Screening of N,N-dietyl-m-toluamid (DEET)

SWECO Environment Screening Report

Client

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SWECO

Content

Sammanfattning

Bakgrund och metoder
Inom den nationella screeningen 2010 har SWECO Environment på uppdrag av Naturvårdsverket undersökt förekomsten av dietyltoluamid (DEET) som är den aktiva substansen i många repellerande insektsmedel. Screeningprogrammen syftar till att öka kunskapen om utsläpp, förekomst och spridning av kemikalier i miljön. Naturvårdsverket huvudfinansierar programmen samt väljer ut vilka ämnen som skall studeras. Länsstyrelserna har också möjlighet att vara med och komplimentera med regional provtagning.

DEET är en av de vanligaste aktiva substanserna i produkter av typen spray, stick, servetter m.m. som appliceras på huden för att undvika myggbett och fästingbett. Välkända svenska varumärken, bland de 12 godkända produkter som finns registrerade i Kemikalieinspektionens Bekämpningsmedelsregister, är MyggA, US 622 och Djungelolja. EU-direktiv begränsar innehållet av DEET till maximalt 20 % i dessa produkter. De senaste 10 åren har användningen av DEET som biocid legat på mellan 4 och 6 ton årligen i Sverige. DEET har använts under lång tid och är av EU klassat som lätt nedbrytbart med hänvisning till standardiserade tester. Samtidigt finns det motstridiga uppgifter som pekar på att DEET inte är lätt nedbrytbart i t.ex. reningsverk. Internationella studier har påvisat DEET i avloppsvatten, ytvatten, havsvatten, dricksvatten och i en Europeisk studie påträffades DEET i 84 % av de insamlade grundvattenproverna. Dessa fynd föranledde den aktuella nationella undersökningen.

Den huvudsakliga användningen av DEET tros ske i hushållen. Även om personer använder myggmedel utomhus så kommer ämnet att hamna i avloppsvattnet när personen tvättar sig. Därför har provtagning skett av inkommande och utgående avloppsvatten samt avloppsslam vid reningsverk. Även recepientmiljöer till reningsverk har kartlagts genom provtagning av vatten och sediment. Ytvatten och sediment har provtagits i bakgrundsmiljöer samt generellt urbant påverkade vatten. Grundvatten har provtagits dels från stadsmiljöer och dels från välkänt myggtäta områden. Grundvattenprovtagningen var motiverad med den Europeiska studien i åtanke. Med tanke på den utbredda användningen av myggmedel sommartid har även badvatten och sediment från badplatser inkluderats i studien. De länsstyrelser som har bidragit ekonomiskt till den regionala förtätningen av den nationella provtagningen är Dalarna, Värmland och Södermanland. Sammanlagt togs 40 vattenprover, 4 slamprover och 4 sedimentprover. I samtliga prov har även alkylfenoler kvantifierats för att erhålla ett mått på graden av urban belastning vid den aktuella mätpunkten.

Slutsats
Den här studien visar i överensstämmelse med tidigare internationella undersökningar att DEET är vanligt förekommande i vatten i reningsverk, i ytvatten nedströms reningsverk samt i grundvatten. Dessutom förekommer DEET i ytvatten och i sediment där människor badar. Kemikalien påträffas inte i avloppsslam och ej heller i ytvatten och sediment från en bakgrundsmiljö eller i diffust urbant påverkad miljö som inte är direkt påverkade av reningsverk eller bad.

De viktigaste slutsatserna från studien är att:

- Resultaten visar att DEET var vanligt förekommande i inkommande och utgående avloppsvatten. Halterna var generellt lägre än i jämförbara studier från utlandet med ett medianvärde av 310 och 101 ng/L i inkommande respektive utgående vatten. Utifrån de individuellt uppmätta koncentrationerna i reningsverkens avloppsvatten uppskattades att 35 % av inkommande DEET bryts ned i reningsverket. Den teoretiska modellering som gjordes med en EPA modell uppskattar nedbrytningseffektivitet till 20 % i reningsverk.
- DEET var också vanligt förekommande i recepientvatten nedströms reningsverk vilket överensstämmer med tidigare utländska studier.
- DEET påträffades inte i avloppsslam vilket inte heller förväntas med tanke på ämnets fysiokemiska egenskaper. DEET påträffades inte heller i några sedimentprover nedströms reningsverk eller i bakgrundsmiljöer.
- DEET påträffades i grundvattenprover från stadsmiljö men inte i grundvatten som användes för dricksvattenproduktion. Totalt detekterades DEET i 67 % av grundvattenproverna från norra och södra Sverige. De uppmätta halterna var strax över 90-percentilen jämfört med en tidigare Europeisk studie som delvis motiverade föreliggande undersökning. En trolig förklaring till högre halter kan vara en mera utbredd användning av myggmedel i Sverige i allmänhet. I andra länder har även DEET påträffats i dricksvatten vilket kan bero på att dessa vattenkällor är påverkade av vatten från reningsverk.
- DEET uppmättes i ytvatten och i sediment på badplatser.
- DEET påträffades inte i ytvatten från en bakgrundslokal.
- En riskbedömning baserad på tillgänglig information om ekotoxikologiska effekter samt uppmätta halterna i recipienter, visade att DEET inte utgör någon ekotoxikologisk risk för den akvatiska miljön. ..

Riskbedömningen tar ingen hänsyn till övriga polära organiska kemikalier som förekommer i Europeiska ytvatten (Loos et al. 2009). Följaktligen har riskbedömningen inte tagit hänsyn till eventuella samverkanseffekter med dessa kemikalier. Det saknas även information kring kroniska effekthalter för DEET och det finns en osäkerhet i att endast använda data kring akuttoxiska halter även om användandet av säkerhetsfaktorer vid framtagande av nolleffektkoncentration (PNEC) till viss del åtgärdar detta problem. En slutsats blir därför att mera information kring ekologiska effekter behövs för att kunna göra en korrekt utvärdering av faran med de förekommande halterna av DEET i vattenmiljön. Det behövs även mera information om DEETs nedbrytningsprodukter och dessa ämnens eventuella toxiska egenskaper.

Rekommendationer

- Ingen ytterligare screening undersökning av DEET är nödvändig i nuläget eftersom miljö-koncentrationerena är långt under effektkoncentrationerna.
- Den här bedömningen bör omprövas när mer data finns tillgänglig för långtidseffekter av DEET eller om samverkanseffekter med andra kemikalier upptäcks.
- Även ny kunskap kring förekomst och effekt av nedbrytningsprodukter till DEET kan föranleda en ny utvärdering.

Summary

Background and methods
In 2010, SWECO Environment performed a screening study of DEET in a number of matrices and at one background locality, fincanced by the Swedish Environmental Protection Agency. The project was initiated because trans-national and national studies had shown a high prevalence of DEET in both surface waters and ground waters in Europe, USA, and Australia.

