SWECO Environment
Screening Report 2008:2

# Screening of musk substances

Client

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# Content

# Sammanfattning

**Bakgrund och metoder**

Myskämnen är benämningen på en rad artificiellt framställda ämnen med likartade doftegenskaper. Dessa används som viktiga komponenter i olika doftblandningar som används i en lång rad produkter:

- Tvättmedelsprodukter
- Mjukgörningsmedel för tyger
- Luftdoftare
- Schampo
- Parfymer
- Kosmetika
- Tobaksprodukter

Inom den nationella screeningen 2007 har SWECO Environment på uppdrag av Naturvårdsverket utfört mätningar för att kartlägga förekomsten i miljön av myskämnena musk xylene, musk ketone, galaxolide, tonalide, celestolide, traesolide, phantolide, cashmeran, musk ambrette, musk moskene och musk tibetene.

Syftet med screeningstudien var att belysa de huvudsakliga källorna till myskämnen, belysa halterna av myskämnen i miljön samt, om ämnena återfinns, mycket summariskt bedöma om halterna utgör någon miljörisk.

Myskämnena har mätts i ett flertal matriser i provpunkter som påverkas av olika punktkällor. Provtagningen genomfördes nationellt med provpunkter som valdes av Naturvårdsverket och SWECO samt regionalt med provpunkter som valdes av respektive länsstyrelse. Resultaten från den regionala samt den nationella provtagningen samutvärderades.

En provtagningsstrategi togs fram omfattande provtagning av inkommande och utgående vatten samt slam vid avloppsreningsverk liksom provtagning av ytvatten, sediment och fisk i vattendrag som tar emot utgående vatten från avloppsreningsverk. Avloppsreningsverk valdes som viktiga provtagningsplatser eftersom de tros vara den stora källan till dessa ämnens förekomst i miljön. Ytterligare ett antal miljömatriser påverkade av urban aktivitet ingick i undersökningen såsom jord, grundvatten, ytvatten, sediment, dagvatten, sediment från dagvattenbrunnar och fisk. Myskämnena analyserades också i blod från förstföderskor.

Bakgrundshalter i ytvatten, fisk och sediment bestämdes i referenssjöar samt i jord som omgav dessa sjöar.

## Slutsatser och rekommendationer

Myskämnen har i den här studien påvisats i in- och utgående vatten samt slam från avloppsreningsverk. De har även påvisats i ytvatten, sediment och fisk i recipienter till avloppsreningsverk. Inga myskämnen har däremot påträffats i prover från bakgrundslokaler, från diffust påverkade lokaler eller från lokaler påverkade av deponier eller hushåll. Inte heller vid analyser av blod kunde myskämnen påvisas.

Myskämnen har påvisats i samtliga slamprover från avloppsreningsverk i den här studien. Koncentrationerna varierar kraftigt, vilket också har visats i tidigare studier. Däremot är det okänt vad denna höga variation beror på (t.ex. storlek och driftsparametrar i avloppsreningsverket samt avloppsvattnets ursprung).

För att avgöra om de halter som påvisats i recipienter till avloppsreningsverk har någon negativ påverkan på miljön har de uppmätta koncentrationerna jämförts med de lägsta halter som kan utgöra någon risk för miljön (PNEC, Predicted No Effect Concentration). Den högsta kvoten mellan påträffade koncentrationer och PNEC var 0,26 för galaxolide i ytvatten. Denna kvot erhölls i ytvatten nedströms ett reningsverk och utgör inte något direkt miljöproblem. Eftersom endast ett fåtal prover gjordes i ytvatten nedströms reningsverk kan man inte utesluta att negativa effekter av myskämnen kan förekomma i sådana recipienter beroende på årstidsvariationer och variationer mellan reningsverk.

De huvudsakliga slutsatserna från denna studie var att:

- Myskämnen är vanliga i inkommande vatten, slam och utgående vatten från avloppsreningsverk
- Myskämnen förekommer i ytvatten och sediment nedströms reningsverk. Studien indikerar att avloppsreningsverk är den troligaste källan till förekomsten av myskämnen i miljön
- De uppmätta koncentrationerna utgör troligtvis inte något direkt miljöproblem eftersom kvoten MEC/PNEC är mindre än ett
- Myskämnen förekommer i fisk, vilket även visats tidigare

Några rekommendationer för framtida studier är:

- Myskämnen skulle kunna mätas över tiden i ett antal ytvatten där utgående vatten från avloppsreningsverk är av kvantitativ betydelse för det totala flödet. Syftet skulle vara att säkerställa att risknivåerna aldrig överskrids.
- Screening av myskämnen skulle vara av intresse att utföra på jord och biota där slam tillförts jordbruksmark.

- En massbalansstudie kan vara av intresse där mängden myskämnen som når avloppsreningsverk, mängden som lagras i slam och mängden som når ytvatten kvantifieras. Dessa mängder skulle kunna jämföras med mängder i produktregistret.
- En studie av hur driftsparametrar i avloppsreningsverken samt källor till inkommande vatten påverkar mängden myskämnen i slam och utgående vatten vore av intresse, för att bättre förstå vad som styr förekomsten av myskämnen i recipienter.
- Metaboliter av musk xylene och musk ketone har återfunnits i slam och utgående vatten från avloppsreningsverk i USA och Tyskland i halter som överstiger ursprungssubstanserna. En screening av myskmetaboliter med fokus enbart på avloppsreningsverk och deras ytvattenrecipienter är därför av intresse.

# Summary

**Background and methods**

Within the screening program of 2007 SWECO Environment has had the assignment from the Swedish Environmental Protection Agency to measure the occurrence of the musk substances Musk Xylene, Musk Ketone, Galaxolide, Tonalide, Celestolide, Traesolide, Phantolide, Cashmeran, Musk Ambrette, Musk Moskene, and Musk Tibetene.

The objectives of the project were to elucidate the main sources of these substances to the environment, to elucidate the levels of these substances in the environment and, if the substances are found, briefly assess whether the levels constitute an environmental and/or health problem.

A national sampling strategy was devised which included sampling of incoming water, effluents and sludge at sewage treatment plants as well as sampling of surface waters, sediments and fish in streams receiving effluents from sewage treatments plants. Sewage treatment plants were chosen as important sampling localities because they are believed to be the major source of these substances to the environment. A number of other environmental matrices impacted by urban activities were also sampled. This included soil, groundwater, surface water, sediments, storm water, sediments from storm water manholes and fish. Musk substances were also analyzed in blood samples from mothers giving birth to their first child.

Swedish environmental background levels in surface water, fish, and sediments were determined in reference lakes and soil and groundwater in the vicinity of the reference lakes.

**Conclusions and recommendations**

The main conclusions from this investigation were:

- Musk compounds are common in incoming water, sludge and effluents at sewage treatment plants
- Musk compounds occur in surface waters and sediments downstream of sewage treatment plants. The study indicates that sewage treatment plants are the most likely source of musk substances to the environment.
- The concentrations found do not seem to pose any direct concern since the MEC/PNEC values were below one.
- As shown earlier, Musk substances occur in fish

Some recommendation for future studies are:

- Musk substances could be measured over time in a few surface waters where the effluent from sewage treatment plants is of quantitative importance for the total flow. The purpose would be to ascertain that risk levels are never exceeded even under worst-case conditions.
- Screening of musk substances could be performed in soil and biota where sludge has been applied to agricultural soils.
- A mass balance study where the amounts of different musk substances reaching waste water treatment plants, the amounts stored in sludge and the amounts reaching surface waters would be of interest to elucidate the magnitude of the problems associated with musk substances.
- A study of how operating parameters in the sewage treatment plants as well as sources of incoming water affect the levels of musk substances in sludge and outgoing water could also be of interest to better understand which circumstances that causes these substances to occur in the downstream recipients.
- Metabolites of Musk Xylene and Musk Ketone have been found in sludge and effluent from sewage treatment plants in USA and Germany. Hence, screening of musk metabolites with focus only on sewage treatment plants and their downstream surface water recipients is of interest.

