessment are according to Table 6.

In order to use the effluent data from the STPs where no recipient water samples were provided a dilution factor of 10 was applied to all effluent concentrations (TGD, 2003). The same dilution factor was applied to leachate water, however many leachate water effluents are directed to STPs. Since the PNEC calculation is associated with certain uncertainties depending on availability and quality of data, differences in applied safety factors and the factum that the screening results are not statistically reliable but rather “a snap shot” of the situation, the results should be interpreted with care and regarded as indications to where there is a need for further investigations. Within this preliminary risk assessment, the MEC/PNEC ratios for all substances were  $<1$  except for one surface water sample from Norway (sample ID= bron 16; Table 16). The risk quota for this sample when using the EQS set by the Finnish Environment Institute was 1.4. However, when using the PNEC based on the long term study on *Daphnia* found within this study the risk quota is again below one.

Regarding uptake in biota this was only observed for triclosan. This is in agreement with the Log Kow values stated in Table 1 where only Triclosan had a Log Kow above 3 and hence an indicated bioaccumulation potential.

**Table 16. Range of PEC/PNEC-quotients for water samples**

	m-Cresol	Resorcinol	Triclosan
Effluent water STP	0.0002–0.02	0.0004–0.013	0.00005–0.0012
Landfill leachates	0.0007–0.03	<LOD	0.00004–0.0005
Surface water	0.004	0.001–0.2 (1.4*)	0.00004

\* using finish EQS



## 6. Summary and conclusions

Despite a high consumption of *bronopol* in the Nordic countries as well as within the EU, it was not found in any of the samples analyzed. Bronopol undergoes rapid hydrolysis as well as biodegradation, which may explain its absence in the environmental matrix samples.

*Resorcinol* is widely used and was also frequently found in the municipal influent water samples. Despite that it is significantly reduced in the STPs and has a high reported biodegradability, it is still present in some effluent water samples and more importantly it can be detected in environmental samples of water and sediment. Levels of resorcinol were higher than concentrations of triclosan in environmental samples collected outside point sources but lower or in the same range as triclosan in environmental samples collected outside STPs. In contrast to triclosan, resorcinol seems to be more present in water samples than in sediment samples.

*Triclosan* was found in high concentrations in STP sludge. This may be a reflection of the use of triclosan in health care products. Triclosan was also frequently detected in STP effluent water and in sediment outside STPs. The present study shows that, despite a stated decreased usage of the compound within the industry, triclosan is still detected in and around STPs and in some environmental samples.

The registered usage volume of *m-cresol* is only available for Sweden, however, the compound was found in several influent and effluent samples from municipal STPs samples in Denmark, Finland and Sweden and in leachate water samples in Finland and Sweden. In effluent water and in environmental samples it was however mainly found in the vicinity of an industrial pulp and paper STP in Finland. Earlier studies have shown the importance of emissions from biomass burning for this compound and this along with the diffuse spreading of the substance from products not registered in SPIN seem to be of importance for concentrations in municipal and industrial STPs. A high removal efficiency in the STPs of the substance seems however to limit its environmental distribution.

- Despite a high usage, bronopol could not be detected in any of the samples.
- Resorcinol was found in environmental samples of water and sediment adjacent to STPs, industrial activities and at sites where the origin is more elusive.
- Resorcinol occurred more frequently and in higher concentrations than triclosan in surface water samples.

- Triclosan was found more frequently and in higher concentrations in STP effluent water samples and sediment samples compared with resorcinol.
- m-Cresol was frequently detected in sewage water. It was, however, only found in one environmental sample indicating efficient degradation in STPs.
- Chemical release via dispersed use related to many different activities and products seems to be the most important emission pathway for all the substances included in this screening.
- According to the risk assessment (MEC/PNEC) only one concentration, found in Norway, showed elevated risks.
- Triclosan was the only substance found in biota.

## 7. Acknowledgements

### *Norway:*

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- HSDB (2006) Hazard Substance Data Bank. US National Library of Medicine <http://toxnet.nlm.nih.gov/cgi-bin/sis/htmlgen?HSDB> (2006-03-01 till 2006-)
- SPIN (2006) Substances in Preparations in Nordic Countries <http://www.spin2000.net/spin.html> (2006-02-01-2006-08-30)

SRC PhysProp Database (2006)

<http://esc.syrres.com/interkow/webpro.exe>

US EPA (2006) <http://www.epa.gov/ecotox/2006-01-15-2006-05-30>





# Appendix 1; Sample characteristics

Country	LAB-ID	Sample blank	Sampling site	Site/sample information	Matrix	Sample information	Sampling date	coordinates	coordinates
Finland	4512		Kuusankoski	Chemical industry and Kuusankoski STP	water	Leachate	2005-11-02	60.926	26.628
Finland	4513		Kuusankoski	Chemical industry and Kuusankoski STP	water	Influent	2005-11-02	60.926	26.628
Finland	4514	4515	Kuusankoski	Kuusankoski STP	water	Effluent	2005-11-02	60.926	26.628
Finland	4516		Kuusankoski	Kuusankoski STP	sludge		2005-11-02	60.926	26.628
Finland	4517		Kuusankoski	River Kymijoki	water	Surface	2005-11-02	60.926	26.628
Finland	4522		Kuusankoski	River Kymijoki	sediment	Sediment	2005-11-09	60.926	26.628
Finland	4523		Kuusankoski	Chemical industry and Kuusankoski STP	water	Leachate	2005-11-14	60.926	26.628
Finland	4524		Kuusankoski	Chemical industry and Kuusankoski STP	water	Influent	2005-11-14	60.926	26.628
Finland	4525		Kuusankoski	Kuusankoski STP	water	Effluent	2005-11-14	60.926	26.628
Finland	4526		Kuusankoski	Kuusankoski STP	sludge		2005-11-14	60.926	26.628
Finland	4527		Kuusankoski	pulp/paper industry STP	water	Influent	2005-11-02	60.926	26.628
Finland	4528		Kuusankoski	pulp/paper industry STP	water	Effluent	2005-11-02	60.926	26.628
Finland	4529		Kuusankoski	pulp/paper industry STP	sludge		2005-11-02	60.926	26.628
Finland	4534		Kuusankoski	pulp/paper industry STP	water	Influent	2005-11-14	60.926	26.628
Finland	4535		Kuusankoski	pulp/paper industry STP	water	Effluent	2005-11-14	60.926	26.628
Finland	4536	4537	Kuusankoski	pulp/paper industry STP	sludge		2005-11-14	60.926	26.628
Finland	4539		Imatra	pulp/paper industry STP	water	Effluent / biological line	2005-11-03	61.192	28.756
Finland	4540		Imatra	pulp/paper industry STP	water	Effluent / chemical line	2005-11-02	61.192	28.756
Finland	4541	4542	Imatra	pulp/paper industry STP	sludge		2005-11-02	61.192	28.756
Finland	4546	4547	Imatra	pulp/paper industry STP	water	Effluent / biological line	2005-11-14	61.192	28.756
Finland	4548		Imatra	pulp/paper industry STP	water	Effluent / chemical line	2005-11-14	61.192	28.756
Finland	4549		Imatra	pulp/paper industry STP	sludge		2005-11-14	61.192	28.756