The objectives of the project were to elucidate the levels of DEET in sewage treatment plants (STPs), in downstream limnic environments and in groundwater. The study also aimed at briefly assessing whether the levels constitute an environmental 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 and sediments in streams receiving effluents from sewage treatments plants. Groundwater from northern Sweden where the usage of mosquito repellents is prevalent as well as from southern Sweden was also included. Finally, surface waters impacted by urban activities in general and bathing activities were also sampled.

Alkyl phenols were also analysed in all of these samples as substances representing anthropogenic influence in general. This was done to support the evaluation since it is believed that the levels of DEET roughly correlate to anthropogenic influence.

To summarize, this and other studies show that DEET frequently occur in water in STPs, surface waters downstream of STPs and in groundwater in Sweden. In addition, DEET was found in water and sediments at recreational bathing sites. The results demonstrate that DEET does not occur in sewage sludge or in surface waters and sediments that are not influenced by STPs.

<u>More detailed conclusions were:</u>

- DEET was common in both incoming and outgoing waste water in STPs in accordance to theoretical predictions and previous studies
- DEET was very prevalent in watercourses downstream of STPs in accordance with results from previous monitoring studies.
- The levels of DEET in STPs and downstream the plants in this study were generally lower than what has been found in other countries.
- DEET was found in groundwater from areas of northern Sweden where mosquitoes are ubiquitous but also in southern Sweden. The levels may be higher than European levels in general.
- DEET was not detected in raw untreated drinking water in the present study which is in contrast to studies from other countries.
- DEET was found in surface water and sediment at recreational bathing sites.

- DEET was not found in a limnic background environment
- The ratio between measured concentrations (MEC) and a predicted no effect concentration (PNEC) based on acute toxicity tests was 0.0046. It is consequently apparent that DEET does not constitute an acute risk to the aquatic environment. The final judgement on risk is however uncertain since only acute toxicological data is available, interactive effects with other compounds is not considered and the levels of degradation products and their toxicity is not taken into consideration.

The final recommendations are:

1. No further screening study of DEET is necessary at present given the indication that the frequently observed concentrations are well below effect concentrations

2. This assessment should be renewed when more data becomes available on:
   a) the long term ecological effects of DEET
   b) toxicological interactions with other compounds that occurs in surface waters
   c) occurrence and effects of DEET degradation products

1 Introduction

1.1 Background

At present there is a lack of knowledge regarding the emission, distribution and exposure to 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 county administrative boards have the option to complement and extend the sampling program by choosing additional sampling points that are of regional interest.

N,N-diethyl-m-toluamide (DEET) is commonly used as the active ingredient in insect repellents. DEET has been detected in drinking water, ocean water, surface water, ground water and sewage water in various parts of the world (Costanzo 2007). Reported concentrations vary between 40 and 3 000 ng/l (Costanza 2007). In a large survey of 59 selected compounds in 164 ground water samples from 23 European countries DEET was the organic pollutant with the highest frequency of detection and the highest concentration was 454ng/L (Loos et al. 2010). The concentration in the six samples from Sweden in this study was between 1.4 and 23 ng/L. DEET has also been detected in leachate water from a Swedish landfill (Öman, 1993). The high prevalence in surface waters does not accord with the fact that only relatively small amounts are used both within the Nordic countries and EU and the fact that DEET is categorized as readily biodegradable (Standing Committee on Biocidal Products 2010).

As a result of these findings the Swedish Environmental Protection Agency has inquired for a follow up on these measurements and a further investigation of the occurrence of DEET in various matrices.

1.2 Objectives

Within the screening program of 2010, SWECO Environment was commissioned by the Swedish EPA to measure the occurrence of DEET in matrices such as surface water, ground water and sewage water.

The objectives of the project were to:

- Elucidate whether DEET occurs in waste water treatment plants, and in receiving surface waters including sewage sludge and surface water sediments
- Supplement the earlier investigation by Loos et al (2010) on the occurrence of DEET in ground water including ground water used as drinking water.
- Elucidate whether DEET is occurring in urban surface waters including sediments in such areas.

- Broadly assess whether DEET constitutes an environmental risk.

1.3 Substance information

1.3.1 Usage

DEET was originally developed by the US Army in 1946 for use by military personnel in insect-infested areas. It is used on humans to repel biting insects and ticks. Products such as sprays, creams, lotions, sticks, foams, and towelettes, containing from 4 to 100% of the active ingredient are applied directly to skin or clothing. There are also some products available for animal use (US EPA ,1998). Within the EU the usage of DEET based insect repellents may be regarded as mainly a way to avoid the annoyance of biting insects but also to some extent as a way to minimise the spreading of Lyme disease (Lyme borreliosis) and meningitis. Outside the DEET based EU insect repellents may be crucial for preventing epidemic disease transmission of Malaria and West Nile virus.

Within the EU the maximum allowed concentration of DEET as active ingredient in products is 20%. There are today 12 products in Sweden that are approved by the Swedish Chemicals Agency (KemI, 2011), including trade names such as Djungelolja II, MyggA, US 622, and Carr & Day & Martin Extra Strength Insect Repellent. The latter of these products is for use on horses. To find out if DEET is much used for horses, telephone interviews where conducted with the Chemicals Agency, personnel at stables and a wholesale company specialized on horses (Höoks). The Chemical Agency claims that products with other active ingredients are more common as insect repellent for horses (KemI, Jenny Karlsson pers. com.). This was also confirmed by the wholesale company who stated that sales of insect repellents for horses are increasing but the most popular products contain permethrin and not DEET as the active component. To conclude, DEET for animal use seems to be of minor significance in comparison to the volumes used in products for humans.

Household consumption of mosquito repellent in Sweden in 2005 was 8 tonnes where most products contained either DEET or p-menthane-3,8-diol as the active component. Total consumption of DEET in Sweden in 2009 was 6.3 ton (KemI stat) and the average annual consumption was 8.6 ton during the years 2000-2009. The corresponding annual usage of DEET as active component was 1800 ton in the year 1990 (US EPA, 1998). Registered volumes, for biocide use and other use, are presented in Figure 1.1.

