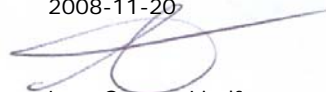


# Results from the Swedish National Screening Programme 2007

Subreport 2:  
Octadecyl 3-(3,5-di-tert-butyl-4-  
hydroxyphenyl)propionate

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<p><b>Title and subtitle of the report</b> Results from the Swedish National Screening Programme 2007 Subreport 2: Ester</p>	
<p><b>Summary</b> IVL has performed a "screening study" of the ester octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate on commission from the Swedish EPA. The objectives of the screening were to determine if octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate is present in different matrices in the Swedish environment, and if so, at what concentrations. In total 64 samples were analysed representing soil, surface water, sediment, biota (fish), influent and effluent waters and sludge from STPs, storm waters, and leachate water and sludge from landfills. Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate was found in all analysed STP sludge samples. It was also found in all analysed influent water samples, but concentrations were generally lower or below the detection limit in effluent water. Thus, removal of the substance from the water phase takes place during the waste water treatment process, plausible by absorption to sludge. Concentrations in the same range as in influent waters or higher was also found in the storm water samples. The chemical could also be detected just above the detection limit in two out of three fish samples, but not in soil, surface water or sediment from the city of Stockholm. Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate could not be detected in any of the samples from background areas. All together, this screening study shows that diffuse emissions of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate in the urban environment take place, resulting in exposure to and uptake in biota. Due to the limited ecotoxicological data available, it is not possible to determine the risk of its presence in the environment, but based on animal studies the chemical is considered to have a low hazard potential for human health.</p>	
<p><b>Keywords</b> Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate, screening, air, water, sediment, sludge, fish, storm-water</p>	
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## Sammanfattning

IVL Svenska Miljöinstitutet AB har på uppdrag av Naturvårdsverket genomfört en screening av estern octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate. Denna kemikalie kan donera en väteatom och därmed neutralisera fria radikaler. Den används därför som en antioxidant, främst vid kemikalietillverkning och i plast och gummiprodukter, men även inom skogsbruket och av massaindustrin.

Experimentella data avseende kemiska egenskaper, nedbrytning, spridning samt transport i miljön är begränsade, men den litteratur som finns tyder på att kemikalien är relativt persistent, kan läcka från produkter samt binder till fasta matriser i miljön.

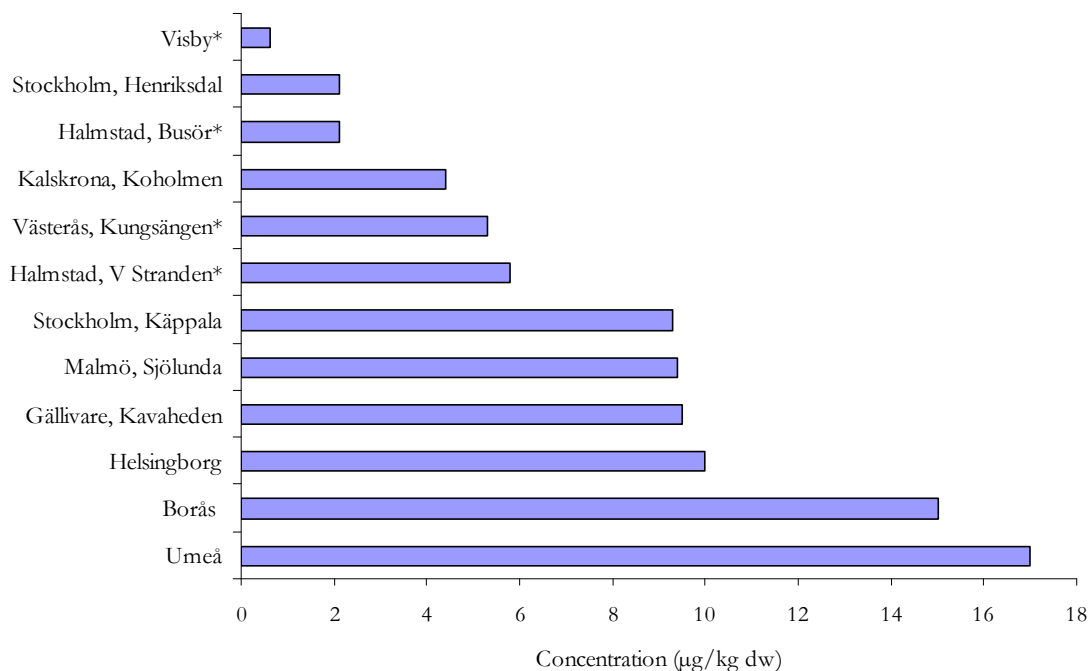
Exponering för octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate kan ske genom migration av kemikalien till födoämnen och vatten, men de toxicitetsstudier som gjorts tyder på att detta inte utgör någon hälsorisk.