Country	LAB-ID	Sample blank	Sampling site	Site/sample information	Matrix	Sample information	Sampling date	coordinates	coordinates
Finland	4550		Imatra	Lake Saimaa	water	Surface	2005-11-14	61.192	28.756
Finland	4551		Imatra	Lake Saimaa	sediment	Sediment	2005-11-14	61.192	28.756
Finland	4552		Lappeenranta	Toikansuo waste deposit	water	Leachate	2005-11-03	61.038	28.187
Finland	4553		Lappeenranta	Toikansuo STP	water	Influent	2005-11-03	61.038	28.187
Finland	4554		Lappeenranta	Toikansuo STP	water	Efluent	2005-11-03	61.038	28.187
Finland	4555		Lappeenranta	Toikansuo waste deposit	water	Leachate	2005-11-15	61.038	28.187
Finland	4556		Lappeenranta	Toikansuo STP	water	Influent	2005-11-15	61.038	28.187
Finland	4557		Lappeenranta	Toikansuo STP	water	Efluent	2005-11-15	61.038	28.187
Finland	4558		Lappeenranta	Toikansuo STP	sludge		2005-11-15	61.038	28.187
Finland	4559		Espoo	Ämmässuo waste deposit	water	Leachate	2005-11-07	60.236	24.538
Finland	4560	4561	Espoo	Suomenoja STP	water	Influent water	2005-11-07	60.157	24.717
Finland	4562		Espoo	Suomenoja STP	water	Efluent water	2005-11-07	60.157	24.717
Finland	4563		Espoo	Suomenoja STP	sludge		2005-11-07	60.157	24.717
Finland	4564		Espoo	Ämmässuo waste deposit	water	Leachate	2005-11-21	60.236	24.538
Finland	4565		Espoo	Suomenoja STP	water	Influent water	2005-11-21	60.157	24.717
Finland	4566		Espoo	Suomenoja STP	water	Efluent water	2005-11-21	60.157	24.717
Finland	4567	4569	Espoo	Suomenoja STP	sludge		2005-11-21	60.157	24.717
Finland	4568		Kotka	River Kymijoki, Huruksela	Biota	Roach, 7 individuals	2005-12-03	60.600	26.760
Sweden	4292		Helsingborg	Helsingborg landfill	water	leachate	2005-10-25		
Sweden	4170		Eskilstuna	Lilla Nyby landfill	water	leachate before cleaning	2005-10-05		
Sweden	4168	4169	Eskilstuna	Ekeby STP	sludge		2005-10-04	6571624	1597900
Sweden	4165	4167	Eskilstuna	Ekeby STP	water	effluent	2005-10-04	6571624	1597900

Country	LAB-ID	Sample blank	Sampling site	Site/sample information	Matrix	Sample information	Sampling date	coordinates	coordinates
Sweden	4166		Eskilstuna	Ekeby STP	water	effluent after wetland	2005-10-04	6571624	1597900
Sweden	4234		Gällivare	Kavaheden STP, Person equivalent: 18500, treatment process :mech/chem/biol.	sludge		2005-10-12	7287700;	1795760
Sweden	4233		Gällivare	Kavaheden STP, Person equivalent: 18500, treatment process :mech/chem/biol.	water	effluent	2005-10-12	7287700;	1795760
Sweden	4208		Hallstahammar	Mölnortorp STP, Person equivalent: 11241, treatment process :chem/biol.	sludge		2005-10-12	6602672	1525476
Sweden	4207		Hallstahammar	Mölnortorp STP, Person equivalent: 11241, treatment process :chem/biol.	water	effluent	2005-10-12	6602672	1525476
Sweden	4288	4289	Helsingborg	Öresundsverket	water	effluent	2005-10-25	6216870	1308060
Sweden	4303		Hudiksvall	Reffelmansverket	sludge		2005-10-26	6844618	1568169
Sweden	4302		Hudiksvall	Reffelmansverket	water	effluent	2005-10-26	6844618	1568169
Sweden	4273	4274	Piteå	Sandholmen STP, Person equivalent: 30500, treatment process :mech/chem/biol.	sludge		2005-10-19	7458000	1714000
Sweden	4271	4272	Piteå	Sandholmen STP, Person equivalent: 30500, treatment process :mech/chem/biol.	water	effluent	2005-10-19	7458000	1714000
Sweden	4195		Ronneby	Rustorp STP	water	effluent	2005-10-11	6228468	1468366
Sweden	4196		Ronneby	Rustorp STP	sludge		2005-10-11	6228468	1468366
Sweden	4215		Skövde	Stadskvarn STP	water	effluent	2005-10-12/13		
Sweden			Grums	pulp industry	air		2006-03-23 – 2006-04-20	6583990	1346570

Country	LAB-ID	Sample blank	Sampling site	Site/sample information	Matrix	Sample information	Sampling date	coordinates	coordinates
Sweden			Grums	pulp industry	air		2006-04-20 – 2006-05-18	6583990	1346570
Sweden			Grums	pulp industry	air		2006-05-18 – 2006-06-14	6583990	1346570
Sweden	4770		Grums	pulp industry Ås Stn 1	water		2006-05-08	6581849	1348252
Sweden	4772		Grums	pulp industry Ås Stn 2	water		2006-05-09	6579050	1349650
Sweden	4774	4777	Grums	pulp industry Ås Stn 3	water		2006-05-08	6571600	1349700
Sweden	4784		Grums	pulp industry	sludge	aerated lagoon		6583990	1346570
Sweden	4785		Grums	pulp industry	water	aerated lagoon		6583990	1346570
Sweden	4771		Grums	pulp industry Ås Stn 1	sediment		2006-05-08	6581849	1348252
Sweden	4773		Grums	pulp industry Ås Stn 2	sediment		2006-05-09	6579050	1349650
Sweden	4775	4776	Grums	pulp industry Ås Stn 3	sediment		2006-05-08	6571600	1349700
Sweden	4722		Grums	pulp industry Fish, Åsfjorden	biota	perch, male	2005, summer	658065	134834
Sweden	4723		Grums	pulp industry Fish, Sätterholmsfjärden (ref .station)	biota	perch,male	2005, summer	658065	134834
Denmark	4655		Roskilde	Bjergmarken STP	water	influent	060208-060204	55° 39 N	12° 03 E
Denmark	4656		Roskilde	Bjergmarken STP	water	effluent	060201-060208	55° 39 N	12° 03 E
Denmark	4657	4661	Roskilde	Bjergmarken STP	sludge		2006-02-08	55° 39 N	12° 03 E
Denmark	4658	4659	Roskilde Fjord	A, B	water	Coast water	2005-11-23	55° 39.55 N	12° 03.78 E
Denmark	4660		Roskilde Fjord		sediment		2005-11-23	55° 39.55 N	12° 03.78 E
Denmark	4662		Roskilde Fjord		biota	Fish, Eelpout, liver	2005-11-09	55° 39.55 N	12° 03.78 E
Denmark	4663		Roskilde Fjord		biota	Fish,Eelpout, muscle	2005-11-09	55° 39.55 N	12° 03.78 E
Denmark	4664	4665	Kattegatt	A, B (St 418)	water	Coast water	2006-02-06	56° 22.38 N	12° 02.15 E