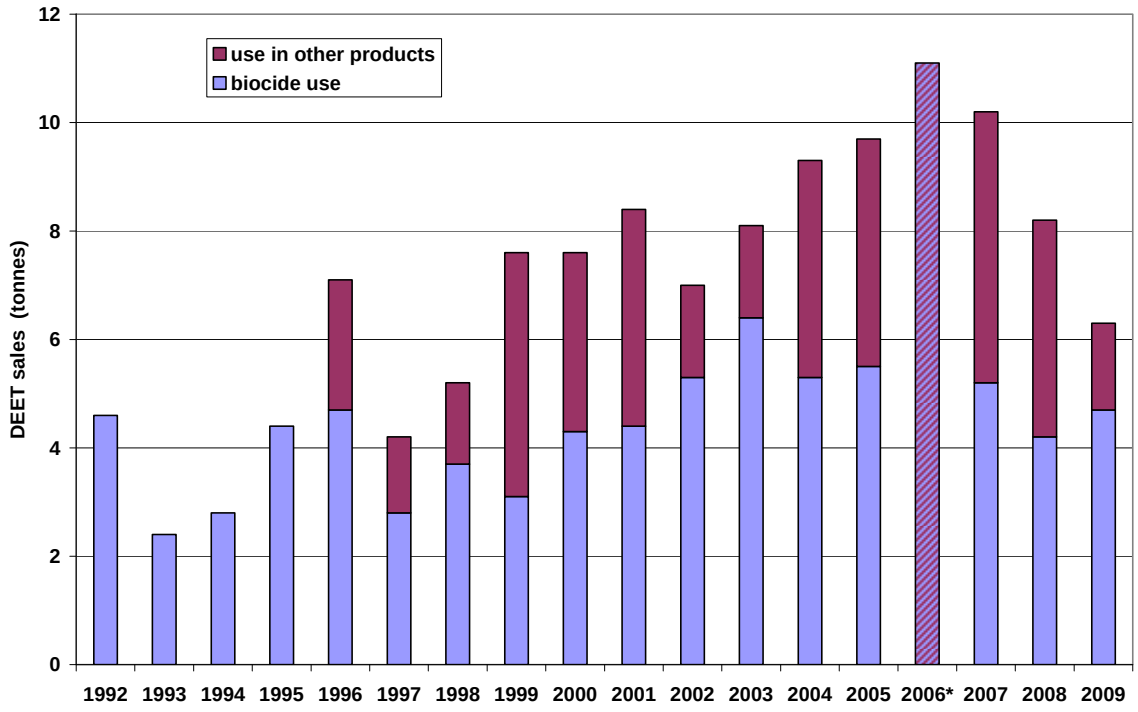

Figure 1.1 Registered sales (tonnes) of DEET (KEMI 2010).

* For the year 2006 only data on total consumption was available.

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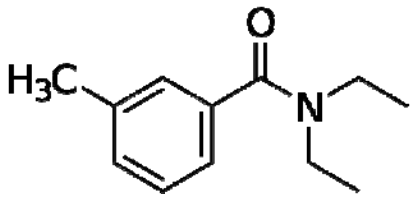
1.3.2 Properties

Table 1.1 presents physical and chemical properties as well as toxicological and ecotoxicological information for DEET. In manufactured pure form the chemical is a clear almost colourless liquid with a mild characteristic odour. The data presented in Table 1.1 suggests that DEET has high water solubility and low degree of volatilization. The log K_{ow} value indicates no potential for DEET to bioaccumulate. DEET is slightly irritating to the skin and clinical signs of neurotoxicity have been shown to occur in dogs after oral dosing (EU 2010).

Table 1.1 Physiochemical and (Eco)toxicological properties of Diethyl toluamide.

Common name	**Diethyl toluamide (DEET)**			
				
Name	N,N-Diethyl-meta-toluamide			
CAS #	134-62-3			
Labelling[3] (Directive 67/548/EEC)	Xn;R22 Xi;R36/38 R52-53 S(2-)61			
		Min	Max	Unit
Physico-chemical properties	Water solubility	<1 at 20°C[1]	11.2[3]	g/L
	Log K_{ow}	2.2[2]		
	Henry's law constant	$2.1X10^{-8}$ [2]		atm-cu m/mole
	Vapour pressure	0.23[3]	7.46[2]	kPa
Ecotoxicology aquatic	LC_{50} 96h fish[1]		72	mg/l
	EC50 algae (*Selenastrum capricornutum*)[2]		43	mg/l
	EC 50 *Daphnia magna*[3]		75	mg/l
	LC50 Rainbow trout [6]		75	mg/l
	$PNEC_{freshwater}$[4]		0.043	mg/l
Toxicology	Oral LD_{50} 2170-3664 mg/kg bw (rat), Inhalation LC_{50} 5.95 mg/l (rat), Dermal LD_{50} 4280 mg/kg bw (rabbit) [6]			
Persistence, Bioaccumulation, Toxicity (PBT)	*P* bT according to PBT profiler. I.e. medium potential for persistence, low potential for bioaccumulation and high toxicity (http://www.pbtprofiler.net/) BCF values of 0.8-2.4, suggests the potential for bioconcentration in aquatic organisms is low [1]			
Theoretical removal in STP	24 %[5]			

[1] Flygfältsbyrån 2009

2 EU 2010

3 ECOTOX database (http://cfpub.epa.gov/ecotox/). Retrieved 2011-03-20

4 EU 2010

[5] Calculated using a fugacity model in EPIWIN; Biodegradation: 21%, sludge adsorption 2%, to air 0%.

[6] US EPA 1988

1.3.3 Biodegradation

There are conflicting data regarding the biodegradability of DEET. In the EU assessment report from 2010 it is suggested that DEET can be considered as readily biodegradable with reference to a reliable study carried out in accordance with OECD TG 301B (EU 2010). On the other hand, there are several reports indicating that DEET is not readily biodegradable in sewage treatment plants (Bernard 2006) or other environmental compartments (Tice et al. 1999, HSDB 2011). Also the Biowin model (http://www.epa.gov/opptintr/exposure /pubs/episuite.htm) which is considered one of the most reliable peer reviewed models for predicting organic substance biodegradability (Boetlinger 1989, 1994, 2003 and 2004) classifies DEET as not being readily biodegradable.

1.3.4 Uptake and human metabolism

Since DEET is mainly used by direct application on skin, a probable exposure route is through direct dermal absorption. Dermal absorption has been investigated in a study where undiluted technical grade DEET and 15 % DEET in ethanol were applied on the skin of volunteers (Selim et al. 1995). The mean uptake was 5.6 % and 8.4 % respectively. Absorbed DEET was metabolized completely as no intact compound was fund in the urine. DEET is mainly excreted through the urine and metabolic studies of DEET showed that there are two predominating metabolic pathways. Through oxidation of the methyl group on the aromatic ring, diethyl-(hydroxymethyl)benzamide is formed. Alternatively, ethyl toluamide is formed through dealkylation of the amide group (Selim et al. 1995).
Involuntary intake of DEET is another possible route of exposure especially for children. Studies with rats estimated that 96% of the ingested DEET is absorbed. Experiments with oral administration of DEET to dogs showed no evidence of accumulation of DEET in the blood following repeated doses (Department of Health Toxicology, 2002).

2 Methods

2.1 Additional substances

Apart from DEET, alkyl phenols were also analysed in each sample. Alkyl phenols were chosen as substances that could represent anthropogenic influence in general. This was done to support the evaluation since it is believed that the levels of DEET roughly correlate to anthropogenic influence. Also, levels of nonylphenol and DEET have been compared in the aquatic compartment in earlier studies since they are prevalent and represent different usage patterns where the former is to be phased out (Quednow and Püttmann 2009).

2.2 Sampling Strategy

Since household consumption is considered to be the main source of DEET a sampling strategy was devised to elucidate the levels of DEET within sewage treatment plants (STPs) and in receiving surface water recipients. One of the STPs included in the sampling scheme receives waste water from a company manufacturing products containing DEET. Because DEET has been detected in a majority of the ground water samples included in a European study (Loos 2010), ground water samples from urban areas as well as ground water used for production of drinking water was also sampled. Finally sea- and lake water and sediments at recreational bathing sites were sampled to assess the possible direct transport from people taking a bath that had applied DEET to their skin.