Det huvudsakliga syftet med föreliggande screening var att utreda om octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate förekommer i den svenska miljön, och i så fall i vilka koncentrationer. Provtagningsprogrammet baserades på tillgänglig information om användning av octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate, dess kemiska egenskaper, modellerad fördelning i miljön, samt studier som visat på visst läckage från plaster. Utifrån dess kemiska egenskaper så som låg vattenlöslighet, hög molekylvikt och lågt ångtryck, predikterades kemikalien att väl i miljön främst binda till fasta matriser. Slam, jord och sediment bedömdes därför vara viktiga matriser. Potentiell spridning av octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate i miljön förväntas främst genom läckage från varor vid användning samt avfallshantering. Utifrån detta valdes provtagningsplatser ut representerande punktkällor (avfallsdeponier) diffusa källor (reningsverk samt matriser i urban miljö), samt bakgrundsområden. För att uppskatta exponering och eventuell bioackumulering i biota analyserades fisk (abborre). Totalt bestämdes octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate i 64 prover av jord, sediment, slam, avloppsvatten, lakvatten, ytvatten, dagvatten, samt fisk.

15 av dessa 64 prover utgjordes av prover från det regionala programmet (1 sediment, 7 avloppsvatten, 4 slam samt 3 lakvatten) vilka skickats in från fyra deltagande länsstyrelser.

Låga men detekterbara halter av octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate uppmättes i ingående och utgående avloppsvatten samt slam från avloppsverk, i tre av fyra dagvatten, samt i två av tre fiskprover från från centrala Stockholm. I de övriga matriserna från den urbana miljön; i jord, ytvatten, samt sediment, kunde kemikalien inte detekteras. Detta visar att det sker en diffus, om än låg, spridning av octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate i den urbana miljön.

Nedan visas de uppmätta halterna i avloppsslam från både den nationella screeningen och det regionala programmet (\*).



För alla reningsverk utom Kungsängens var halterna lägre i utgående- än i ingående avloppsvatten, se tabell nedan.

Avloppsverk	Ingående vatten (µg/l)	Utgående vatten (µg/l)
Nyköping, Brandholmen <sup>a</sup>		0.0022
Halmstad, Busör <sup>a</sup>	0.0024	< 0.001
Stockholm, Henriksdal	0.011	0.0010
Västerås, Kungsängen <sup>a</sup>	0.0048	0.0071
Stockholm, Käppala	0.0037	0.0029
Umeå	0.016	< 0.001
Halmstad, V Stranden <sup>a</sup>	0.013	< 0.001

<sup>a</sup> Prover från det regionala programmet

De högsta vattenhalterna detekterades i dagvatten från centrala Stockholm, se nedan. Det högsta värdet var i storleksordningen 10 gånger högre än i inkommande avloppsvatten.

Provtagningsplats dagvatten	Koncentration (µg/l)
Eugeniattunneln	< 0.001
Torstenssonsgatan	0.038
Starrängsringen	0.0053
Sveavägen	0.11

Nedan presenteras uppmätta halter i fisk från centrala Stockholm. Halter strax över detektionsgränsen uppmättes i två av tre prover. Detta visar att det sker en exponering, samt att kemikalien tas upp i biota.

<b>Provtagningsplats fisk</b>	<b>Koncentration (<math>\mu\text{g}/\text{kg}</math> våtvikt)</b>
Årstaviken	<0.02
Stora Essingen	0.07
Riddarfjärden	0.04

Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate kunde inte detekteras in någon undersökt matris från bakgrundsområden.