Country	LAB-ID	Sample blank	Sampling site	Site/sample information	Matrix	Sample information	Sampling date	coordinates	coordinates
Denmark	4666		Lynetten STP	5combined samples	water	influent	051128-051202	55° 41.76 N	12° 37.00 E
Denmark	4667		Lynetten STP	5combined samples	water	effluent	051128-051202	55° 41.76 N	12° 37.00 E
Denmark	4668		Lynetten STP		sludge			55° 41.76 N	12° 37.00 E
Denmark	4669		Lynetten	Öresund	water	Coast water	2005-10-17	55° 43.8 N	12° 37.7 E
Denmark	4670	4671	Lynetten	Öresund	sediment		2005-10-17	55° 43.8 N	12° 37.7 E
Denmark	4672		Lynetten	Öresund	biota	Fish, flounder, liver	2005-11-21	55° 43.8 N	12° 37.7 E
Denmark	4673		Lynetten	Öresund	biota	Fish, flounder, filet	2005-11-21	55° 43.8 N	12° 37.7 E
Denmark	4674		Sikeborg Langsö,	lake (St. 90901) Downstream sewage treatment plant	sediment		2005-02-16	56° 10.31 N	9° 35.40 E
Denmark	4675		Slåen sø	lake (St. 90918)	sediment		2005-05-13	56° 07.408 N	9° 37.00 E
Denmark	4803	4804	Slåen sø	Lake (St. 90918)	water	surface water	2006-04-20	56° 07.408 N	9° 37.00 E
Denmark	4805	4806	Silkeborg Langsö	lake (ST. 90901) Downstream sewage treatment plant	water	surface water	2006-04-20	56° 10.31 N	9° 35.40 E
Norway	Bron 01		VEAS		water	influent			
Norway	Bron 03		VEAS		water	effluent			
Norway	Bron 05		VEAS		sediment	Sediment+TOC			
Norway	Bron 06		Bekkelaget		water	effluent			
Norway	Bron 07		Bekkelaget		water	influent			
Norway	Bron 08		Bekkelaget		sludge				

Country	LAB-ID	Sample blank	Sampling site	Site/sample information	Matrix	Sample information	Sampling date	coordinates	coordinates
Norway	Bron 09	x	Bekkelaget	0-2cm , taken at 51m water depth, 80m west of the STPdiffusor in "Bekkelagsbassenget"	sediment	sea	2005-10-17	N59°52.969 5	E10045.418
Norway	Bron 10	x	Bekkelaget	Taken at 30m water depth 80m west of the STP diffusor in "Bekkelagsbassenget"	water	Sea water	2005-10-17	N59°52.969 2	E10045.418
Norway	Bron 11		VEAS	Taken at 30m depth 80m north of VEAS diffusor	water	Sea water	2005-10-17	N59°47.645	E10030.949
Norway	Bron 15	x	E-Agder industrial site 1		water	Surface water	2005-11-01	32VO44525 4	UTM64755 43
Norway	Bron 16	x	E-Agder industrial site 2		water	Surface water	2005-11-01	32VO44526 2	UTM64755 23
Norway	Bron 19		Stasjon 30b i JAMP-prg.	Trawling at 80 - 90m depth	biota	fish Cod (5 stk.)	2005-10-06	N59°49'	E10033'
Norway	Bron 20	x	Svellet V1	Nitelva down stream factory area	water	Surface water	2005-11-07	UTM28040 1	6651166
Norway	Bron 21	x	Svellet V2	Central of Svellet	water	Surface water	2005-11-07	UTM28260 4	6649365
Norway	Bron 22	x	Svellet V3	Outlet Svellet/Nitelva towards Storråka/Øyeren	water	Surface water	2005-11-07	UTM28243 2	6647066
Norway	Bron 24	x	Svellet S1	Eastern part of Svellet	sediment	Sediment	2005-11-07	UTM28300 7	6650036
Norway	Bron 25	x	Svellet S2	Nitelva (bay) down stream factory area	sediment	Sediment	2005-11-07	UTM28040 1	6651473
Norway	Bron 26		Svellet		sediment	Feltblank Sediment	2005-11-07		
Norway	Bron 27		Svellet	Nitelva (bay) down stream factory area	biota	Fish (Mort/Rutilus-rutilus)	2005-11-07	UTM28040 1	6651454

Country	LAB-ID	Sample blank	Sampling site	Site/sample information	Matrix	Sample information	Sampling date	coordinates	coordinates
Norway	Bron 28		Svellet	Nitelva (bay) down stream factory area	biota	Fish (Pike/Esox lucius)	2005-11-07	UTM280401	6651454
Norway	Bron 29		Svellet	Nitelva (bay) down stream factory area	biota	Fish (Aspius aspius)	2005-11-07	UTM280401	6651454
Norway	Bron 30	x	Öyern Industrial	From a tap	water	influent	2006-01-06	N 615232	Ø6646905
Norway	Bron 31		Öyern Industrial	From collection tap	water	effluent	2006-01-06	N 615232	Ø6646905
Iceland	Bron 36		Island KI1	Klettagarðar, sewage treating plant in Reykjavik	sludge	Non processed, non dehydrated	2006-01-09	64° 9'19.84"N	21°52'23.17 "W
Iceland	Bron 37		Island KI2	Klettagarðar, sewage treating plant in Reykjavik	sludge	Non processed, non dehydrated	2006-01-09	64° 9'19.84"N	21°52'23.17 "W
Iceland	Bron 38		Island An1	Ánanaust, sewage treating plant in Reykjavik	sludge	Non processed, non dehydrated	2006-01-09	64° 9'10.84"N	21°57'20.18 "W
Iceland	Bron 39		Island An2	Ánanaust, sewage treating plant in Reykjavik	sludge	Non processed, non dehydrated	2006-01-09	64° 9'10.84"N	21°57'20.18 "W
Iceland	Bron 43		Island Alf4	Coast near Álfnes, Reykjavík's waste landfill site	water	Coastal recipient water	2006-01-09	64°11'10.28 "N	21°45'30.67 "W
Faroe Islands	Bron 45		Husahagi	Runoff from waste deposit Sample 1 taken in well	water	Leachate	2005-12-28	62°01,712' N,	006048,306 'V
Faroe Islands	Bron 46		Sersjantsvikin	Effluent from the sewage treatment plant taken in the sampling well	water	effluent	2005-12-28	62°00,495' N,	006045,712 'V
Faroe Islands	Bron 47		Torshavn	Seawater from Torshavn harbour in "Vágsbotn"	water	Surface water	2005-12-28	62°00,507' N,	006046,316 'V
Faroe Islands	Bron 48		Sersjantsvikin	Sample taken in the open pipe near the grating.	water	influent	2005-12-28	62°00,495' N,	006045,712 'V
Faroe Islands	Bron 49			Influent to sewage treatment plant at hospital in Torshavn, Faroe Island sample 5	water	influent	2006-01-11	62000,5'N, 006046'V	62000,5'N, 006046'V