# 1 Introduction

## 1.1 Background

At present there is a lack of knowledge regarding the emission, distribution and exposure for many of the chemicals emitted to the environment. The aim of the screening program financed by the Swedish Environmental Protection Agency is to alleviate this lack of knowledge by estimating the occurrence of different chemicals in the environment in relevant matrices (soil, water etc.).

To maximize the information gained from the screening program measurements are made in many matrices at many sites, but with few samples per site. The Swedish EPA is responsible for the screening at the national level and selects the chemicals that are to be included. The regional county boards have the option to complement and extend the sampling program by choosing additional sampling point that are of regional interest.

Within the screening program of 2007 SWECO Environment has had the assignment from the Swedish Environmental Protection Agency to measure the occurrence of the following musk substances:

- Musk Xylene (MX)
- Musk Ketone (MK)
- Galaxolide (HHCB)
- Tonalide (AHTN)
- Celestolide (ADBI)
- Traesolide (ATII)
- Phantolide (AHDI)
- Cashmeran (DPMI)
- Musk Ambrette (MA)
- Musk Moskene
- musk tibetene

## 1.2 Objectives

The objectives of the project were to:

- Elucidate the main sources of these substances to the environment

- Elucidate the levels of these substances in the environment
- If the substances are found, briefly assess whether the levels constitutes an environmental problem

## 1.3 Substance information

### 1.3.1 Usage

Musk is the name originally given to a substance with a penetrating odor obtained from a gland of the male musk deer and the substance has been used as a perfume since ancient times. The name "musk" has later on come to encompass a wide variety of substances with somewhat similar odors although many of them are quite different in their chemical structures (Wikipedia, Merriam Websters Dictionary, Sommer 2004). This includes artificial substances with similar odors which at present are the ones that are used almost exclusively. Artificial musk substances are also the focus of this screening study.

The artificial musk compounds (hereafter named only musk compounds) are used as ingredients in fragrance compositions which are complex mixtures prepared by blending many fragrance ingredients in varying concentrations. The musk containing fragrance compositions are used in a large number of products. This includes (European Union 2005, Kallenborn et al 1999):

- washing and cleaning agents
- fabric softeners
- air fresheners
- shampoos
- perfumes
- cosmetic products
- food additives in fish baits
- cigarettes

There is no production of nitro musk compounds in the European Union. Instead, producers in China are now the most important source for the European imports (European Union 2005a ,2005b). Galaxolide and Tonalide are produced at one inland plant in the EU for each substance (OSPAR 2004). Musk ketone, Musk Xylene, Galaxolide and Tonalide represented about 95 percent of the European market 2004.

Table 1.1 shows the usage/import of the most common musk compounds by fragrance compounding facilities in Europe. The amounts in table 1.1 supposedly account for approximately 90% of the total use in Europe as 32 of the major fragrance companies involved in fragrance compounding responded to the survey (OSPAR

2004). In the Swedish product registry there is only registered data on Galaxolide (Figure 1.1). Other musk substances either lack data or there are only zero tones registered yearly. The reason is most likely that these substances are imported in products at low concentrations in which case they will not be reported to the registry. Consumption of Musk Ketone and Musk Xylene in Norway in 2001 was 400 kg and 200 kg respectively (TemaNord 2004).

Consumption in Denmark according to the Danish product register is given in table 1.2 below. Information about use of fragrances forwarded to the Danish product register is given voluntarily and the consumption indicated in table 1.2 is therefore a minimum value.

*Table 1.1 Usage/import of the most common musk compounds by fragrance compounding facilities in Europe 2001. Source: (OSPAR 2004).*

| Year | Musk ketone | Musk xylene | Galaxolide | Tonalide | Celestolide | Musk moskene | Musk tibetene |
|---|---|---|---|---|---|---|---|
| 1992 | 124 | 174 | 2400 | 885 | | | |
| 1995 | 61 | 110 | 1482 | 585 | 34 | 5 | 0.8 |
| 1996 | 54 | 105 | | | | | |
| 1998 | 40 | 86 | 1473 | 385 | 18 | | |
| 2000 | 35 | 67 | 1427 | 358 | 15 | | |

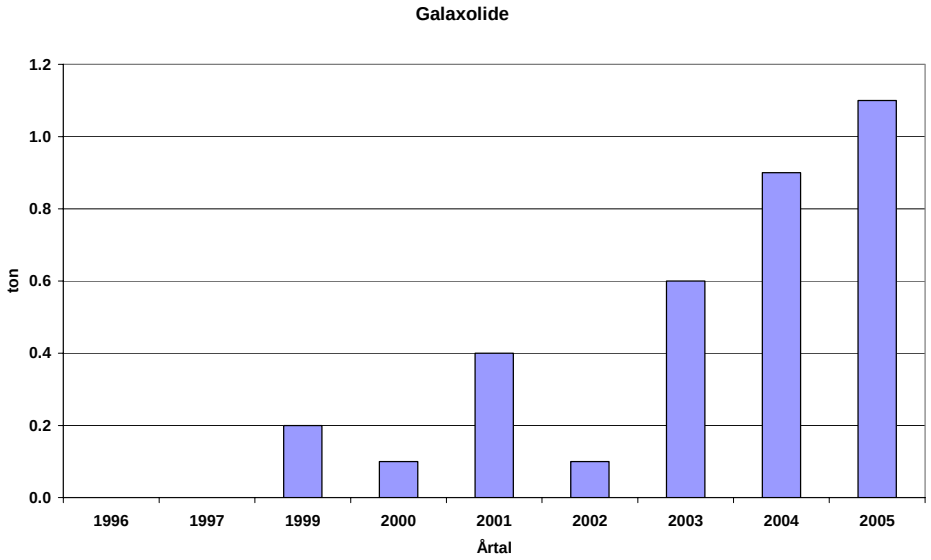



*Figure 1.1 Registered amounts of Galaxolide from the year 1996 – 2005.*

*Table 1.2 Consumption and usage of musk substances in Denmark (year unknown). Source: TemaNord (2004).*

| substance | amount per year (kg) | main use |
|---|---|---|
| Musk ketone | 20 | cosmetics |
| Musk xylene | 134 | cosmetics |
| Galaxolide | 333 | cosmetics and cleaning agents |
| Tonalide | 33 | cleaning agents |
| Traesolide | 14 | cleaning agents |
| Cashmeran | 1 | |
| Celestolide | < 1 | |
| Phantolide | < 1 | |

### 1.3.2 Properties

The musk substances can be divided into three major classes (Sommer 2004):

aromatic nitro musks:

- Musk Baur
- Musk Ketone
- Musk Xylene
- Musk Ambrette
- Musk Moskene

polycyclic musk compounds:

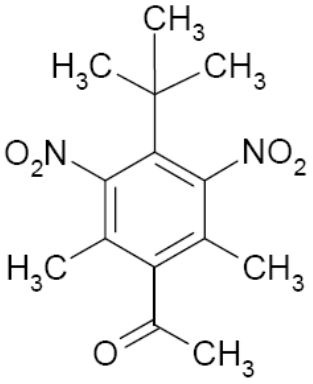
- Galaxolide (HHCB)
- Tonalide (AHTN)
- Phantolide
- Celestolide (Crysolide)
- Traesolide
- Cashmeran

macrocyclic musk compounds:

- Globalide (Habanolide)
- Ambrettolide
- Muscone
- Thibetolide (Exaltolide)
- Velvione

*Table 1.3* to table 1.13 presents physical and chemical properties as well as toxicological and ecotoxicological information for the substances included in this study. For most musk substances there is only scant available information. The exception is Musk Xylene and Musk Ketone which have been the focus of risk assessments within the European Union (European Union 2005a ,2005b).