Apart from the sampling and analysis financed by the Swedish EPA, regional county administrative boards financed sampling and analysis of additional samples, mostly in sewage treatment plants. Participating counties were Dalarna, Värmland, Södermanland, and Gotland.

The different matrices chosen and types of sampling points are presented in Table 2.1. Detailed results are presented in appendix 1 which shows the types of samples taken at the different locations.

Table 2.1 Sampling matrices and the types of samples investigated for the occurrence of DEET and alkyl phenols. The first value denotes samples taken within the national screening program. The second value (after the slash) denotes samples financed by the regional screening program.

	Sample matrices DEET					
	Incoming water	Outgoing water	Sludge	Surface water	Sediment	Ground water
Drinking water production						2
Background				1	1	
Sewage treatment plant	5 / 1	9 / 5	4	4	1	
Urban affected areas				5 / 2	2	6
Total	5/1	9/5	4	10/2	4	8
Total	40 / 8					

2.3 Sampling methods

Sampling instructions were given to all sampling personnel. The instructions included sampling procedures and handling of samples. The importance of avoiding use of insect repellents was emphasized in the instructions.

2.3.1 Sewage water

The staff at the sewage treatment plants collected water samples in glass containers or stainless steel containers. Plastic containers were avoided to exclude cross-contamination of alkyl phenols. A time integrated or flow-proportional pooled sample was collected for 4-7 days often in coordination with the ongoing operational monitoring program. Samples were kept cool until analysis. A volume of 1 l of the sample was analysed for DEET and in addition a volume of 0.5 l was analysed for alkyl phenols. All sewage samples were collected in September and October.

2.3.2 Sewage sludge

The staff at the sewage treatment plants collected untreated sludge samples in acid rinsed pre burned dark glass jars. A pooled sludge sample was collected to represent approximately the same time span as the sewage samples but may in some cases, due to the operational procedures, represent a somewhat longer time span. All sludge samples were collected in September and October.

2.3.3 Surface water

Pooled grab samples of unfiltrated water was collected in clean glass bottles. Water samples were stored cold until analysis. A volume of 1 l of the sample was analysed for DEET and in addition a volume of 0.5 l was analysed for alkyl phenols. Water samples from recreational bathing sites were collected in July and August and remaining surface water samples were collected in September and October. Water at recreational bathing sites were collected in close vicinity to bathers at bathing localities where mosquitos were present. The water samples are consequently not representative of the whole lake.

2.3.4 Ground water

Incoming unprocessed ground water used for production of drinking water was collected in clean glass bottles as a grab sample at the waterworks. A volume of 1 l of the sample was analysed for DEET and in addition a volume of 0.5 l was analysed for alkyl phenols. Ground water in urban areas was collected from monitoring wells. A volume of 1 l of the sample was analysed for DEET and in addition a volume of 0.5 l was analysed for alkyl phenols. Ground water samples from urban areas were collected in November and samples from ground water intended for production of drinking water were collected in December.

2.3.5 Sediment

Sediment samples were collected by means of a core sampler or Ekman dredge. All sediment samples were transferred to pre-burned and dark glass jars. They were stored cold until analysis. Sediment from recreational bathing sites and background were collected in July and August. Sediment samples from recipient were collected in September. Sediments at recreational bathing sites were collected in close vicinity to bathers at bathing localities where mosquitos were present. The samples are consequently not representative of the whole lake.

2.4 Analytical methods

2.4.1 Water samples:

Analysis of DEET in water and sewage sludge was performed by ALS laboratories. After adding internal standard DEET-D7 to 1 l of liquid sample, liquid-liquid-extraction with dichloromethane was performed. The extract was evaporated and final analysis was performed with GC-MS (Sim-mode). Quantification was done against the internal standard. Before the onset of this study the analytical method was discusses with the laboratory. The present method has a limit of quantification (LOQ) of 1-2 ng/L which was considered acceptable based on the levels of DEET found in the previous study by Loos *et al.* (2010). The analytical methods chosen for this study had also been used for a large number of water samples by the laboratory with generally good results. .

2.4.2 Sludge and sediment samples:

Solid samples of 10-20 g were air-dried and then liquid-extracted with dichloromethane after adding internal standard DEET-D7. The extract was evaporated and final analysis was performed with GC-MS (Sim-mode). Quantification was done against the internal standard. Before the onset of this study the analytical method was discusses with the laboratory. No previous studies of DEET in solid samples were found for comparison. A limit of quantification (LOQ) of 5 µg/kg TS was considered sufficient for the purposes of this study.

3 Results

The complete list of sampling points and results are presented in Appendix 1.

DEET was not detected (limit of detection, LOD = 2 µg / kg TS) in any of the four sludge samples from sewage treatment plants. The compound was however detected in most of the incoming and in all of the outgoing water samples from the sewage treatment plants. Results from sewage water samples are presented in Figure 3.1 together with the concentrations of the reference substance nonylphenol[1]. Table 3.1 presents summary statistics on concentrations of DEET in different environmental matrixes.

Levels of nonylphenol were almost always higher than DEET in incoming water and effluent from STPs. Also, there was no significant relationship (correlation coefficient, $R^2 < 0.1$), between levels of DEET and nonylphenol in waste water or surface water.

Levels of DEET was higher in incoming water compared to effluent in three STPs (Borlänge, Krylbo, and Strängnäs) and roughly equal in one STP (Österfärnebo). The sample of incoming water to Fiskartorpet STP may very well also contain DEET but the matrix interference was unusually high in this sample which led to an elevated LOQ of 0,09 µg/L. The final sample of incoming sewage water was an extra sampling at Främby and consisted of untreated industrial waste water delivered by tank truck to the plant. The matrix interference in this sample was so severe that it was not possible to quantify DEET in this sample. Although incoming and outgoing water was only sampled in five STPs, the results indicate a (significant) relationship between DEET concentrations in these two water compartments (Figure 3.2).

DEET was detected in both ground water samples from Falun, in one of the two samples from Sundsvall and in one of the two samples from Malmö.

DEET was also found in surface waters, with the highest concentrations found in close vicinity of bathers at bathing sites. Also, one bathing water sample with unrealistic high concentration of DEET (>1 mg/L) were excluded from the comparison. This sample was collected under similar circumstances but was probably contaminated with non-dissolved droplets of insect repellent.