Sammanfattningsvis visar denna screening på att diffus spridning av octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate sker och att detta leder till exponering av biota.



## Summary

As an assignment from the Swedish Environmental Protection Agency, IVL Swedish Environmental Research Institute has during 2007/2008 performed a "Screening Study" of the ester octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate. The overall objectives of the screening were to determine if octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate is present in different matrices in the Swedish environment, and if so, in what concentrations. The screening programme included measurements in background areas, as well as in urban areas reflecting diffuse emission pathways from the society. Sample types analysed were soil, surface water, sediment, biota (fish), influent and effluent waters and sludge from STPs, storm waters, and leachate water and sludge from landfills. A total of 64 samples were included.

Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate was found in all analysed STP sludge samples. It was also found in all analysed influent water samples, but concentrations were generally lower or below the detection limit in effluent water. Thus, removal of the substance from the water phase takes place during the waste water treatment process, plausible by absorption to sludge.

Concentrations in the same range as in influent waters or higher was also found in the storm water samples. The chemical could also be detected just above the detection limit in two out of three fish samples, but not in soil, surface water or sediment from the city of Stockholm.

For the identified potential point sources, landfills and waste treatment plants, concentrations above the detection limits were only found in two out of eight leachate water samples and in one out of two sludge samples.

Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate could not be detected in any of the samples from background areas.

All together, this screening study shows that diffuse emissions of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate in the urban environment take place, resulting in exposure to and uptake in biota. Due to the limited ecotoxicological data available, it is not possible to determine the risk of its presence in the environment, but based on animal studies the chemical is considered to be of low hazard potential for human health.

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Appendix 1: Information on samples and results from the National Screening Programme

Appendix 2: Information on samples and results from the Regional Screening Programme



## 1 Introduction

As an assignment from the Swedish Environmental Protection Agency, IVL has during 2007/2008 performed a "Screening Study" of amines, esters, pigments, linear alkyl benzene sulfonate (LAS) and silver. These substances are emitted and distributed in the environment via a variety of sources, e.g. different point sources and/or diffusive sources. Some of them are used in consumer products.

The overall objectives of the screening studies are to determine the concentrations of the selected substances in a variety of media in the Swedish environment, to highlight important transport pathways, and to assess the possibility of current emissions in Sweden.

The results are given in five subreports according to Table 1.

Table 1. Substances / substance groups included in the screening

Substance / Substance group	Sub-report #
Amines:	1
3,6,9,12-Tetraazatetradecane-1,14-diamine	
N-cyclohexyl-2-benzothiazolamine (NCBA)	
N-isopropyl-N'-phenyl-p-phenylenediamine (IPPD)	
N-Phenyl-benzeneamine	
Dicyclohexylamine	
Esters:	2
Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate	
Pigments:	3
Pigment yellow 1 (CI 11680)	
Pigment orange 5 (CI 12075)	
Pigment red 53:1 (CI 15585:1)	
Pigment red 170 (CI 12475)	
Linear alkyl benzene sulfonate (LAS)	4
Silver	5

This subreport considers the screening of the ester, octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate (CAS 2082-79-3). There are several synonyms for this compound, e. g. octadecyl 3,5-di-tert-butyl-4-hydroxyhydrocinnamate and benzenepropanoic acid, 3,5-bis(1,1-dimethylethyl)-4-hydroxy octadecyl ester, and several trade names such as Irganox 1076, Ethanox 376, and Antioxidant 1076. Extensive lists of synonyms and trade names can be found in the OECD SIDS Initial Assessment Report (OECD SIDS, 2007) in ChemIDplus (2008).

## 2 Chemical properties, fate and toxicity

### 2.1 Properties and fate

Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate (CAS 2082-79-3) belongs to the class sterically hindered phenol antioxidants and acts as a radical scavenger by the donation of a hydrogen atom, forming a stable phenoxy radical.

The molecular structure of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate is shown in Figure 1. This ester protects materials against thermo-oxidative degradation. It is a white powder produced by transesterification of stearyl alcohol and methyl 3,5-di-tert-butyl-4-hydroxyphenylpropionate.