*Table 1.3 Physiochemical and (Eco)toxicological properties of Musk Xylene. Source European Union, 2005b)*

| Common name | **Musk xylene (MX)** | | | |
|---|---|---|---|---|
|  | | | | |
| Name | 5-tert-butyl-2,4,6-trinitro-m-xylene | | | |
| CAS # | 81-15-2 | | | |
| Labelling | E; Xn; N<br>R: 2-40-50/53<br>S: (2-)36/37-46-60-61 | | | |
| | | Min | Max | Unit |
| Physico-chemical properties | Water solubility | 0.15[1] | 0.46[2] | mg/l |
| | Log $K_{ow}$ | 3.4[1] | 4.9[1] | |
| | Henry's law constant | 0.0595 | | Pa*m$^3$/mol |
| | Vapour pressure | 3*10$^{-5}$ | | Pa |
| | | | | |
| Ecotoxicology aquatic | $EC_{50}$ and $LC_{50}$ | 0.12 | 3.75 | mg/l |
| | NOEC and LOEC | 0.056 | | mg/l |
| | $PNEC_{water}$ | 0.0011 | | mg/l |
| | $PNEC_{sed}$ | 0.3 | | mg/kg |
| Ecotoxicology terrestrial | $EC_{50}$ and $LC_{50}$ | | | mg/kg soil |
| | NOEC and LOEC | >50 | | mg/kg soil |
| | PNEC | 0.26 | | mg/kg soil |
| Toxicology | Oral $LD_{50}$ >2000 = mg/kg; dermal $LD_{50}$ >2000 = mg/kg | | | |
| Persistence, Bioaccumulation, Toxicity (PBT) | Musk xylene is considered to be a PBT candidate substance. | | | |
| Theoretical removal in STP | 98.7 % | | | |

[1] Measured

[2] Calculated

*Table 1.4 Physiochemical and (Eco)toxicological properties of Musk Ketone. Source European Union, 2005a)*

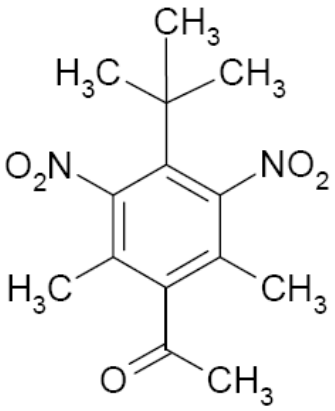
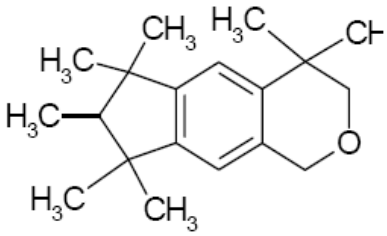
| Common name | **Musk ketone (MK)** | | | |
|---|---|---|---|---|
|  | | | | |
| Name | 1-tert-Butyl-3,5-dimethyl-2,6-dinitrobenzene | | | |
| CAS # | 81-14-1 | | | |
| Labelling | Xn; N<br>R40-50/53<br>S(2)-36/37-46-60-61 | | | |
| | | Min | Max | Unit |
| Physico-chemical properties | Water solubility | 0.46 | 1.9 | mg/l |
| | Log $K_{ow}$ | 3.2 | 4.3 | |
| | Henry's law constant | 0.0256 | | Pa*m$^3$/mol |
| | Vapor pressure | 4*10$^{-5}$ | | Pa |
| | | | | |
| Ecotoxicology aquatic | $EC_{50}$ and $LC_{50}$ | 0.17 | 2.5 | mg/l |
| | NOEC and LOEC | 0.010 | 0.100 | mg/l |
| | PNEC | 0.001 | 0.37 | mg/l |
| Ecotoxicology terrestrial | $EC_{50}$ and $LC_{50}$ | | 1 | mg/kg soil |
| | NOEC and LOEC | 32 | 316 | mg/kg soil |
| | PNEC | 0.22 | | mg/kg soil |
| Toxicology | Oral $LD_{50}$ >2000 = mg/kg; dermal $LD_{50}$ >2000 = mg/kg | | | |
| Persistence, Bioaccumulation, Toxicity (PBT) | Musk ketone is considered not to be a PBT candidate substance. | | | |
| Theoretical removal in STP | 92 % | | | |

*Table 1.5 Physiochemical and (Eco)toxicological properties of Galaxolide.*

| Common name | **Galaxolide (HHCB)** | | | |
|---|---|---|---|---|
|  | | | | |
| Name | 1,3,4,6,7,8-Hexahydro-4,6,6,7,8,8-hexamethylcyclopenta[g]-2-benzopyrane | | | |
| CAS # | 1222-05-5 | | | |
| Labelling | Xi N - Irritant, Dangerous for the environment.<br>R 38 50/53[1] | | | |
| | | Min | Max | Unit |
| Physico-chemical properties | Water solubility | 1.65[2] | 1.75[1] | mg/l |
| | Log $K_{ow}$ | 5.9[2] | 6.6[1] | |
| | Henry's law constant | $1.1*10^{-43}$ | | atm*$m^3$/mol |
| | Vapour pressure | 0.068[2] | 0.0727[3] | Pa |
| | | | | |
| Ecotoxicology aquatic | $EC_{50}$ and $LC_{50}$ | 0.24[4] | 0.29[4] | mg/l |
| | NOEC and LOEC | | 0.14[4] | mg/l |
| | PNECaqua[6] | | 0.00068[6] | mg/l |
| | PNECsediment[6] | | 0.32 [6] | mg/kg |
| Ecotoxicology terrestrial | $EC_{50}$ and $LC_{50}$ | 105[4] | | mg/kg soil |
| | NOEC and LOEC | 45[4] | 19.5[4] | mg/kg soil |
| Toxicology | Oral $LD_{50}$ >3000 mg/kg; dermal $LD_{50}$ >6500 mg/kg[3] | | | |
| Persistence, Bioaccumulation, Toxicity (PBT) | Not PBT (Pbt)[5] | | | |
| Theoretical removal in STP | 118 % av daglig belastning[5] | | | |

[1] http://www.thegoodscentscompany.com/data/rw1007751.html

[2] http://www.norden.org/pub/miljo/miljo/sk/rw1007751 .pdf

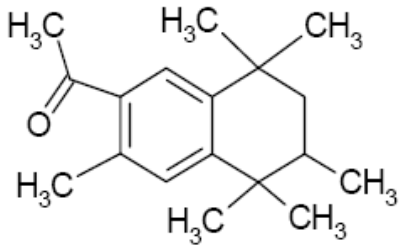
[3] HERA 2004b

[4] http://jhs.pharm.or.jp/52(3)/52_276.pdf

[5] http://www.heraproject.com/files/28-E-36551E10-F8EF-E807-E4199B9BB0076A9F.pdf

[6] Data from freshwater species, but valid for Marine environment. OSPAR 2004.