[1] sum of all nonylphenol moieties except 4-n-nonylphenol

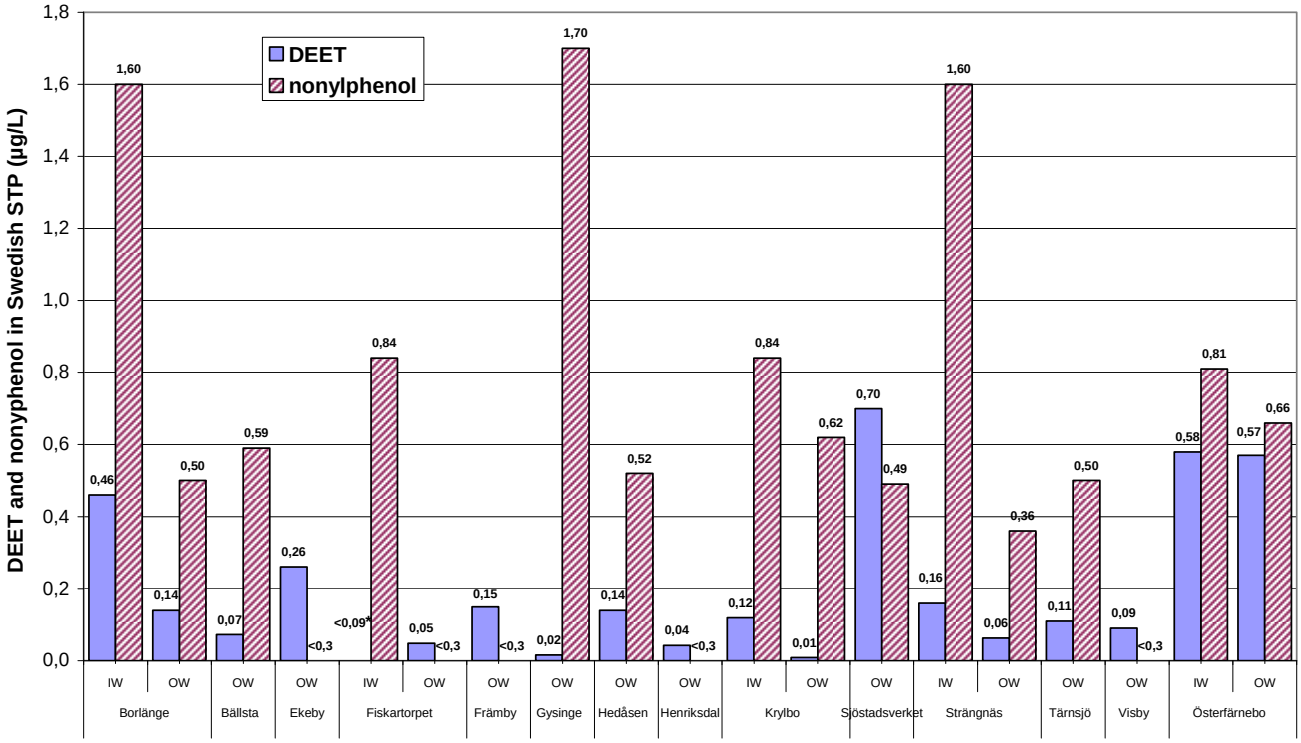


Figure 3.1 ***Levels of DEET and nonylphenol in incoming water and outgoing water (effluent) of STPs. IW = incoming water, OW = outgoing water. For sample location and specification of samples of national and regional interest see Appendix 1.***
**** elevated LOQ due to matrix interference***

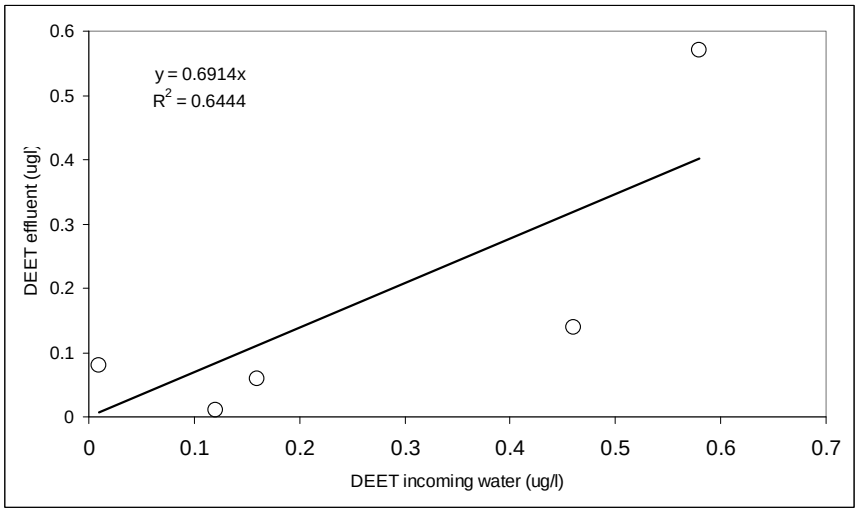


Figure 3.2 Relationship between DEET concentrations in incoming (X axis) and outgoing/effluent water(Y axis) from five STPs.

Table 3.1 Limit of quantification (LOQ), number above LOQ / number of samples (n), median concentration, 25th and 75th percentile of DEET in different matrices. Note that only concentrations above LOQ were included in the statistical calculations.

Source/matrix	LOQ	n	median	25th percentile	75th percentile
Sludge (µg/kg TS)	2	0/4	-	-	-
Incoming sewage water (ng/l)	90	4/6	310	130	550
Outgoing sewage water (ng/l)	1	14/14	110	53	147
Surface water downstream STP (ng/l)	2	4 / 4	52	13	203
Surface water with diffuse urban influence (ng/l)	2	0/2	-	-	-
Surface water at recreational bathing sites (ng/l)	1	4/4	120	60	255
Urban ground water (ng/l)	2	4/6	12	3	30
Sediment (µg/kg)	5	1/4	3800*	-	-
Untreated drinking water (ng/l)	2	0/2	-	-	-

* Presented value is not median but only one detected concentration.

4 Discussion

4.1 Broad comparison with other studies

An overview on the levels of DEET in the present study compared with findings from other countries is given in Table 4.1.

No comparison between the usage of DEET in Sweden and other countries is made here, but it should be emphasised that many of the samples were taken in parts of Sweden were usage of mosquito repellents is very common due to high incidence of mosquitoes. If mosquito repellent usage is a main factor behind environmental levels, it could be expected that the levels in the present study should be at par or higher than levels in other. In fact, the levels in STPs and downstream the plants in this study are generally lower than what has been found in other countries. One explanation is that other parameters, i.e. population density, may control environmental levels.

Table 4.1 Concentrations of DEET in the present study compared to studies from other countries.

Matrix	unit	present study	other studies (max or min-max)	Countries	Reference
Incoming sewage water	ng/l	<90 – 580	1500 3000	Australia Germany	a b
Sludge	µg/kg	< 0.002			
Outgoing sewage water	ng/l	9 - 700	60 140 1500 2100	Norway Australia Germany USA	c a b d
Surface water	ng/l	<2 – 1100*	490 30 40 190 1130 640 130	Australia Germany Netherlands USA USA USA USA	a f g h i d j
Ground water	ng/l	<2 - 60	<0.4 – 454 13000 30	EU USA Spain	l m n
Sediment	µg/kg	<1**	none found		
Raw and treated drinking water	ng/l	n.d. (<2)	8 – 13 3 - 270	USA USA and Europe	O p

[a] Costanzo et al. 2007

[b] Knepper. 2004

[c] Weigel et al. 2004.