Country	LAB-ID	Sample blank	Sampling site	Site/sample information	Matrix	Sample information	Sampling date	coordinates	coordinates
Faroe Islands	Bron 50		Torshavn	Effluent from sewage treatment plant at the hospital in Torshavn	water	effluent	2006-01-11	62000,5'N, 006046'V	62000,5'N, 006046'V
Faroe Islands	Bron 52		Husahagi	Soil samples at waste deposit. Sampling in vicinity of well where sample 1 was taken	soil		2005-12-28	62°01,712' N,	006048,306 'V
Faroe Islands	Bron 53		Sersjantsvikin	Semi-digested sludge from sewage treatment plant "Sersjantvikin", sampled at surface in tank/bed.	sludge	Digested	2005-12-28	62°00,495' N,	006045,712 'V
Faroe Islands	Bron 54		Strendu	Strendu (Heygsstöð) Near the salmon slaughterery and the paint factory	biota	Fish-Sculpin 5 individuals, approx 20cm	27-29.12.05	62°06,056' N,	06045,578' V
Faroe Islands	Bron 55		Skala	At the marina in Skála	biota	Fish-Dab 11 individuals, approx 20cm	2005-12-28	62°09,326' N,	06046,505' V
Faroe Islands	Bron 56		Torshavn	Torshavn Harbour, at the fish factory "Friskavirking"	biota	Fish-Saith 12 individuals, 15-20cm, ageclass:0 yrs,	2005-12-31	62°00,5'N,	06046,5'V
Faroe Islands	Bron 57		Torshavn	Torshavn Harbour, at Bursatanga and Kongabrugvin	biota	Fish-Cod 10 individuals	31.12.05-06.01.06	62°00,5'N,	06046,5'V
Faroe Islands	Bron 58		Streymoy	West of Streymoy, samples taken on 3.09.05		Seabird- Fulmar Five whole livers, Fg-179(M)16g, 180(M)22g, 181(F)16g, 182(M)13g, 183(M)16g. Age group: 0 yrs not fully fledged (ie young birds).	2005-09-02	62°11'N,	07021'V
Faroe Islands	Bron 59	x	Strendur	Runoff from factory hall. Faroe Island sample 15	water	Surface water	2005-12-29	62°05,850' N,	006046,102 'V
Iceland	Bron 60		Island GUF 1	A coast near Gufunes, an old waste landfill site	water	Coastal recipient water	2006-03-16	64° 8'57.00"N	21°49'13.87 "W

Country	LAB-ID	Sample blank	Sampling site	Site/sample information	Matrix	Sample information	Sampling date	coordinates	coordinates
Iceland	Bron 61		Island GUF 2	A coast near Gufunes, an old waste landfill site	water	Coastal recipient water	2006-03-16	64° 8'57.00"N	21°49'13.87 "W
Iceland	Bron 62		Island ALF 1	Coast near Álfnes, Reykjavík's waste landfill site	water	River recipient water	2006-03-16	64°11'11.72 "N	21°45'16.06 "W
Iceland	Bron 63		Island ALF 2	Coast near Álfnes, Reykjavík's waste landfill site	water	River recipient water	2006-03-16	64°11'11.72 "N	21°45'16.06 "W
Iceland	Bron 64		Island SNA 1	Snarfari, a harbour of yachts and motorboats	water	Coastal recipient water	2006-03-16	64° 7'59.85"N	21°50'24.45 "W

# Appendix 2. Sampling Manual

Analysis of bronopol and resorcinol in a Nordic  
cooperation on screening

Sampling and sample handling manual

NILU/IVL

1. Sediment, sludge, soil, and water samples
2. Biological samples
3. Sampling forms

## 1. Sampling of sediment, sludge, soil, and water

### *General remarks*

Bronopol is frequently used in cosmetic formulations. Whereas resorcinol is used in adhesives and in some pharmaceutical preparations. On the day of sampling, please, do not use products such as antiperspirant, eye shadow, hair spray, or skin lotions and avoid contact with adhesives, glues and similar technical products. Only specially cleaned sampling containers provided by the laboratory should be used.

In contrast to the compounds of the preceding NMR-screening studies especially bronopol must be characterized as rather unstable. Therefore samples must be frozen ( $<-18^{\circ}\text{C}$ ) immediately after sampling and must be kept frozen at any time before analysis.

To check for contamination sampling blanks are used. The sampling blank to be used for sediment, sludge, and soil sampling contains silica particles, the one to be used for water contains MilliQ-water. The sampling blanks should not be emptied or filled. They shall only be opened and closed at the time of sampling.

The number of sampling blanks is limited. If the number of samples per sample type (water, sediment, sludge, soil) and country is one to four the number of sampling blanks is one. If the number of samples per sample type and country is five or more the number of sampling blanks is two. The sites used for blank sampling should be selected at random before the start of sampling.

### *Sampling*

- For each *sludge/sediment* sample fill *one* sample container.
- For each *water* sample please fill *two* sample bottles.
- Arrange the sampling bottle to be used (and, if the site is selected for blank sampling, one sampling blank) on a clean spot on the sampling site.
- Immediately before sampling open the lid of the sampling container (and the sampling blank).
- Fill the sample container, if required using the enclosed spoon, and close the lid on the sample bottles (and sample blank). Label the sample containers. Put each container in a plastic bag.
- Fill in the sample protocol.

### *Storage and transport*

Store the samples frozen and send to the laboratory as soon as possible in such a way that the samples will reach the laboratory within one day. To assure that samples reach the destination within short time (usually within

the same day), they should be sent early in the morning and not on a Friday (preferably Monday to Wednesday). *When sending the samples a notice including the airway bill number (AWB) of the package must be sent to the addressee.* The delivery should be marked with “*samples NMR-screening study*” to avoid unnecessary delays during the registration procedure at the analysing institute.