*Table 1.6 Physiochemical and (Eco)toxicological properties of Tonalide.*

| Common name | **Tonalide (AHTN)** | | | |
|---|---|---|---|---|
|  | | | | |
| Name | 7-Acetyl-1,1,3,4,4,6-hexamethyltetrahydro-naphtlene | | | |
| CAS # | 1506-02-1 | | | |
| Labelling[1] | Xn N<br>R 22 50/53 | | | |
| | | Min | Max | Unit |
| Physico-chemical properties | Water solubility<br>Log $K_{ow}$<br>Henry's law con-stant<br>Vapour pressure | 0.015[1]<br>5.7[2]<br><br>0.068[3] | 0.22[2]<br>6.6[1]<br><br> | mg/l<br><br><br>Pa |
| | | | | |
| Ecotoxicology aquatic | $EC_{50}$ and $LC_{50}$<br>NOEC and LOEC<br>PNECaqua[6]<br>PNECsediment[6] | 0.28[3] | 255.2[3]<br>0.14[3]<br>0.00035[6]<br>0.2[6] | mg/l<br>mg/l<br>mg/l<br>mg/kg |
| Ecotoxicology terrestrial | $EC_{50}$ and $LC_{50}$<br>NOEC and LOEC | 0.28[3]<br>105[3] | <br>25.5[3] | mg/kg soil<br>mg/kg soil |
| Toxicology | Oral $LD_{50}$ 570 mg/kg; dermal $LD_{50}$ >5000 mg/kg[4] | | | |
| Persistence, Bioaccumulation, Toxicity (PBT) | Not PBT (Pbt)[6] | | | |
| Theoretical removal in STP | 82 % of daily load[5] | | | |

[1] http://www.thegoodscentscompany.com/msds/md102487.html

[2] http://www.norden.org/pub/miljo/miljo/sk/TN2004503.pdf

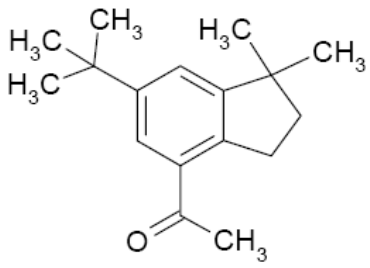
[3] http://jhs.pharm.or.jp/52(3)/52_276.pdf

[4] HERA 2004c

[5] http://www.heraproject.com/files/28-E-36551E10-F8EF-E807-E4199B9BB0076A9F.pdf

[6] Marine environment. OSPAR 2004.

*Table 1.7 Physiochemical and (Eco)toxicological properties of Celestolide.*

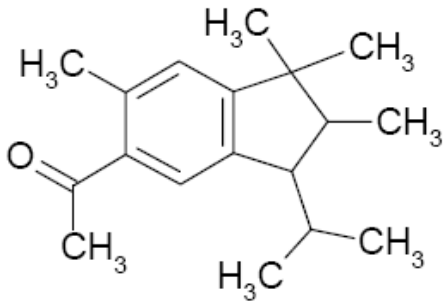
| Common name | **Celestolide (ADBI)** | | | |
|---|---|---|---|---|
| |  | | | |
| Name | 4-Acetytl-1,1-dimethyl-6-tertbutyldihydroindene | | | |
| CAS # | 13171-00-1 | | | |
| Labelling | Xi[1] | | | |
| | | Min | Max | Unit |
| Physico-chemical properties | Water solubility | 0.015[1] | 0.22[2] | mg/l |
| | Log $K_{ow}$ | 5.9[2] | 6.6[1] | |
| | Henry's law constant | | | |
| | Organic carbon-water partition coefficient (Koc) | 4.47[3] | 4.47[3] | |
| | | | | |
| Ecotoxicology aquatic | $EC_{50}$ and $LC_{50}$ | | | mg/l |
| | NOEC and LOEC | | | mg/l |
| Ecotoxicology terrestrial | $EC_{50}$ and $LC_{50}$ | | | mg/kg soil |
| | NOEC and LOEC | | | mg/kg soil |
| Toxicology | | | | |
| Persistence, Bioaccumulation, Toxicity (PBT) | | | | |
| Degradation | No mineralisation under standard test conditions: not inherently biodegradable MITI II, <1 % biodegradation after 28 days[3] | | | |

[1] http://www.thegoodscentscompany.com/data/rw1007691.html

[2] http://www.norden.org/pub/miljo/miljo/sk/TN2004503.pdf

[3] http://www.sea.eawag.ch/inhalt/sites/stoffe/pdf/PMV_e.pdf

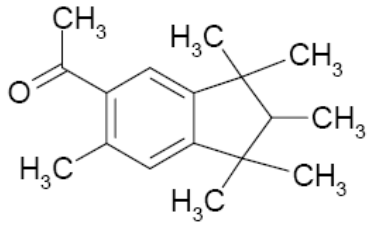
*Table 1.8 Physiochemical and (Eco)toxicological properties of traseolide.*

| Common name | **Traseolide (ATII)** | | | |
|---|---|---|---|---|
|  | | | | |
| Name | 5-Acetyl-1,1,2,6-tetramethyl-3-isopropyl-dihydroindene | | | |
| CAS # | 68140-48-7 | | | |
| Labelling | Xi – Irritant1 | | | |
| | | Min | Max | Unit |
| Physico-chemical properties | Water solubility | 0.085[1] | 0.09[2] | mg/l |
| | Log $K_{ow}$ | 6.14[1] | 6.3[2] | |
| | Henry's law constant | | | |
| | Vapour pressure | | | Pa |
| | | | | |
| Ecotoxicology aquatic | $EC_{50}$ and $LC_{50}$ | | | mg/l |
| | NOEC and LOEC | | | mg/l |
| Ecotoxicology terrestrial | $EC_{50}$ and $LC_{50}$ | | | mg/kg soil |
| | NOEC and LOEC | | | mg/kg soil |
| Toxicology | | | | |
| Persistence, Bioaccumulation, Toxicity (PBT) | | | | |
| Theoretical removal in STP | | | | |

[1] http://www.thegoodscentscompany.com/data/rw1023771.html

[2] http://www.norden.org/pub/miljo/miljo/sk/TN2004503.pdf

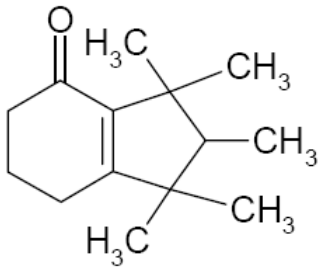
*Table 1.9 Physiochemical and (Eco)toxicological properties of Phantolide.*

| Common name | **Phantolide (AHDI)** | | | |
|---|---|---|---|---|
|  | | | | |
| Name | 6-Acetyl-1,1,2,3,3,5-hexamethyldihydroindene | | | |
| CAS # | 15323-35-0 | | | |
| Labelling | Xi[1] | | | |
| | | Min | Max | Unit |
| Physico-chemical properties | Water solubility | 0.027[1] | | mg/l |
| | Log $K_{ow}$ | 5.8[1] | 5.85[2] | |
| | Henry's law constant | | | |
| | Vapour pressure | | | Pa |
| | | | | |
| Ecotoxicology aquatic | $EC_{50}$ and $LC_{50}$ | 0.33[2] | | mg/l |
| | NOEC and LOEC | 0.044[2] | 0.90[2] | mg/l |
| Ecotoxicology terrestrial | $EC_{50}$ and $LC_{50}$ | | | mg/kg soil |
| | NOEC and LOEC | | | mg/kg soil |
| Toxicology | | | | |
| Persistence, Bioaccumulation, Toxicity (PBT) | | | | |
| Degradation | No mineralization under standard test conditions (low mineralisation, ISO 10708)[2] | | | |