[d] Glassmeyer et al. 2005

[e] Weigel et al. 2002.

[f] Schwartzbauer and Heim 2005.

[g] Hendriks et al 1994

[h] Oros et al 2003

[i] Kolpin et al 2002

[j] Kolpin et al 2004

[k] Stackelberg et al. 2004

[l] Loos et. al. 2010

[m] Barnes et al 2005

[n] Guardiola et al 1989

[o] Thacker 2005

[p] Lorraine and Petigrove 2006

* Note that the concentration of 1100 ng/l was an outlier found in bathing water being > 10 times higher than any other concentration found in this study

** DEET was detected in one sediment sample, but at a very (unrealistically) high concentration

4.2 Background levels

DEET was not found in sediments or surface waters from the regional background lake Ljusacksen situated in the county of Dalarna. There is at present no data available on the background levels of DEET in other areas or in precipitation. Due to its physicochemical properties, DEET displays a low potential for long range atmospheric transport. On the other hand, the personal usage of repellents by humans may result in the direct spreading of DEET to remote areas.

4.3 DEET in STPs

DEET was common in both incoming and outgoing waste water in STPs, and the levels were on average 2 – 3 times higher in incoming water.

Physicochemical properties (log K_{oc} and water solubility) of DEET indicates that the substance do not partition to sludge to any large degree which is confirmed by the lack of detection in sludge in this study, and the lack of detection and/or generally low levels in other studies. Also, the Henrys law constant and the vapour pressure of DEET indicates that volatilization is not an important process. Instead, biodegradation is the most important reduction process in STPs (Bernard et al. 2006, Kagle et al. 2009).

Bernard et al. (2006) used full scale studies at STPs to show that DEET belonged to a group of polar organic compounds that was less biodegradable in activated sludge. The total biodegradation loss amounted to 17 % during half a year of operation of the treatment plant. In the present project, the loss in of DEET in a STP was modelled using US EPAs STPWin (Seth et al. 2001) which is based on fugacity principles and attempts to predict the fate of an organic chemical in a conventional wastewater treatment plant that uses activated sludge secondary treatment. The model predicted that

biodegradation would amount to ca 20 % in a STP[2] and that no other processes (volatilization and sorption to sludge) would cause any loss in the STP. The slope of the correlation between DEET in incoming water and effluent of this study (Figure 3.2) indicate that the loss in STPs was approximately 35 % which is relatively close to a theoretical prediction of 20 % loss.

To conclude, modelling and measurements in this and other studies indicate that STPs do not retain DEET to any large degree. The most important fate process in for DEET in a STP is most likely biodegradation.

4.4 DEET in surface waters and sediments

DEET has been detected in a wide range of surface waters across the world with a detection frequency ranging between 60 – 100% in surface waters of Germany, Australia, USA, and the Netherlands (Costanzo et al. 2007). Data from different monitoring efforts were analysed (op. cit.) taking into account, potential sources of DEET. This analysis shows that the majority of DEET enters waterways via sewage effluent, probably following washing off and absorption/excretion by humans. This accord with the results from the present study, where DEET was found in all waterways downstream of STPs, and in water influenced by bathing activities, but not in waterways generally influenced by urban activities.

The lack of detection of DEET in most sediment samples accords with previous studies (USGS 2004) and with the physiochemical properties of DEET. Fugacity modelling of DEET using a level III[3] fugacity model shows that < 0.5 % of the substance is expected to occur in sediments.

The direct spreading of DEET from humans may be transiently important, as indicated by the concentration of DEET in surface waters at sites for recreational bathing. In one sediment sample, a very high DEET concentration of 3.8 mg/kg was found. Given the lack of detection in other sediment samples as well as the lack of occurrence in sediments in earlier studies this value should be viewed with some scepticism. The value may be a result of direct contact with sediment/sand of people that had applied DEET to their skin and/or analytical errors.

In summary, DEET is very prevalent in streams downstream of STPs in which is in accordance with the results from previous monitoring studies. It is also prevalent in bathing water that is in direct contact with bathers who have applied DEET to their skin.

4.5 DEET in ground water

One of the underlying studies motivating the screening of DEET in Sweden was the study by Loos et al (2010) where DEET was found in 84% of 164 samples of ground water in 23 countries. It is therefore not surprising to note that DEET was detected in 67 % of the tested ground water samples. DEET was detected in three of the four samples from the northern part of Sweden and in one of the two samples from the south. The need for protection against mosquitoes and ticks in Sweden is

[2] Using the Biowin EPA draft method. Biowin also predicts that DEET is Not readily biodegradable.

[3] A general, fugacity models predict the partitioning of an organic compound in an evaluative environment. A Level III model assumes steady-state but not equilibrium conditions.

probably bigger than in many of the other countries included in the former study. The occurrence in ground water may very well be related to this usage, since DEET was found in groundwater from areas of northern Sweden where mosquitoes and repellent usage is frequent. DEET was also found in groundwater from southern Sweden which was not expected since mosquitoes are much less prevalent there. The 90th percentile in the European wide study was 9 ng/l, while the concentrations in groundwater from northern Sweden were 9, 14, 34, and 60 ng/l. One of the samples from the northern parts of Sweden was taken close to a facility where DEET is used in production. Still, it may be the case that the levels in Sweden are higher than European levels in general.

4.6 DEET in drinking water

DEET was not detected in raw untreated drinking water in the present study, this in contrast to studies from other countries where DEET has been one of the more common organic substances occurring in drinking water (Lorraine and Petigrove 2006). However, it is believed that the prevalence of DEET in drinking water is mostly due to the fact that the water source in many countries are affected by STPs, either directly when water is extracted from surface water or indirectly, when ground water is affected by polluted surface waters (Thacker 2005). In the present study, the analysed drinking water does not originate from water sources that are influenced by STPs.

4.7 Risk assessment

For the risk assessment, only DEET concentrations downstream of STPs were used. The main reason is that water samples taken in the close vicinity of bathers that has applied DEET to their skin can not be viewed as representative of concentrations in the water of these lakes. The PNEC value for limnic surface waters is 47 000 ng/l (table 1.1) while the maximum concentration in surface waters was 200 ng/l. This gives a MEC/PNEC ratio of 0.0046. Given available data on ecotoxicological effects this indicates that, despite its prevalent occurrence in surface waters, DEET does not constitute an ecological risk to aquatic ecosystems. This assessment does not take into account:

1. The large number of polar organic substances that are prevalent in European surface waters (Loos et al. 2009) and possible toxicological interactions between DEET and these substances.
2. Levels and effects of DEET degradation products.

The major risk with polluting chemicals in groundwater is their transport to surface waters where they may cause detrimental ecological effects and the effect on stygofauna[4]. There is however no data available on the effects of most organic pollutants on stygofaunal animals which make it impossible to assess the risks of groundwater contaminants to this organism group. Furthermore, since the levels of DEET in groundwater are below the levels in surface water it is not necessary to assess the risks of DEET in groundwater to surface water ecosystems.