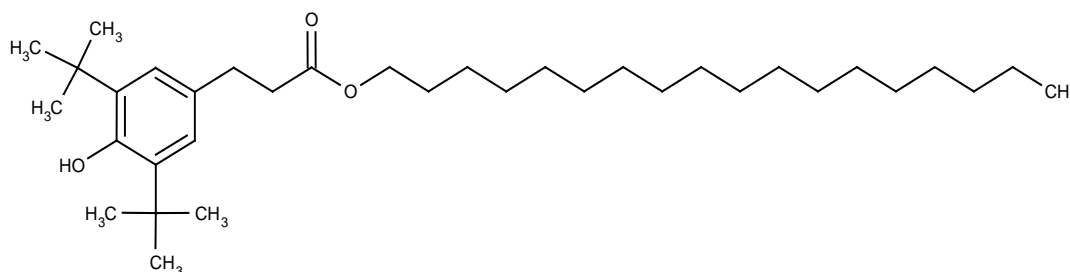


Figure 1. Molecular structure of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate.

The physico-chemical properties of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate are presented in Table 2. The data is retrieved from the OECD SIDS Initial Assessment Report, most of it has been generated by QSAR due to lack of experimental data (OECD SIDS, 2007). Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate has a high molecular weight, low water solubility, low vapour pressure, and a high log Kow.

Table 2. Physico-chemical properties of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate.

Property	Value
Molecular weight	531 g/mol
Physical state	Fine powder
Melting point	49-54 °C
Boiling point	561 °C (decomposes > 200 °C)
Vapour pressure	5.5E-7 hPa (25 °C)
Water solubility	2.85 µg/l (20 °C)
Log Kow	13.4
Log Koc	6.6-8.9
pKa	11.7

OECD SIDS (2007)

It can thus be expected to be found bound to solids, which is also demonstrated by fugacity modelling using EPI Suite v3.20, see Table 3. The results of the model exercise show that after equal releases of 1000 kg/h to air, water and soil, almost 99% of the chemical is expected to be

found in soil and sediments, minor amounts will partition to water and negligible amounts will be found in air.

Table 3. Predicted distribution and half-lives in each medium after equal releases to air, water and soil. Fugacity model level III, EPI Suite v3.20.

Compartment	Percentage in each medium	Half-life (h)
Air	0.04	6
Water	1.3	1400
Soil	33	2900
Sediment	66	13000

As can be seen from the results of the model exercise, estimated half-lives suggest that octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate is relatively persistent (Table 3). Estimated half-life in sediment, the compartment to which the major fraction of the discharged substance is expected to partition to, is more than 540 days. The persistent nature of the substance is also indicated by the results from degradation tests. Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate is not readily biodegradable, but 32-35% biodegradation measured as CO<sub>2</sub> evolution (OECD 301 B) and 21-39% measured as biological oxygen demand (OECD 301 C) after 28 days have been found (OECD SIDS, 2007).

Several degradation products of the antioxidant have been identified. In the OECD tests, 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid and 1-octadecanol were shown to be the major degradation products. Fischer *et al.* (1999) identified 7,9-di-tert-butyl-1-oxaspiro[4,5]deca-6,9-dien-2,4-dion as a major degradation product in waste compost and in soil, and the methyl ester of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid in activated sludge. Skjevraak *et al.* (2003) found migration of 2,6-di-tert-butyl-benzoquinone from high-density polyethylene (HDPE) drinking water pipes when filled with stagnant water for 72 hours (concentrations in the water varied between 60 and 600 ng/l). This potential degradation product of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate and the antioxidant Irganox 1010 (CAS 6683-19-8; the neopentetetrayl ester of 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionic acid), has also been found in biofilms lining HDPE pipes with turbulent water flows (Skjevraak *et al.*, 2005). This metabolite, 2,6-di-tert-butyl-benzoquinone, may also be a transformation product of 2,6-di-tert-butyl-phenols. The reaction is not reversible but the 2,6-di-tert-butyl-1,4-benzoquinone may be reduced to the corresponding 2,6-di-tert-butyl-1,4-hydroxybenzene (Paxeus 1996; Lopez-Avila and Hites 1981; Oros and David 2002).