[1] http://www.thegoodscentscompany.com/data/rw1007691.html

[2] http://www.sea.eawag.ch/inhalt/sites/stoffe/pdf/PMV_e.pdf

*Table 1.10 Physiochemical and (Eco)toxicological properties of Cashmeran..*

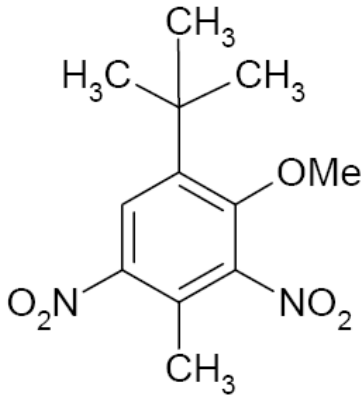
| Common name | **Cashmeran (DPMI)** | | | |
|---|---|---|---|---|
|  | | | | |
| Name | 6,7-Dihydro-1,1,2,3,3-pentamethyl-4(5H)indanone | | | |
| CAS # | 33704-61-9 | | | |
| Labelling | Xi, R 36/37/38, S 26, S 36[3] | | | |
| | | Min | Max | Unit |
| Physico-chemical properties | Water solubility | 0.17[1] | | mg/l |
| | Log $K_{ow}$ | 4.62[1] | 4.9[2] | |
| | Henry's law constant | | | |
| | Vapour pressure | 5.2[2] | | Pa |
| | | | | |
| Ecotoxicology aquatic | $EC_{50}$ and $LC_{50}$ | | | mg/l |
| | NOEC and LOEC | | | mg/l |
| Ecotoxicology terrestrial | $EC_{50}$ and $LC_{50}$ | | | mg/kg soil |
| | NOEC and LOEC | - | - | mg/kg soil |
| Toxicology | Oral $LD_{50}$ =2900 mg/kg[1]. | | | |
| Persistence, Bioaccumulation, Toxicity (PBT) | | | | |
| Theoretical removal in STP | | | | |

[1] http://www.thegoodscentscompany.com/data/rw1024091.html

[2] Chen et al. (2007)

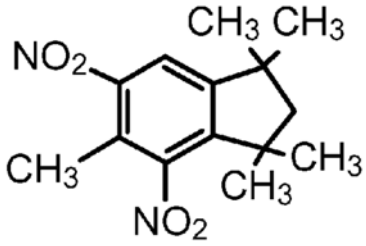
[3] http://www.thegoodscentscompany.com/msds/md103295.html

*Table 1.11 Physiochemical and (Eco)toxicological properties of Musk Ambrette.*

| Common name | **Musk ambrette (MA)** | | | |
|---|---|---|---|---|
|  | | | | |
| Name | 1-tert-Butyl-2-methoxy-4-methyl-3,5-dinitrobenzene | | | |
| CAS # | 83-66-9 | | | |
| Labelling | | | | |
| | | Min | Max | Unit |
| Physico-chemical properties | Water solubility | | | mg/l |
| | Log $K_{ow}$ | 3.71[1] | | |
| | Henry's law constant | | | |
| | Vapour pressure | | | Pa |
| | | | | |
| Ecotoxicology aquatic | $EC_{50}$ and $LC_{50}$ | | | mg/l |
| | NOEC and LOEC | | | mg/l |
| Ecotoxicology terrestrial | $EC_{50}$ and $LC_{50}$ | | | mg/kg soil |
| | NOEC and LOEC | | | mg/kg soil |
| Toxicology | | | | |
| Persistence, Bioaccumulation, Toxicity (PBT) | | | | |
| Theoretical removal in STP | | | | |

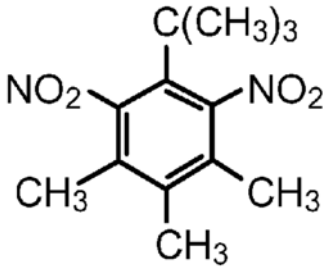
[1] http://www.thegoodscentscompany.com/data/rw1008671..html

*Table 1.12 Physiochemical and (Eco)toxicological properties of Musk Moskene.*

| Common name | **Musk moskene (MM)** | | | |
|---|---|---|---|---|
|  | | | | |
| Name | 4,6-dinitro-1,1,3,3, 5-pentamethylindane | | | |
| CAS # | 116-66-5 | | | |
| Labelling | | | | |
| | | Min | Max | Unit |
| Physico-chemical properties | Water solubility | 0.046[1] | 0.046[1] | mg/l |
| | Log $K_{ow}$ | 5.3[1] | 5.3[1] | |
| | Henry's law constant | | | |
| | Vapour pressure | | | Pa |
| | | | | |
| Ecotoxicology aquatic | $EC_{50}$ and $LC_{50}$ | | | mg/l |
| | NOEC and LOEC | | | mg/l |
| Ecotoxicology terrestrial | $EC_{50}$ and $LC_{50}$ | | | mg/kg soil |
| | NOEC and LOEC | | | mg/kg soil |
| Toxicology | | | | |
| Persistence, Bioaccumulation, Toxicity (PBT) | | | | |
| Theoretical removal in STP | | | | |

[1] OSPAR 2004

*Table 1.13 Physiochemical and (Eco)toxicological properties of musk tibetene.*

| Common name | **Musk tibetene (MM)** | | | |
|---|---|---|---|---|
|  | | | | |
| Name | 1-tert-butyl-2,6-dinitro-3,4,5-trimethylbenzene | | | |
| CAS # | 145-39-1 | | | |
| Labelling | | | | |
| | | Min | Max | Unit |
| Physico-chemical properties | Water solubility | 0.052[1] | 0.052[1] | mg/l |
| | Log $K_{ow}$ | 5.0[1] | 5.0[1] | |
| | Henry's law constant | | | |
| | Vapour pressure | | | Pa |
| | | | | |
| Ecotoxicology aquatic | $EC_{50}$ and $LC_{50}$ | | | mg/l |
| | NOEC and LOEC | | | mg/l |
| Ecotoxicology terrestrial | $EC_{50}$ and $LC_{50}$ | | | mg/kg soil |
| | NOEC and LOEC | | | mg/kg soil |
| Toxicology | | | | |
| Persistence, Bioaccumulation, Toxicity (PBT) | | | | |
| Theoretical removal in STP | | | | |

[1] OSPAR 2004

# 2 Methods

## 2.1 Sampling Strategy

A national sampling strategy was devised based on two objectives:

- Elucidate the main sources of these substances to the environment
- Elucidate the levels of these substances in the environment

The most important sources within the national screening were:

Waste water treatment plants which according to a large number of studies are the main source of musk compounds to the environment

Diffuse sources. Since these substances are widespread in society, sites generally affected by urban load were also investigated.

Landfills receiving household and industrial waste. Given that some of the substances are persistent and common in many products this was deemed a relevant sampling matrix.

Households / detached houses and villa gardens where a number of products containing musk compound are used.

The different matrices chosen and types of sampling points is presented in table 2.1.

Environmental background levels in fish soils and sediments were determined in samples from background reference lakes where the influence from human activities are considered to be minimal; Lake Abiksojaure in the northernmost part of Sweden, Ljusacksen in the middle part of Sweden and Krageholmsjön in the southernmost part of Sweden (Figure 2.1). Soil was also sampled from the areas around these lakes.


*Figure 2.1 Background sampling stations*

*Table 2.1 Sampling matrices and the types of sources investigated for musk compounds. The first value denotes samples taken within the national screening programme. The second value (after the slash) denotes samples financed by the regional screening programme.*

| Sampling matrices musk substances | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| **Sources** | Soil | sludge | Incoming water | Outgoing water | Surface water | Runn-off water | Ground water | Fish | Sediment | Human blood |
| background | 3 / 0 | | | | | | | 2 / 0 | 3 / 0 | |
| Diffuse sources | 1 / 0 | | | | 2 / 1 | | | 3 / 1 | 3 / 1 | 10 / 0 |
| Waste water treatment plants | | 3 / 14 | 4 / 5 | 3 / 22 | | | | | | |
| At locations influenced by the following sources | | | | | | | | | | |
| a. Waste water treatment plants | | | | | 2 / 2 | | | 3 / 2 | 3 / 1 | |
| b. Industries | | 0 / 1 | | 0 / 1 | | 2 / 0 | 1 / 0 | | 3 / 0 | |
| c. Households | 2 / 0 | | | | | 4 / 0 | 3 / 0 | | | |
| d. Landfills | | | | | | 3 / 0 | | 1 / 0 | | |
| Total | 6 / 0 | 3 / 15 | 4 / 5 | 3 / 23 | 4 / 3 | 9 / 0 | 4 / 0 | 9 / 3 | 12 / 1 | 10 / 0 |
| Total | 64 / 47 | | | | | | | | | |

## 2.2 Sampling methods

Sampling instructions were given to all sampling personnel. The instructions included sampling procedures and handling of samples during transport.