[4] Fauna that live within groundwater systems. Usually small aquatic groundwater invertebrates. Stygofauna can live within freshwater aquifers and within the pore spaces of limestone, calcrete or laterite, but are also found in marine caves and wells along coasts.

5 Conclusions and recommendations

The most important conclusions were:

- The levels of DEET in this study are generally lower than what has been found in other countries, with the exception of DEET compared to European groundwater.
- DEET was not found in the limnic environment of a regional background lake Ljusacksen which is in accordance to theoretical predictions
- DEET was common in both incoming and outgoing waste water in STPs. Previous studies have shown that biodegradation is the only removal process in STPs, and predicted STP removal (20 %) was relatively close to measured removal (35 %).
- DEET was very prevalent in watercourses downstream of STPs which is in accordance with the results from previous monitoring studies. In accordance to theory and previous studies, DEET was not found in sediments apart from one bathing water site.
- DEET was also prevalent in water samples collected close to bathers at locations where mosquitos were present.
- DEET was found in groundwater from areas of northern Sweden where mosquitoes and repellent usage is frequent, but also in groundwater from southern Sweden. The levels in Sweden may be higher than European levels in general given that the lowest concentration was equal to the 90th percentile from a pan-European study.
- DEET was not detected in raw untreated drinking water in the present study which is in contrast to studies from other countries. The difference may be because water sources in many countries are affected by STPs which was not the case for the water sources in the present study.
- Given available knowledge on ecotoxicological effects, it apparent that DEET does not constitute an acute risk to the aquatic environment.

To summarize, DEET is found in surface water where people are bathing, in sewage and in surface waters that are influenced by STPs. This and other studies also show that DEET frequently occur in groundwater in Sweden. DEET is not found in solid samples like sludge or sediments.

Using available data on ecotoxicological effects indicates that the high prevalence do not constitute an acute risk to aquatic ecosystems.

This risk assessment does not take into account the large number of polar organic substances that are prevalent in European surface waters (Loos et al. 2009) and possible toxicological interactions

between DEET and these substances. Furthermore, the conclusion is only based on laboratory studies toxicity, and there is currently a lack of information on ecological effects., particularly with regards to mechanisms of toxicity and chronic toxicity. In general, acute laboratory ecotoxicity studies is less appropriate for environmental risk assessment since these tests do not reflect true exposure conditions (Kummerer, 2004), although the usage of assessment factor to some degree alleviates this problem. Further information is needed on chronic ecological effects of DEET before a final assessment on the environmental risk of the high prevalence of DEET in the aquatic environment. Also, levels of DEET degradation products and their possible ecotoxicological effects are largely unknown, and this issue may also have to be determined before a final assessment can be made.

The final recommendations are consequently:

1. No further screening study of DEET is necessary at present given the indication that the frequently observed concentrations are well below effect concentrations

2. This assessment should be renewed when more data becomes available on:
    a. the long term ecological effects of DEET
    b. toxicological interactions with other compounds that occurs in surface waters
    c. occurrence and effects of DEET degradation products

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Appendix 1, Results Screening 2010 - 2011

DEET and Alkyl phenols

Incoming sewage water

compound	county	national/regional	Sample name/location	Concentration	Unit
DEET	Gävleborg	N	Österfärnebo ARV, inkommande avloppsvatten	0,580	µg/L
DEET	Dalarna	N	Borlänge ARV, inkommande avloppsvatten	0,460	µg/L
DEET	Värmland	N	Sjöstadsverket ARV, inkommande avloppsvatten	0,160	µg/L
DEET	Dalarna	N	Krylbo ARV, inkommande avloppsvatten	0,120	µg/L
DEET	Värmland	N	Fiskartorpet ARV, inkommande avloppsvatten	<0,09	µg/L
DEET	Dalarna	R	Främby ARV, inkommande avloppsvatten (tankbil)	<5	µg/L
nonyl phenols	Värmland	N	Sjöstadsverket ARV, inkommande avloppsvatten	2,300	µg/L
nonyl phenols	Dalarna	N	Borlänge ARV, inkommande avloppsvatten	1,600	µg/L
nonyl phenols	Dalarna	N	Främby ARV, inkommande avloppsvatten	1,100	µg/L
nonyl phenols	Dalarna	N	Krylbo ARV, inkommande avloppsvatten	0,840	µg/L
nonyl phenols	Värmland	N	Fiskartorpet ARV, inkommande avloppsvatten	0,840	µg/L
nonyl phenols	Gävleborg	N	Österfärnebo ARV, inkommande avloppsvatten	0,810	µg/L

Outgoing sewage water

compound	county	national/regional	Sample name/location	Concentration	Unit
DEET	Värmland	R	Sjöstadsverket ARV, utgående avloppsvatten	0,700	µg/L
DEET	Gävleborg	N	Österfärnebo ARV, utgående avloppsvatten	0,570	µg/L
DEET	Södermanland	N	Ekeby ARV, utgående avloppsvatten	0,260	µg/L
DEET	Dalarna	R	Främby ARV, utgående avloppsvatten	0,150	µg/L
DEET	Dalarna	R	Borlänge ARV, utgående avloppsvatten	0,140	µg/L
DEET	Gävleborg	N	Hedåsen ARV, utgående avloppsvatten	0,140	µg/L
DEET	Uppsala	N	Tärnsjö ARV, utgående avloppsvatten	0,110	µg/L
DEET	Gotland	N	Visby ARV, utgående avloppsvatten	0,091	µg/L
DEET	Västernorrland	N	Bällsta ARV, utgående avloppsvatten	0,073	µg/L
DEET	Södermanland	R	Strängnäs ARV, utgående avloppsvatten	0,063	µg/L
DEET	Värmland	R	Fiskartorpet ARV, utgående avloppsvatten	0,049	µg/L
DEET	Stockholm	N	Henriksdal ARV, utgående avloppsvatten	0,043	µg/L
DEET	Gävleborg	N	Gysinge ARV, utgående avloppsvatten	0,016	µg/L
DEET	Dalarna	N	Krylbo ARV, utgående avloppsvatten	0,009	µg/L
nonyl phenols	Gävleborg	N	Gysinge ARV, utgående avloppsvatten	1,700	µg/L
nonyl phenols	Gävleborg	N	Österfärnebo ARV, utgående avloppsvatten	0,660	µg/L
nonyl phenols	Dalarna	N	Krylbo ARV, utgående avloppsvatten	0,620	µg/L
nonyl phenols	Västernorrland	N	Bällsta ARV, utgående avloppsvatten	0,590	µg/L
nonyl phenols	Gävleborg	N	Hedåsen ARV, utgående avloppsvatten	0,520	µg/L
nonyl phenols	Dalarna	N	Borlänge ARV, utgående avloppsvatten	0,500	µg/L
nonyl phenols	Uppsala	N	Tärnsjö ARV, utgående avloppsvatten	0,500	µg/L
nonyl phenols	Värmland	N	Sjöstadsverket ARV, utgående avloppsvatten	0,490	µg/L
nonyl phenols	Södermanland	N	Strängnäs ARV, utgående avloppsvatten	0,360	µg/L
nonyl phenols	Dalarna	N	Främby ARV, utgående avloppsvatten	<0,3	µg/L
nonyl phenols	Gotland	N	Visby ARV, utgående avloppsvatten	<0,3	µg/L
nonyl phenols	Stockholm	N	Henriksdal ARV, utgående avloppsvatten	<0,3	µg/L
nonyl phenols	Södermanland	N	Ekeby ARV, utgående avloppsvatten	<0,3	µg/L
nonyl phenols	Värmland	N	Fiskartorpet ARV, utgående avloppsvatten	<0,3	µg/L