### Address

Samples from	
Faroe Islands, Island, and Norway	Denmark, Finland, and Sweden
to be sent to	
Norwegian Institute for Air Research (NILU) Att. Arve Bjerke Instituttvegen 18 NO-2027 Kjeller Norway <a href="mailto:Arve.Bjerke@nilu.no">Arve.Bjerke@nilu.no</a>	IVL Swedish Environmental Research Institute Att. Lennart Kaj Valhallavägen 81 SE-114 27 Stockholm. Sweden. <a href="mailto:lennart.kaj@ivl.se">lennart.kaj@ivl.se</a>

## 2. Biological samples

### General remarks

Bronopol is frequently used in cosmetic formulations. Whereas resorcinol is used in adhesives and in some pharmaceutical preparations. On the day of sampling, please, do not use products such as antiperspirant, eye shadow, hair spray, or skin lotions and avoid contact with adhesives, glues and similar technical products. Only specially cleaned sampling containers provided by the laboratory should be used.

In contrast to the compounds of the preceding NMR-screening studies especially bronopol must be characterized as rather unstable. Therefore samples must be frozen (<-18°C) immediately after sampling and must be kept frozen at any time before analysis.

### Sampling

#### Fish and fish liver

Fish should be dissected immediately after collection. Small fishes shall be packed and sent as whole fish. From bigger fishes at least a 10 cm chop from the middle part of each fish shall be sent. Liver samples shall be packed and sent as whole pieces.

**Bird eggs**

Only newly laid eggs are usable for this study. Fresh eggs shall be packed separately, frozen and sent.

**Seal and whale**

The seal or whale should be dissected immediately after collection. From each individual a 5 \* 5 cm slice of meat or blubber shall be taken, packed and sent.

Fill in the sample protocol.

*Storage and transport*

Store the samples frozen and send to the laboratory as soon as possible in such a way that the samples will reach the laboratory within one day. To assure that samples reach the destination within short time (usually within the same day), they should be sent early in the morning and not on a Friday (preferably Monday to Wednesday). *When sending the samples a notice including the airway bill number (AWB) of the package must be sent to the addressee.* The delivery should be marked with “*samples NMR-screening study*” to avoid unnecessary delays during the registration procedure at the analysing institute.

*Address*

Samples from	
Faroe Islands, Island, and Norway	Danmark, Finland, and Sweden
to be sent to	
Norwegian Institute for Air Research (NILU) Att. Arve Bjerke Instituttvegen 18 NO-2027 Kjeller Norway <a href="mailto:Arve.Bjerke@nilu.no">Arve.Bjerke@nilu.no</a>	IVL Swedish Environmental Research Institute Att. Lennart Kaj Valhallavägen 81 SE-114 27 Stockholm. Sweden. <a href="mailto:lennart.kaj@ivl.se">lennart.kaj@ivl.se</a>

### 3. Sampling form – Sediment, sludge, soil and water samples

Sample type:

- Sewage water
- Digested sludge
- Surface water

- Sediment
- Soil
- Sampling blank included

Sample name / identity:

Coordinates for the sample site:

Sampling day:

Shipped to NILU/IVL:

Received at NILU/IVL:

Used sampling equipment:

Responsible person:

Sample storage:

- Freezer
- Fridge
- Other

### Address

Samples from	
Faroe Islands, Island, and Norway	Denmark, Finland, and Sweden
to be sent to	
Norwegian Institute for Air Research (NILU) Att. Arve Bjerke Instituttvegen 18 NO-2027 Kjeller Norway <a href="mailto:Arve.Bjerke@nilu.no">Arve.Bjerke@nilu.no</a>	IVL Swedish Environmental Research Institute Att. Lennart Kaj Valhallavägen 81 SE-114 27 Stockholm. Sweden. <a href="mailto:lennart.kaj@ivl.se">lennart.kaj@ivl.se</a>

### Sampling form – Biological samples

Sample name:	Sample material:
--------------	------------------

Sampling	Comments
Date:	
Site (description, preferably with latitude/longitude data):	
Number and size of individuals used for the pooled sample:	
Age and sex of the individuals	
Storage temp. after sampling:	
Date and site of dissection:	

Total sample amount:		
Special observations:		

*Address*

<b>Samples from</b>	
<b>Faroe Islands, Island, and Norway</b>	<b>Danmark, Finland, and Sweden</b>
<b>to be sent to</b>	
Norwegian Institute for Air Research (NILU) Att. Arve Bjerke Instituttvegen 18 NO-2027 Kjeller Norway <a href="mailto:Arve.Bjerke@nilu.no">Arve.Bjerke@nilu.no</a>	IVL Swedish Environmental Research Institute Att. Lennart Kaj Valhallavägen 81 SE-114 27 Stockholm. Sweden. <a href="mailto:lennart.kaj@ivl.se">lennart.kaj@ivl.se</a>



## Appendix 3; Result table



(n.a = not analysed). LODs are summarised in Table 11 a and 11 b.

Sample ID	Country	Site	Matrix	Matrix information	dw,%	unit	m-Cresol	Bronopol	Resorcinol	Triclosan	unit	m-Cresol	Bronopol	Resorcinol	Triclosan
	Sweden	Grums pulp industry	air			ng/m3	5.2	<0.07	<0.1	<0.003					
	Sweden	Grums pulp industry	air			ng/m3	3.8	<0.07	<0.1	<0.003					
	Sweden	Grums pulp industry	air			ng/m3	3.1	<0.07	<0.1	<0.003					
MR 4662	Danmark	Roskilde Fjord	Biota			ng/g ww	<5	<10	<5	0.13					
MR 4663	Danmark	Roskilde Fjord	Biota			ng/g ww	<5	<10	<5	<0.1					
MR 4672	Danmark	Öresund, Lynetten	Biota			ng/g ww	<5	<10	<5	0.29					
MR 4673	Danmark	Öresund, Lynetten	biota			ng/g ww	<5	<10	<5	0.15					
Bron 19	Norway	Stasjon 30b i JAMP-prg.	Biota	Cod (5 )		µg/l	<0.01	<0.005	<0.005	<0.001					
Bron 29	Norway	Svellet	Biota	Fish (Aspius aspius)		ng/g ww	<10	<5	<5	<1					
Bron 28	Norway	Svellet	Biota	Fish (Pike/Esox lucius)		ng/g ww	<10	<5	<1	<1					
Bron 27	Norway	Svellet	Biota	Fish (Roach/Rutilus-rutilus)		ng/g ww	<10	<5	<1	<1					
Bron 57	Faroe Islands	Torshavn harbour, Faroe Island sample 13	biota	Fish-Cod		ng/g ww	<10	<5	<2	<1					
Bron 55	Faroe Islands	Skála, Faroe Island sample 11	biota	Fish-Dab		ng/g ww	<10	<5	<5	<1					
Bron 56	Faroe Islands	Torshavn harbour, Faroe Island sample 12	biota	Fish-Saith		ng/g ww	<10	<5	<1	<1					
Bron 54	Faroe Islands	Strendur, HeygsstøðFaroe Island sample 10	biota	Fish-Sculpin		ng/g ww	<10	<5	<1	<1					
MR4722	Sweden	Grums pulp industry	Biota	perch, male		ng/g ww	<5	<5	<10	<0.1					
MR4723	Sweden	Ref stn	Biota	perch,male		ng/g ww	<5	<5	<10	<0.1					