### 2.2.1 Soil

Soil was sampled from the topmost layer after the removal of dead and living plant parts. Also, stones and larger objects were avoided. Soil samples were collected into diffusion proof clean sampling plastic bags and sent to the laboratory within a day of sampling. Samples were kept cold until analysis.

### 2.2.2 Sediment

Sediment samples were collected by means of a core sampler. All sediment samples were transferred to pre-burned and dark glass jars and sent to laboratory within one or two days of collection. They were stored cold until analysis.

### 2.2.3 Sewage Treatment Plant (STP) sludge and water

The staff at the sewage treatment plants collected the sludge samples and water samples in acid rinsed pre burned dark glass bottles. All STP samples were sent to the laboratory within one or two days of collection. They were stored cold until analysis.

### 2.2.4 Fish

Samples from Abisakojaure (Arctic char, *Salvelinus alpinus)* and Krageholmssjön (perch, *Perca fluviatilis*) were supplied from the Environmental Specimen Bank at the Museum of Natural history (A. Bignert and colleagues). Fish from Ljusacksen (perch, *Perca fluviatilis*) and all other fish were collected using fishing net. All fish samples were stored frozen until analysis.

### 2.2.5 Water

Unfiltrated water was collected in clean in acid rinsed pre-burned dark glass bottles. Water samples were stored cold until analysis.

## 2.3 Analytical methods

A mixture of polar solvents was used for extraction, both from water samples and from solid matrices. Soil, sediment and sludge samples were cleaned from interfering compounds using solid phase extraction. 3 musk substances were used as internal standards and all analyzed musk substances were used as external standards. Gas chromatography was used for chromatographic separation and mass spectrometry was used for peak/substance identification.

# 3 Results

The results are summarized in figure 3.2 – 3.5. Figure 3.1 explains the statistics that is presented in figure 3.2 – 3.5.

Figure 3.2 present the result for those musk substances that were only found in sludge. Figure 3.3 presents levels of Galaxolide in a number of compartments. The levels of Galaxolide in surface water are from samples downstream of waste water treatment plants. Figure 3.4 presents levels of Tonalide in a number of compartments. The levels of Tonalide in surface water are from samples downstream of waste water treatment plants. Figure 3.5 presents levels of Musk Ketone in a number of compartments.

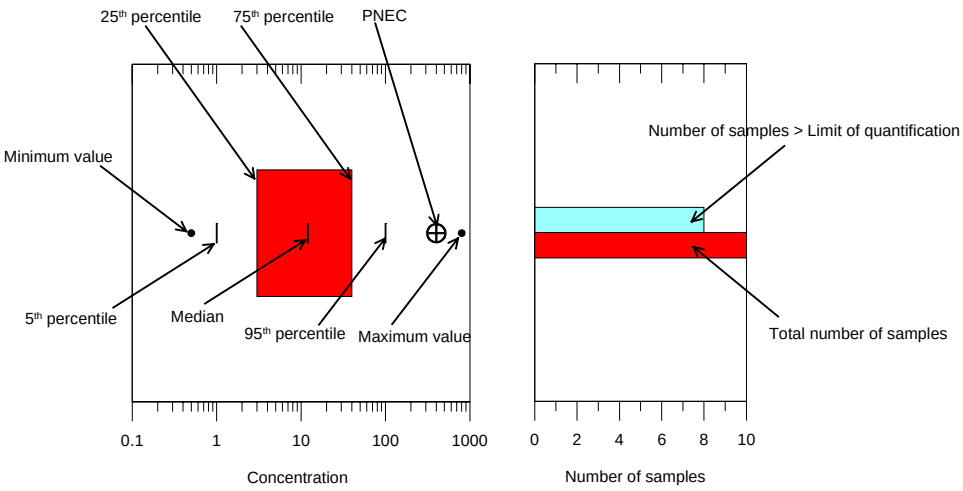



*Figure 3.1* Explanation of graphs summarizing the results from the sampling of musk substances.

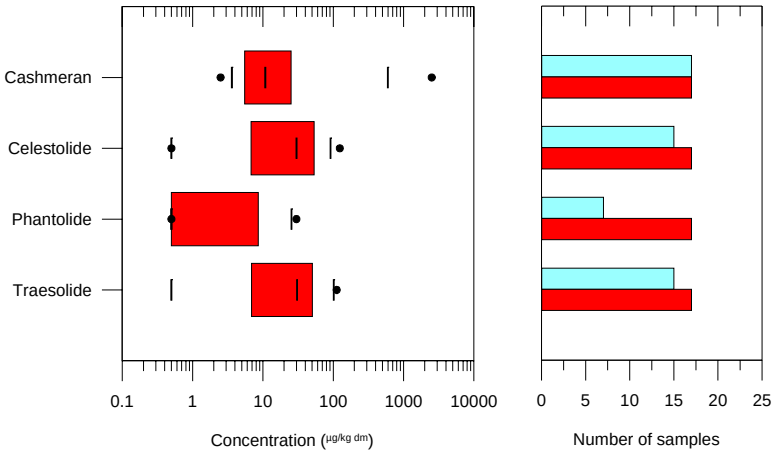



*Figure 3.2 Levels of musk substances that were only found in the sludge samples. See figure 3.1 for explanation of the graph.*

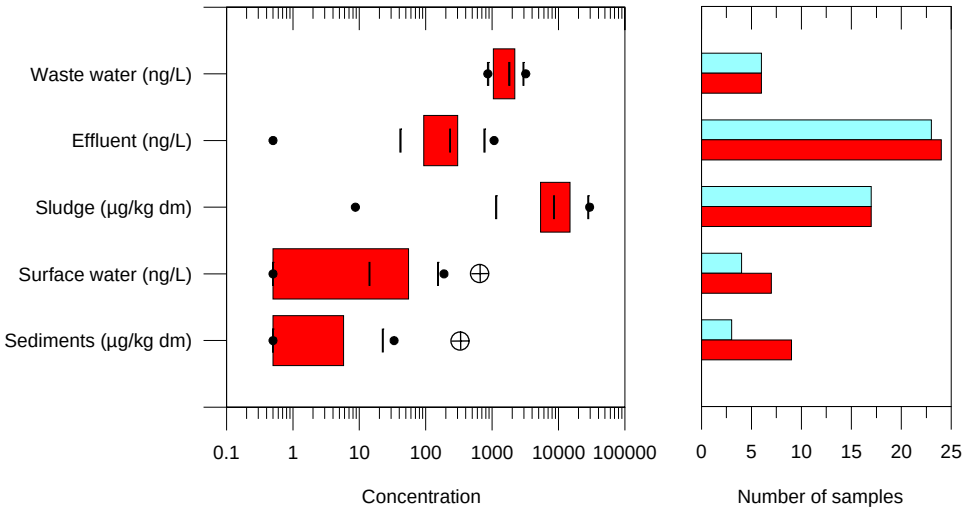



*Figure 3.3 Levels of Galaxolide in different compartments. See figure 3.1 for explanation of the graph.*