## 2.2 Toxicity

Based on animal studies, octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate is considered to be of low hazard potential for human health (OECD SIDS, 2007). Available data indicate that the chemical is of low acute toxicity, that it is not skin or eye irritating, it is neither mutagenic nor carcinogenic, and it is not toxic for reproduction.

For the aquatic environment, only acute toxicity testing has been done (OECS SIDS, 2007). In two acute fish toxicity tests, no effects could be seen at concentrations far above the solubility of the substance. The same results were seen in an acute test with the invertebrate *Daphnia magna*. In a 72 h algae test, inhibition of biomass could be observed, but all tested concentrations in this study were far above the substance water solubility (ECB10: < 0.5-1 mg/l).

The bioaccumulation potential for octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate is not clear (OECD SIDS, 2007). Estimated bioconcentration factors (BCF) vary between 3 and 25000 depending on model used. For the only experimentally derived BCF found in the literature, exposure concentration was above water solubility. Recalculation based on the water solubility of the substance gave a BCF value of 1473 (OECD SIDS, 2007), indicating that the substance could be regarded as having a “moderate” bioaccumulation potential.

## 2.3 Human exposure and metabolism

Human exposure to octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate may take place through leachage of the chemical to foodstuffs and water. One animal study, reviewed in the OECD SIDS Initial Assessment Report, has shown that, once ingested, the substance can be taken up from the gastro-intestinal tract (OECD SIDS, 2007). The results from this study showed that 7 days after a single orally administered dose, 35% had been eliminated through urine and 61% through faeces, and low concentrations could still be found in different tissues.

No data on metabolic pathways are available, but it has been shown in rats that octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate is a potent inducer of several hepatic enzymes involved in xenobiotic metabolism (OECD SIDS, 2007).

## 3 Production, use and emissions

Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate is produced by transesterification of stearyl alcohol and methyl 3,5-di-tert-butyl-4-hydroxyphenylpropionate (OECD SIDS, 2007). In 2004, the world wide production was about 50000 tonnes. In Sweden, the total use seems to be fairly constant, whereas the number of preparations containing octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate is increasing (SPIN, 2008), see Figure 2.

The antioxidant is almost exclusively used in the manufacture of chemicals, chemical products, rubber and plastic products. Minor amounts are also used in forestry and in the manufacture of pulp, paper and paper products (SPIN, 2008).

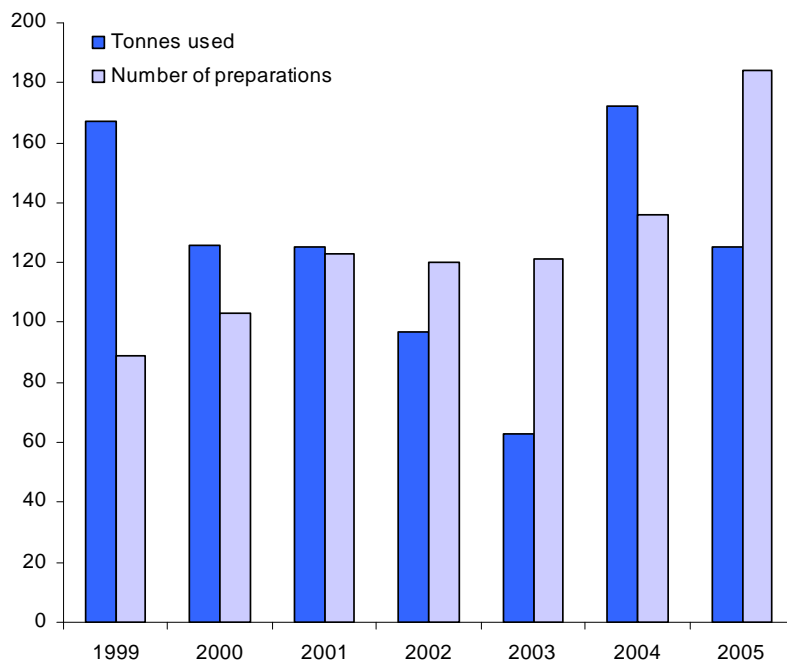


Figure 2. Total use and number of preparations containing octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate in Sweden 1999-2005 (SPIN, 2008).