Sample ID	Country	Site	Matrix	Matrix information	dw,%	unit	m-Cresol	Bronopol	Resorcinol	Triclosan	unit	m-Cresol	Bronopol	Resorcinol	Triclosan
MR4568	Finland	Huruksela River Kymijoki	Biota	roach (7 ind.)		ng/g ww	<5	<10	<5	<0.1					
Bron 58	Faroe Islands	West of Streymoy, Faroe Island sample 14	biota	Seabind-Fulmar		ng/g ww	<10	<5	<5	<1					
MR4660	Danmark	Roskilde Fjord, Bjergmarken	sediment		46.9	ng/g dw	<0.39	<2.5	<1.3	14	ng/g ww	<0.18	<1.2	<0.6	6.7
MR4670	Danmark	Öresund, Lynetten STP	sediment		76.4	ng/g dw	0.41	<1.9	<1	0.6	ng/g ww	0.31	<1.5	<0.76	0.44
MR4674	Danmark	Sikeborg Langsö,	sediment		31.1	ng/g dw	2.8	<4.2	3.2	2.0	ng/g ww	0.87	<1.3	1.0	0.61
MR4675	Danmark	Slåen sö	sediment		6.52	ng/g dw	5.2	<20	<10	<4.5	ng/g ww	0.34	<1.3	<0.68	<0.29
MR4522	Finland	Kuusankoski River Kymijoki	sediment		31	ng/g dw	<0.66	<4.2	<2.2	2.3	ng/g ww	<0.2	<1.3	<0.67	0.71
MR4551	Finland	Imatra	sediment		7.45	ng/g dw	<2.7	<17	<8.9	<3.8	ng/g ww	<0.2	<1.3	<0.66	<0.29
Bron 09	Norway	Bekkelaget	sediment			ng/g dw	<10	<5	<2	n.a.					
Bron 24	Norway	Svellet S1	Sediment			ng/g dw	<10	<5	<2	<1					
Bron 25	Norway	Svellet S2	Sediment			ng/g dw	<10	<5	17	<1					
MR4771	Sweden	Grums pulp industry	sediment		22.3	ng/g dw	1.4	<6	<3	2	ng/g ww	0.30	<1.4	<0.71	0.38
MR4773	Sweden	Grums pulp industry	sediment		15.8	ng/g dw	<1	<8	<4	<2	ng/g ww	<0.21	<1.3	<0.67	<0.29
MR4775	Sweden	Grums pulp industry	sediment		30	ng/g dw	<1	<5	<2	<1	ng/g ww	<0.22	<1.4	<0.72	<0.31
Bron 05	Norway	VEAS	Sediment+TOC			ng/g dw	<10	<5	3	175					
MR4657	Danmark	Roskilde, Bjergmarken	sludge		15.7	ng/g dw	30	<18	11	2000	ng/g ww	4.7	<2.9	2	310
MR4668	Danmark	Öresund, Lynetten STP	sludge		86	ng/g dw	4.7	<13	<6.6	1600	ng/g ww	4.0	<11	<5.6	1300
MR4516	Finland	Kuusankoski STP	sludge		17.9	ng/g dw	<2.3	<15	<7.5	850	ng/g ww	<0.41	<2.6	<1.3	150
MR4526	Finland	Kuusankoski STP	sludge		15.4	ng/g dw	<2.6	<17	<8.6	3200	ng/g ww	<0.4	<2.6	<1.3	480
MR4529	Finland	Kuusankoski pulp and	sludge		42	ng/g dw	<1.8	<12	<5.9	<2.6	ng/g ww	<0.76	<4.8	<2.5	<1.1

Sample ID	Country	Site	Matrix	Matrix information	dw,%	unit	m-Cresol	Bronopol	Resorcinol	Triclosan	unit	m-Cresol	Bronopol	Resorcinol	Triclosan
		paper industry													
MR4536	Finland	Kuusankoski pulp and paper industry	sludge		35.5	ng/g dw	<1.7	<11	<5.7	<2.5	ng/g ww	<0.62	<3.9	<2	<0.88
MR4541	Finland	Imatra	sludge		25.4	ng/g dw	110	<17	<8.7	<3.7	ng/g ww	27	<4.2	<2.2	<0.94
MR4549	Finland	Imatra	sludge		5.34	ng/g dw	<7	<45	24.0	<9.9	ng/g ww	<0.37	<2.4	1.3	<0.53
MR4558	Finland	Lappeenranta. Toikansuo STP	sludge		23.8	ng/g dw	18	<11	56	590	ng/g ww	4.3	<2.7	13	140
MR4563	Finland	Espoo, Suomenoja STP	sludge		27.9	ng/g dw	30	<12	9.2	2200	ng/g ww	8.5	<3.5	2.6	610
MR4567	Finland	Espoo, Suomenoja STP	sludge		29.7	ng/g dw	<1.9	<12	15	2000	ng/g ww	<0.57	<3.6	4.3	580
Bron 08	Norway	Bekkelaget	sludge			ng/g dw	<10	<5	<2	n.a.					
MR4168	Sweden	Ekilstuna, Ekeby STP	sludge		28.2	ng/g dw	5.9	<15	13	4000	ng/g ww	1.7	<4.2	3.6	1100
MR4196	Sweden	Ronneby, Rustorp STP	sludge		17.6	ng/g dw	8.0	<21	11	1600	ng/g ww	1.4	<3.8	2.0	280
MR4208	Sweden	Hallstahammar, Möintorp STP	sludge		26.5	ng/g dw	4.5	<15	20	2300	ng/g ww	1.2	<3.9	5.4	600
MR4217	Sweden	Skövde, Stadskvarn STP	sludge		16.6	ng/g dw	8.9	<20	15	3300	ng/g ww	1.5	<3.3	2.4	550
MR4234	Sweden	Gällivare, Kavaheden STP	sludge		20.2	ng/g dw	28	<24	82	2300	ng/g ww	5.6	<4.9	17	460
MR4273	Sweden	Piteå, Sandholmen STP	sludge		25.4	ng/g dw	36	<17	16	3600	ng/g ww	9.0	<4.3	4.0	920
MR4303	Sweden	Hudiksvall, Reffelmansverket STP	sludge		27.7	ng/g dw	10	<16	26	3000	ng/g ww	2.8	<4.4	7.3	840
MR4784	Sweden	Grums pulp industry	sludge		1.6	ng/g dw	<7	<45	48	<10	ng/g ww	<0.11	<0.72	0.77	<0.16
Bron 53	Faroe Islands	Sersjantvikin sewage treatment plant, Faroe Island sample 9	sludge	digested		ng/g dw	106	<5	219	44					
Bron 36	Iceland	Island KI1	Sludge	Non proc-		ng/g dw	<10	<5	9	77					