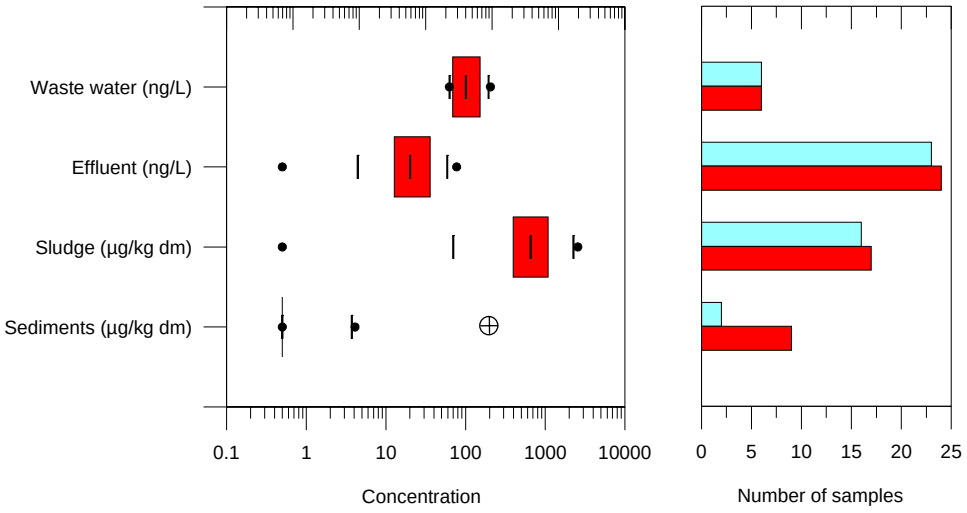



*Figure 3.4 Levels of Tonalide in different compartments. See figure 3.1 for explanation of the graph.*

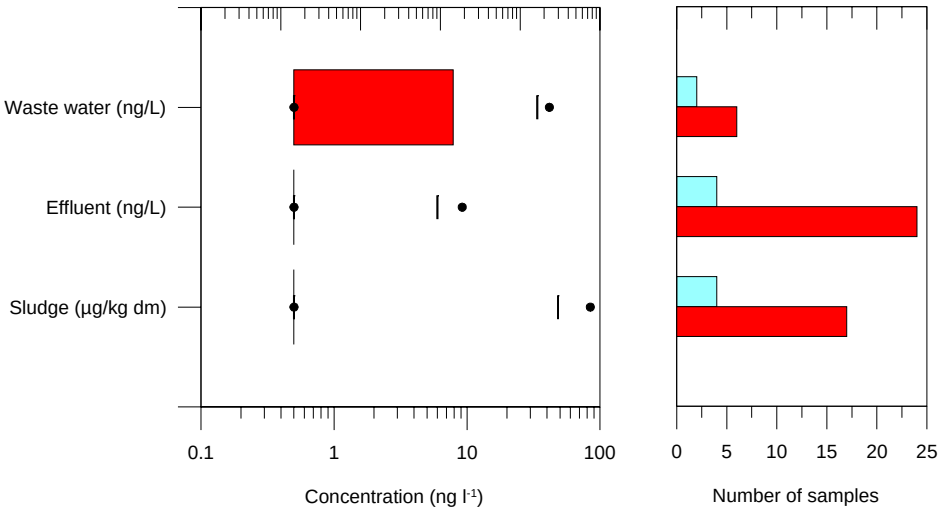



*Figure 3.5 Levels of Musk Ketone in different compartments. See figure 3.1 for explanation of the graph.*

# 4 Discussion

Musk compounds were not found at background localities. This indicates a lack of (atmospheric) transport of musk substances to remote areas. This corroborates with a Nordic screening study where Musk compounds, with a few exceptions, were generally not detected in rainwater (TemaNord 2004). On the other hand, there are studies demonstrating that musk substances are present at remote localities. In studies performed 1990 – 1995, Musk Ketone, Musk Xylene, Tonalide and Galaxolide was found in water sampled from a depth of 7 - 10 m in the North Sea at concentrations of 0.08 ng/l, 0.17 ng/l, 0.26 ng/l and 0.2 ng /l (no other depths were sampled) (TemaNord 2004). A major difference between the remote localities in this study and the North Sea is that the North Sea is directly influenced by sewage treatment plants which are important sources of musk substances.

In this study, no musk compounds were found in association with diffuse urban load, landfills and households. Consequently, these sources are most likely not important for musk compounds. Previous monitoring studies in various countries have not investigated these types of sources (European Union 2005a ,2005b, OSPAR 2004, TemaNord 2004, HERA 2004a) and the results from this study does not indicate that such monitoring is necessary in the future.

All sewage sludge samples analyzed within this study contained musk compounds (Table 4.1) which corroborates with a number of other studies where musk compounds have been found to be very common in sludge (European Union 2005a, 2005b, Kallenborn et al. 1999 OSPAR 2004, TemaNord 2004, Hera 2004a). The sludge usually contained a mixture of all measured polycyclic musk compounds while Musk Ketone was the only nitro musk compound that occurred (Table 4.1). This is in accordance with the fact that nitro musks are being replaced by polycyclic musks since the latter have been associated with risks to consumers (OSPAR 2004, HERA 2004a).

The levels of musk compounds in sludge in a Nordic screening study are at par with the levels in the present study (Table 4.1). The major difference is that Musk Ketone was detected in the present study but not in the Nordic screening study and, that Cashmeran was detected at levels 30 times higher than in the Nordic screening study. The high levels of Cashmeran occurred at one sewage treatment plant and the reason for this is unknown.

As found in earlier studies, the concentrations of musk compound were highly variable in the sludge samples (Figure 3.3 – 3.5). The reason for the high variability is unknown. There are no studies that have attempted to relate the concentrations of musk compounds in the sludge to specific properties of the waste water treatment

plant. Such properties could be size and operations parameters of the plant itself and the characteristics of operations that are responsible for the incoming water. This could be of interest to elucidate in the future.

*Table 4.1 Concentrations of different musk compounds in sludge in the present study and in a Nordic screening study (TemaNord 2004).*

| | Sludge (µg/kg) in Nordic screening study (TemaNord 2004) | | | | | Sludge (µg/kg) in the present screening study |
|---|---|---|---|---|---|---|
| | Denmark | Finland | Iceland | Norway | Sweden | Sweden |
| | | | | **Aromatic nitro musks** | | |
| Musk Xylene (MX) | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. (<1) |
| Musk Ketone (MK) | n.d. | n.d. | n.d. | n.d. | n.d. | 2 – 84.5 |
| Musk Ambrette (MA) | 35 - 83 | n.d. | n.d. | n.d. | n.d. | n.d. |
| | | | | **Polycyclic musk compounds** | | |
| Galaxolide (HHCB) | 11400 - 26500 | 495 - 8750 | 408 - 2160 | 254 - 22400 | 7800 - 24000 | 1430 – 29400 |
| Tonalide (AHTN) | 1130- 3610 | 97 - 2270 | 98 - 553 | 68 - 3500 | 950 - 3350 | 88 – 2600 |
| Celestolide (ADBI) | 63 - 294 | 55 - 66 | 167 - 175 | 50 - 170 | 52 - 153 | 6.4 – 124 |
| Phantolide (AHDI) | <12 - 24 | 14 - 55 | n.d. | 12 - 64 | 12 - 33 | 2.9 – 30 |
| Traesolide (ATII) | 252 - 891 | 46 - 150 | n.d. | 99 - 601 | 38 - 252 | 4 – 99 |
| Cashmeran (DPMI) | 25 - 54 | 12 - 41 | n.d. | 100 - 128 | 12 - 17 | 2.8 – 3510 |

The polycyclic musk substances found at the lowest concentrations in sludge (Celestolide, Phantolide, Traesolide, Cashmeran, figure 3.2) did not occur in any other matrices. These substances has been found in sludge but only rarely in outgoing water in earlier studies (Kanda et al. 2003, OSPAR 2004), showing that they most likely are removed to a large degree in the sewage treatment works, either through incorporation into the sludge or through biodegradation.