Due to the use of antioxidants in materials used for food packaging, migration from these materials to food simulants has been investigated from a health perspective. Migration of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate from low density polyethylene into food simulants have been shown by for example Jeon *et al.* (2007). The migration levels found are not considered a risk for human health, but they indicate the potential for leachage of the antioxidant to the environment during products use and waste disposal. Loss of the antioxidant from materials to other compartments has also been shown in a few studies. In the study by Fisher *et al.* (1999), releases from polypropylene to activated sludge, soil, waste compost, and disposal site material were shown. Möller and Gevert (1996) have shown that octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate diffuse from polyethylene to surrounding antioxidant free polyethylene relatively fast. Möller *et al.* (2001) attribute a 75% loss of the antioxidant from a polyethylene film, after 15 year use as an air and vapour barrier in a wall construction, to migration to surrounding materials.

## 4 Previous measurements in the environment

Data regarding environmental levels of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate is scarce. As previously discussed, the physical-chemical properties and usage patterns suggests environmental exposure through losses from products and leachage from landfills, with subsequent partitioning to solids such as sludge, sediments and soil. Interestingly the only environmental measurements found in the literature concerned emissions to atmospheric aerosols due to open plastic and refuse burning (Simoneit *et al.*, 2005). In Santiago, Chile, atmospheric concentrations ranged 7 - 132 ng/m<sup>3</sup> over the years 1997-2000. Since the model exercise (Table 3) predicted only

minor amounts of the chemical to partition to air, and open refuse burning is not a common practice in Sweden, air was not a compartment given priority in the present screening.

## 5 Sampling strategy and study sites

### 5.1 National screening programme

A national sampling strategy was developed in order to determine concentrations of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate in different matrices in the Swedish environment.

The sampling programme was based on the identified possible emission sources and use, the predicted environmental distribution, and the limited available experimental data on the behaviour of the chemical. The sampling programme is summarised in Table 4, and details of the samples are given in Appendix 1.

Emissions of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate is predicted to be mainly due to losses from products during use and waste disposal. Once in the environment, the high molecular weight, low estimated vapour pressure and high estimated hydrophobicity, results in a predicted distribution with almost 99% of the substance partitioning to solids. Soil, sludge and sediments were thus considered important matrices. The occurrence in biota was investigated in fish (*Perca fluviatilis*) samples.

In order to determine background levels, samples of surface water, sediment and fish from three background lakes used within the national monitoring programme of contaminants in biota, were analysed ([www.nrm.se](http://www.nrm.se)). A pooled soil sample from the area of Lake Gårdsjön, a background site at the Swedish west coast was also included in the sampling program.

Since no industrial point source could be identified, the sampling programme for diffuse sources in the urban environment was expanded. These samples included surface water, soil, sediment and fish from the city of Stockholm, and sewage treatment plant (STP) sludge and water samples from both Stockholm and other municipal STPs. Since octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate is mainly used in rubber and plastics, materials extensively found in constructions and products used in the urban environment, storm waters from the city of Stockholm were also included in the sampling programme.

Due to possible losses of the chemical from waste material, landfills and waste treatment plants were identified as potential point sources. Leachate water and sludge from two waste treatment plants and one landfill were thus included in the sampling programme. For these sources, there were difficulties obtaining sludge samples. Only two out of six samples were obtained due to low sedimentation rates in the leachate water recipients. To compensate for this, the sampling programme for diffuse sources was expanded with an additional four STP sludge samples.