Sample ID	Country	Site	Matrix	Matrix information	dw,%	unit	m-Cresol	Bronopol	Resorcinol	Triclosan	unit	m-Cresol	Bronopol	Resorcinol	Triclosan
				essed, non dehydrated											
Bron 37	Iceland	Island K12	Sludge	Non processed, non dehydrated		ng/g dw	<10	<5	<1	79					
Bron 38	Iceland	Island An1	Sludge	Non processed, non dehydrated		ng/g dw	<10	<5	107	17					
Bron 39	Iceland	Island An2	Sludge	Non processed, non dehydrated		ng/g dw	<10	<5	25	40					
Bron 52	Faroe Islands	Husahagi landfill, Faroe Island sample 8	soil			ng/g dw	96	<5	<2	<1					
MR4785	Sweden	Grums pulp industry	water			µg/l	<0.014	<0.01	<0.01	<0.0021					
MR4656	Danmark	Roskilde, Bjergmarken	water	Effluent		µg/l	0.014	<0.1	<0.0054	0.077					
MR4667	Danmark	Öresund, Lynetten TP	water	effluent		µg/l	<0.014	<0.1	0.24	0.046					
Bron 03	Norway	VEAS	water	effluent		µg/l	<0.1	<0.1	<0.1	<0.1					
Bron 06	Norway	Bekkelaget	water	effluent		µg/l	<0.1	<0.1	<0.1	<0.1					
Bron 31	Norway	Öyern Industrial	water	Effluent		µg/l	<0.1	<0.1	<0.1	<0.1					
MR4165	Sweden	Ekilstuna, Ekeby STP	water	effluent		µg/l	<0.014	<0.1	<0.0056	0.040					
MR4195	Sweden	Ronneby, Rustorp STP	water	effluent		µg/l	<0.014	<0.1	<0.0055	0.044					
MR4207	Sweden	Hallstahammar, Möntorp STP	water	effluent		µg/l	<0.014	<0.1	<0.0056	0.018					
MR4215	Sweden	Skövde, Stadskvarn STP	water	effluent		µg/l	<0.014	<0.1	<0.0055	0.013					
MR4233	Sweden	Gällivare, Kavaheden STP	water	effluent		µg/l	0.018	<0.1	0.008	0.016					

Sample ID	Country	Site	Matrix	Matrix information	dw,%	unit	m-Cresol	Bronopol	Resorcinol	Triclosan	unit	m-Cresol	Bronopol	Resorcinol	Triclosan
MR4271	Sweden	Piteå, Sandholmen STP	water	effluent		µg/l	<0.014	<0.1	<0.0057	0.044					
MR4288	Sweden	Helsingborg, Öresunds-verket STP	water	effluent		µg/l	<0.014	<0.1	<0.0056	0.031					
MR4302	Sweden	Hudiksvall, Reffelmans-verket STP	water	effluent		µg/l	0.030	<0.1	0.007	0.024					
MR4166	Sweden	Ekilstuna, Ekeby STP	water	effluent after wetland		µg/l	<0.014	<0.1	<0.0056	0.0051					
MR4514	Finland	Kuusankoski STP	water	Efluent		µg/l	<0.014	<0.1	<0.0058	0.071					
MR4525	Finland	Kuusankoski STP	water	Efluent		µg/l	<0.014	<0.1	<0.0058	0.12					
MR4528	Finland	Kuusankoski pulp and paper industry	water	Efluent		µg/l	<0.013	<0.1	<0.0054	<0.0021					
MR4535	Finland	Kuusankoski pulp and paper industry	water	Efluent		µg/l	<0.014	<0.1	<0.0057	<0.0022					
MR4554	Finland	Lappeenranta, Toikansuo STP	water	Efluent		µg/l	0.017	<0.1	<0.0056	0.10					
MR4557	Finland	Lappeenranta, tokansuo STP	water	Efluent		µg/l	<0.014	<0.1	<0.0057	0.10					
MR4562	Finland	Espoo, Suomenoja STP	water	Efluent		µg/l	<0.013	<0.1	0.011	0.10					
MR4566	Finland	Espoo, Suomenoja STP	water	Efluent		µg/l	<0.014	<0.1	<0.0056	0.09					
MR4539	Finland	Imatra	water	Efluent / biological line		µg/l	<0.014	<0.1	0.0088	<0.0022					
MR4546	Finland	Imatra	water	Efluent / biological line		µg/l	0.015	<0.1	<0.0054	<0.0021					
MR4540	Finland	Imatra	water	Efluent / chemical line		µg/l	0.16	<0.1	<0.0056	<0.0021					
MR4548	Finland	Imatra	water	Efluent / chemical line		µg/l	1.2	<0.1	0.071	<0.0021					
MR4655	Danmark	Roskilde ,Bjergmarken	water	Influent		µg/l	3.0	<0.1	0.075	0.084					
MR4666	Danmark	Öresund, Lynetten STP	water	influent		µg/l	0.87	<0.1	3.50	0.13					