Surface water

compound	county	national/regional	Sample name/location	Concentration	Unit
DEET	Västra Götaland	N	badsjö 1 ytvatten	1,100	µg/L
DEET	Västra Götaland	N	badsjö 3 ytvatten	0,240	µg/L
DEET	Västra Götaland	N	badsjö 2 ytvatten	0,080	µg/L
DEET	Jönköping	N	badsjö 5 ytvatten	0,080	µg/L
DEET	Gävleborg	N	Österfärnebo nedströms ARV, Norrån före Fängsjön, ytvatten	0,068	µg/L
DEET	Dalarna	R	Runn Främbyviken, ytvatten	<0,002	µg/L
DEET	Dalarna	N	Ljusacksen ytvatten	<0,05	µg/L
DEET	Värmland	R	Varnumsviken, ytvatten diffus	<0,002	µg/L
DEET	Södermanland	N	Eskilstunaån, ytvatten nedströms Ekeby ARV	0,004	µg/L
DEET	Södermanland	N	Ekeby ARV, ytvatten utgående efter våtmark	0,200	µg/L
DEET	Uppsala	N	Tärnsjö ARV, bäck nedströms ARV	0,210	µg/L
nonyl phenols	Gävleborg	N	Österfärnebo nedströms ARV, Norrån före Fängsjön, ytvatten	1,100	µg/L
nonyl phenols	Uppsala	N	Tärnsjö ARV, bäck nedströms ARV	0,330	µg/L
nonyl phenols	Dalarna	N	Runn Främbyviken, ytvatten	<0,3	µg/L
nonyl phenols	Västra Götaland	N	badsjö 1 ytvatten	<0,3	µg/L
nonyl phenols	Västra Götaland	N	badsjö 2 ytvatten	<0,3	µg/L
nonyl phenols	Jönköping	N	badsjö 5 ytvatten	<0,3	µg/L
nonyl phenols	Västra Götaland	N	badsjö 3 ytvatten	<0,3	µg/L
nonyl phenols	Dalarna	N	Ljusacksen ytvatten	<0,3	µg/L
nonyl phenols	Värmland	N	Varnumsviken, ytvatten diffus	<0,3	µg/L
nonyl phenols	Södermanland	N	Eskilstunaån, ytvatten nedströms Ekeby ARV	<0,3	µg/L
nonyl phenols	Södermanland	N	Ekeby ARV, ytvatten utgående efter våtmark	<0,3	µg/L

Ground water

compound	county	national/regional	Sample name/location	Concentration	Unit
DEET	Skåne	N	Malmö S 1009, grundvatten	0,060	µg/L
DEET	Dalarna	N	Falun stadsmiljö östra grundvatten	0,035	µg/L
DEET	Västernorrland	N	Sundsvall GV 1009, grundvatten	0,014	µg/L
DEET	Dalarna	N	Falun stadsmiljö västra, grundvatten	0,009	µg/L
DEET	Skåne	N	Malmö S 1001, grundvatten	<0,002	µg/L
DEET	Västernorrland	N	Sundsvall GV 1003, grundvatten	<0,002	µg/L
DEET	Gävleborg	N	Österfärnebo, grundvatten råvatten vattenverk	<0,002	µg/L
DEET	Gävleborg	N	Årsunda, grundvatten råvatten vattenverk	<0,002	µg/L
nonyl phenols	Dalarna	N	Falun stadsmiljö västra, grundvatten	0,420	µg/L
nonyl phenols	Dalarna	N	Falun stadsmiljö östra grundvatten	<0,3	µg/L
nonyl phenols	Skåne	N	Malmö S 1001, grundvatten	<0,3	µg/L
nonyl phenols	Skåne	N	Malmö S 1009, grundvatten	<0,3	µg/L
nonyl phenols	Västernorrland	N	Sundsvall GV 1003, grundvatten	<0,3	µg/L
nonyl phenols	Västernorrland	N	Sundsvall GV 1009, grundvatten	<0,3	µg/L
nonyl phenols	Gävleborg	N	Österfärnebo, grundvatten råvatten vattenverk	<0,3	µg/L
nonyl phenols	Gävleborg	N	Årsunda, grundvatten råvatten vattenverk	<0,3	µg/L

Sludge

compound	county	national/regional	Sample name/location	Concentration	Unit
DEET	Dalarna	N	Krylbo ARV, slam	<0,002	mg/kg TS
DEET	Dalarna	N	Främby ARV, slam	<0,002	mg/kg TS
DEET	Gävleborg	N	Gysinge ARV, slam	<0,002	mg/kg TS
DEET	Dalarna	N	Borlänge ARV, slam	<0,002	mg/kg TS
4-tert-octylphenol	Dalarna	N	Borlänge ARV, slam	0,370	mg/kg TS
4-tert-octylphenol	Dalarna	N	Krylbo ARV, slam	0,250	mg/kg TS
4-tert-octylphenol	Gävleborg	N	Gysinge ARV, slam	0,110	mg/kg TS
4-nonylphenol	Dalarna	N	Borlänge ARV, slam	18,000	mg/kg TS
4-nonylphenol	Dalarna	N	Krylbo ARV, slam	14,000	mg/kg TS
4-nonylphenol	Gävleborg	N	Gysinge ARV, slam	2,400	mg/kg TS

Sediment

compound	county	national/regional	Sample name/location	Concentration	Unit
DEET	Västra Götaland	N	badsjö 2 sediment	3,800	mg/kg TS
DEET	Södermanland	N	Eskilstunaån, sediment nedströms Ekeby ARV	<0,005	mg/kg TS
DEET	Västra Götaland	N	badsjö 1 sediment	<0,01	mg/kg TS
DEET	Dalarna	N	Ljusacksen sediment bakgrund	<0,01	mg/kg TS
4-tert-octylphenol	Södermanland	N	Eskilstunaån, sediment nedströms Ekeby ARV	<0,015	mg/kg TS
4-nonylphenol	Västra Götaland	N	badsjö 2 sediment	0,030	mg/kg TS
4-nonylphenol	Västra Götaland	N	badsjö 1 sediment	<0,02	mg/kg TS
4-nonylphenol	Dalarna	N	Ljusacksen sediment bakgrund	<0,02	mg/kg TS
4-nonylphenol	Södermanland	N	Eskilstunaån, sediment nedströms Ekeby ARV	<0,15	mg/kg TS