The reason that a number of musk substances were found in sludge but not in incoming water is either that they occurred below the level of quantification in the incoming water (1 ng/l) or that the levels seen in sludge are the result of historical usage. Previous studies have focused more on sludge and outgoing water but one study has

investigated some of these substances in incoming water also (Table 4.2, Kanda et al. 2003).

*Table 4.2 Levels of several polycyclic musk compounds in incoming waters in the United Kingdom sampled 2001. Source: Kanda et al. 2003.*

| substance | Instances above LOQ (10 ng/l) | Concentration range (ng/l) |
|---|---|---|
| Cashmeran | 1 of 14 | 400 |
| Celestolide | 1 of 14 | 440 |
| Phantolide | 5 of 14 | 30 - 100 |
| Traesolide | 2 of 14 | 1700 - 2900 |

Galaxolide and Tonalide occurred in incoming water (waste water), sludge and effluents. They occasionally occurred in surface water and sediments downstream of the sewage treatment plants, but not in other surface waters and sediments (Figure 3.3 – 3.4). This demonstrates that sewage treatment plants are the major source of these substances to the environment. This has also been the case in studies from other countries (TemaNord 2004, OSPAR 2004, Kanda et al. 2003, HERA 2004b-c).

The reason that Galaxolide and Tonalide occurs in compartments downstream of sewage treatment plants is a combination of a high usage (section 1.3.1) and a suboptimal removal in sewage treatment plants (Table 4.3). Simonich et al. (2002) related the degree of removal of Galaxolide and Tonalide in wastewater treatment mainly to the degree of removal of total suspended solids which correlates well with the high levels of these substances found in sludge (Table 4.1).

*Table 4.3 Degree of removal of musk substances in sewage treatment plants in Europe and USA. Source: Simonich et al. (2002), Osemwengie and Gerstenbergerb (2004). Certain types of sewage treatment plants were excluded since they are only used in USA.*

| substance | Mimimum removal | Maximum removal |
|---|---|---|
| Galaxolide | 39% | 94% |
| Tonalide | 57% | 96% |
| Musk Xylene | 89% | 98% |
| Musk Ketone | 85% | 91% |

Nitro musks are much less used than polycyclic musk substances (section 1.3.1). This may explain why Musk Ketone, which is the most common Nitro Musk, was the only one that occurred in other compartments than sludge (Figure 3.5). The very low levels in effluent water is most likely a result of the relatively low amounts used, the low concentrations in sludge and a high degree of removal in sewage treatment plants (Table 4.3).

The question remains whether the concentrations in recipient surface waters and sediments are of any environmental concern. A risk comparison between the maximum measured concentrations of Galaxolide and Tonalide in surface waters and sediments and the predicted no effect concentrations (PNEC) of these substances, results in a maximum risk ratio is 0.26 for Galaxolide in surface waters (Table 4.4). Although this does not give rise to any immediate concern, the risk ratio is high enough that the environmental effects of some musk substances can not be totally ignored. The risk assessment in table 4.4 is based on a limited number of surface waters and also on one-time samplings. In the future, samplings in a few surface waters at many occasions may be warranted to ascertain that no direct negative effects of these compounds are seen in surface waters.

*Table 4.4 Risk assessment for Galaxolide and Tonalide in surface waters and sediments. PNEC values are given in Table 1.5 and Table 1.6.*

| substance | Maximum measured concentration (MEC) in this study | PNEC | MEC/PNEC ratio |
|---|---|---|---|
| Galaxolide surface water (ng/l) | 180 | 680 | 0.26 |
| Galaxolide sediment (µg/kg) | 33 | 320 | 0.10 |
| Tonalide sediment (µg/kg) | 4 | 200 | 0.02 |

Apart from the ecological risks that are associated with elevated water concentrations, there are also risks associated with the accumulation of musk substances in biota. Neither Galaxolide nor Tonalide are classified as PBT compounds although they are occasionally found in fish (Kallenborn et al. 1999). In this study Galaxolide was found in two of seven perch samples (muscle) in stream receiving discharge from sewage treatment plants at concentrations of 6.7 µg/kg wet weight muscle (690 ng/g lipid) and 48 µg/kg wet weight muscle (4650 µg/g lipid). No musk substances were found in fish from less affected background lakes. The frequency of detection was less than in an earlier study from 2001 where Galaxolide was found in a majority of the perch samples from 5 Swedish Lakes. Other musk compound were also found in the earlier study, including Tonalide in a majority of the perch samples, as well as Traesolide, Musk Ketone and Musk Xylene in fewer perch samples (reported in TemaNord 2004). Since the reporting levels do not differ to a large degree be-

tween the present study and the earlier study these differences may reflect a declining trend of musk substances in Perch or simply a high level of variability of musk levels in Perch.

Levels of organic pollutants and metals in blood from employees at the County Council of Uppsala has been studied (Landstinget i Uppsala Län 2007). Galaxolide was found in a majority of the samples at levels from 0.2 – 1.3 ng / g serum. In the present study, where the same laboratory was used, Galaxolide levels were always below the limit of quantification (1 ng/g blood) in blood samples from mothers that had recently given birth to their first child. Since serum makes up about 50% of the total blood volume this translates to a limit of quantification of 2 ng/g serum in this study. The reason for the higher limit of quantification is due to smaller amounts of blood available in this study. A tentative conclusion from these two studies is that the blood concentrations of Galaxolide in the Swedish population may be in the range of > 0.2 ng/g serum to < 2 ng/g serum.

Sludge that contains musk substances is probably spread on agricultural lands. This may cause human exposure through cops that take up musk substances as well as ecological exposure for organisms living in agricultural soils. This exposure route was not investigated in the present study, but may be the focus of future screening studies since musk compounds are not evaluated when the spreading of sludge is appraised. This issue has very recently come in to focus since a study by the USGS has found a number of typical sludge associated chemicals (including Galaxolide and Tonalide) in Earth Worms from agricultural fields (Kinney et al. 2008).

# 5 Conclusions and recommendations

This and other studies show that musk compounds frequently occur in sewage treatment plants and occasionally in receiving surface waters. Nevertheless, the concentrations found in this study do not constitute any risk, since the MEC/PNEC values are below one. The following recommendations are put forward.

- Musk substances could be measured over time in a few surface waters where the effluent from sewage treatment plants is of quantitative importance for the total flow. The purpose would be to ascertain that risk levels are never exceeded even under worst-case conditions.
- Screening of musk substances in soil and biota where sludge has been applied to agricultural soils could also be of interest.
- A mass balance study of the amounts of different musk substances reaching waste water treatment plants, the amounts stored in sludge and the amounts reaching surface waters would be of interest to elucidate the magnitude of the environmental problems with musk substances.
- A study of how operating parameters in the sewage treatment plants, as well as sources of incoming water, affect the levels of musk substances in sludge and outgoing water could also be of interest. The purpose would be to better understand why these substances occur in the downstream recipients.

- Finally, the issue of musk compound metabolites needs to be addressed. Metabolites of the nitro musk compounds Musk Xylene and Musk Ketone have been found in levels higher or much higher than the parent compounds in sludge and effluents of municipal sewage plants and in water samples from rivers in Germany and USA (Rimkus et al. 1999, Osemwengie and Gerstenbergerb 2004). Also, the transformation products of Musk Xylene and Musk Ketone frequently occurred in aquatic biota samples (Rimkus et al. 1999, Osemwengie and Gerstenbergerb 2004) although this may be the result of transformation within the organism as well as intake of the metabolites.

# 6 References

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