Sample ID	Country	Site	Matrix	Matrix information	dw, %	unit	m-Cresol	Bronopol	Resorcinol	Triclosan	unit	m-Cresol	Bronopol	Resorcinol	Triclosan
MR4513	Finland	Chem industry and Kuusankoski STP	water	Influent		µg/l	1.20	<0.1	0.48	0.15					
MR4524	Finland	Chem industry and Kuusankoski STP	water	Influent		µg/l	0.40	<0.1	0.74	0.12					
MR4527	Finland	Kuusankoski pulp and paper industry	water	Influent		µg/l	0.24	<0.1	<0.0057	<0.0022					
MR4534	Finland	Kuusankoski pulp and paper industry	water	Influent		µg/l	2.3	<0.1	<0.0056	<0.0021					
MR4553	Finland	Lappeenranta, Toikansuo STP	water	Influent		µg/l	0.51	<0.1	1.6	0.15					
MR4556	Finland	Lappeenranta, Toikansuo STP	water	Influent		µg/l	2.1	<0.1	0.32	0.12					
MR4560	Finland	Espoo, Suomenoja STP	water	Influent		µg/l	0.97	<0.1	2.5	0.15					
Bron 01	Norway	VEAS	water	influent		µg/l	<0.1	<0.1	<0.1	0.057					
Bron 07	Norway	Bekkelaget	water	influent		µg/l	<0.1	<0.1	<0.1	<0.1					
Bron 30	Norway	Öyern Industrial	water	Influent		µg/l	<0.1	<0.1	0.085	<0.1					
MR4565	Finland	Espoo, Suomenoja STP	water	Influent water		µg/l	0.38	<0.1	0.74	0.12					
Bron 45	Faroe Islands	Husahagi landfill, Faraoe Island sample 1	water	leachate		µg/l	<0.1	<0.1	<0.1	0.0464					
MR4512	Finland	Chem industry and Kuusankoski STP	water	Leachate		µg/l	<0.014	<0.1	<0.0057	0.010					
MR4523	Finland	Chem industry and Kuusankoski STP	water	Leachate		µg/l	0.72	<0.5	<0.0058	<0.0022					
MR4552	Finland	Lappeenranta, Toikansuo landfill	water	Leachate		µg/l	1.4	<0.1	<0.0056	0.0036					



Sample ID	Country	Site	Matrix	Matrix information	dw,%	unit	m-Cresol	Bronopol	Resorcinol	Triclosan	unit	m-Cresol	Bronopol	Resorcinol	Triclosan
MR4555	Finland	Lappeenranta, Toikansuo landfill	water	Leachate		µg/l	0.22	<0.1	<0.0056	<0.0021					
MR4559	Finland	Espoo, Ämmässuo landfill	water	Leachate		µg/l	0.043	<0.1	<0.8	0.010					
MR4564	Finland	Ämmässuo landfill	water	Leachate		µg/l	1.8	<0.1	<0.4	0.009					
MR4292	Sweden	Helsingborg, Öresunds-verket STP	water	leachate		µg/l	77	<0.1	<0.0075	<0.07					
MR4170	Sweden	Eskilstuna	water	leachate before cleaning		µg/l	0.058	<0.1	<0.0054	<0.0021					
Bron 60	Iceland	Island GUF 1	water	Coastal recipient water		µg/l	<0.1	<0.1	0.0551	<0.1					
Bron 61	Iceland	Island GUF 2	water	Coastal recipient water		µg/l	<0.1	<0.1	<0.1	<0.1					
Bron 62	Iceland	Island ALF 1	water	River recipient water		µg/l	<0.1	<0.1	0.0084	<0.1					
Bron 63	Iceland	Island ALF 2	water	River recipient water		µg/l	<0.1	<0.1	0.0126	<0.1					
Bron 64	Iceland	Island SNA 1	water	Coastal recipient water		µg/l	<0.1	<0.1	0.069	<0.1					
Bron 48	Faroe Islands	Sersjantvikin sewage treatment plant, Faroe Island sample 4	water	Sewage		µg/l	<0.1	<0.1	<0.1	<0.1					
Bron 49	Faroe Islands	Inflow to hospital sewage treatment plant, Torshavn, Faroe Island sample 5	water	Sewage		µg/l	<0.1	<0.1	<0.1	0.13					
Bron 50	Faroe Islands	Effluent from hospital sewage treatment plant Torshavn, Faroe Island	water	Sewage		µg/l	<0.1	<0.1	0.05	<0.1					

Sample ID	Country	Site	Matrix	Matrix information	dw,%	unit	m-Cresol	Bronopol	Resorcinol	Triclosan	unit	m-Cresol	Bronopol	Resorcinol	Triclosan
		sample 6													
Bron 46	Faroe Islands	Sersjantvikin sewage treatment plant, effluent water. Faroe Island sample 2	water	Sewage water		µg/l	<0.1	<0.1	<0.1	<0.1					
MR4658	Danmark	Roskilde Fjord	water	Surface		µg/l	<0.014	<0.1	<0.0056	0.0027					
MR4664	Danmark	Kattegatt	water	Surface		µg/l	<0.014	<0.1	<0.0055	<0.0021					
MR4669	Danmark	Öresund, Lynetten STP	water	Surface		µg/l	<0.014	<0.1	<0.0055	<0.0021					
MR4803	Danmark	Slåen sø	water	surface		µg/l	<0.014	<0.1	<0.0056	<0.0021					
MR4805	Danmark	Sillkeborg Langsø	water	surface		µg/l	<0.014	<0.1	<0.0056	<0.0021					
Bron 47	Faroe Islands	Torshavn harbour, sea-water. Faroe Island sample 3	water	Surface		µg/l	<0.1	<0.1	0.0857	<0.1					
Bron 59	Faroe Islands	Runoff from factory hall, Strendur. Faroe Island sample 15	water	Surface		µg/l	<0.1	<0.1	<0.005	<0.005					
MR4517	Finland	Kuusankoski River Kymijoki	water	Surface		µg/l	<0.014	<0.1	<0.0056	<0.0021					
MR4550	Finland	Imatra	water	Surface		µg/l	0.022	<0.1	<0.006	<0.0023					
Bron 43	Iceland	Island Alf4	water	Coastal recipient water		µg/l	<0.1	<0.1	0.0018	<0.1					
Bron 10	Norway	Bekkelaget	water	Surface		µg/l	<0.1	<0.1	<0.1	<0.1					
Bron 11	Norway	VEAS	water	surface		µg/l	<0.1	<0.1	<0.1	<0.1					
Bron 15	Norway	E-Agder industrial site 1	water	Surface		µg/l	<0.1	<0.1	<0.1	<0.1					
Bron 16	Norway	E-Agder industrial site 2	water	surface		µg/l	<0.1	<0.1	0.354	<0.1					

Sample ID	Country	Site	Matrix	Matrix information	dw, %	unit	m-Cresol	Bronopol	Resorcinol	Triclosan	unit	m-Cresol	Bronopol	Resorcinol	Triclosan
Bron 20	Norway	Svellet V1	water	surface		µg/l	<0.1	<0.1	<0.1	<0.1					
Bron 21	Norway	Svellet V2	water	surface		µg/l	<0.1	<0.1	0.0398	<0.1					
Bron 22	Norway	Svellet V3	water	surface		µg/l	<0.1	<0.1	0.0789	<0.1					
MR4770	Sweden	Grums pulp industry	water	Surface		µg/l	<0.014	<0.1	<0.0056	<0.0021					
MR4772	Sweden	Grums pulp industry	water	Surface		µg/l	<0.013	<0.1	<0.0054	<0.0021					
MR4774	Sweden	Grums pulp industry	water	Surface		µg/l	<0.013	<0.1	<0.0053	<0.002					