Table 4. National sampling programme

Site	STP waters	Sludge	Storm/Leachate waters	Surface water	Sediment	Soil	Fish	Total
<b>Background</b>								
Gårdsjön						1		1
National background lakes				3	3		3	9
<b>Diffuse sources</b>								
National municipal STPs	2	6						8
Stockholm	4	4	4	3	3	3	3	24
<b>Point source</b>								
Högbytorp landfill			2					2
Kovik waste treatment plant		1	1					2
Löt waste treatment plant		1	2					3
<b>Total</b>	<b>6</b>	<b>12</b>	<b>9</b>	<b>6</b>	<b>6</b>	<b>4</b>	<b>6</b>	<b>49</b>

## 5.2 Regional screening programme

A regional screening programme was carried out by different Swedish county administrative boards that had the possibility to collect and send samples to IVL for analysis. In the case of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate, 4 county administrative boards participated with a total of 15 samples consisting of 4 sludge and 7 water samples from sewage treatment plants, 1 sediment sample, and 3 landfill leachate water samples. Details of the samples are shown in Appendix 2.

## 6 Methods

### 6.1 Sampling

The staff at the different sewage treatment plants collected de-watered **sludge** samples from the anaerobic chambers. The sludge was transferred into glass jars and stored in a freezer (-18°C) until analysed. **Influent** and **effluent waters** were sampled in 1l glass bottles.

Surface **sediment** (0-2 cm) samples were collected by means of a Kajak sampler. The sediment was transferred into muffled (400°C) glass jars and stored in a freezer (-18°C) until analysed. Three sediment samples from the national background lakes were provided from the specimen bank at the Swedish Museum of Natural History.

The **fish** samples were collected by means of fishing net. The net fishing was approved by the fishery authorities in Stockholm and the ethical board for animal testing in northern Stockholm (D. no. 572/07). From the total catch approximately 10 individuals of Perch (*Perca fluviatilis*) were selected, representing the second-fifth year classes.

The fish samples were individually wrapped in cleaned aluminium foil and stored in a freezer (-18°C) until analysed. Fish muscle was dissected for analysis by means of solvent washed scalpels. Samples of dissected fish muscle from three national background lakes were provided from the specimen bank at The Swedish Museum of Natural History. These samples were stored at -18°C in pre-cleaned glass jars.

Surface water samples from background lakes were provided by the Swedish Museum of Natural History. Surface water samples from the city of Stockholm were sampled in glass bottles.

A pooled surface Soil (0-2 cm) sample from a background area was collected in a glass jar.

Storm water samples, collected during periods of sufficient precipitation to allow sampling, were provided by "Vägverket" and "Gatukontoret", Stockholm.

Leachate water and leachate water sludge samples were collected in 11 glass bottles and glass jars.

## 6.2 Analysis

### 6.2.1 General remarks

Octadecyl 3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)propionate is an additive used in plastic materials and this may potentially cause contamination of samples (Grosjean and Logan, 2007). Thus, plastic equipment *e.g.* disposable solid phase extraction columns were avoided. In this investigation clean-up columns were prepared using glass columns (Pasteur pipettes) and the stationary phase (silica gel) was heated (400°C) prior to use. Otherwise, we experienced no problems concerning background levels in the field- and analytical blank samples.

### 6.2.2 Water samples

Acidified surface water, influents to and effluents from STPs, leachate, and storm waters were filtrated on pre-heated (400°C) glass fibre (GF/C) filters prior to solid phase extraction. Filtered samples were spiked with recovery standard (3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)propionic acid propyl ester; synthesised in this laboratory by esterification of propyl alcohol and 3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)propionic acid) and extracted onto C<sub>18</sub> cartridges activated with acetone and phosphate buffer (0.05 M; pH 2). Sample volume varied between 200 and 1000 ml depending on sample type. The analytes were subsequently eluted with acetone and a mixture of hexane and MTBE (1:1). The acetone was removed by extraction with acidified water. The GF/C filters were acidified with 100 µl 3M H<sub>3</sub>PO<sub>4</sub> and extracted with acetone. The acetone extracts were diluted with acidified ultra pure water and extracted with a mixture of hexane and MTBE (9:1). The resulting extracts from water and filter were combined and cleaned-up on silica gel columns prior to GC-MS analysis.

### 6.2.3 Sediment, sludge and soil samples

Freeze dried sediment, soil and sludge samples were spiked with recovery standard (3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)propionic acid propyl ester), acidified and extracted with acetone. Sample size varied between approximately 1 and 4 g (dw) depending on sample type. Acetone extracts were



diluted with acidified ultrapure water and extracted with a mixture of hexane and MTBE (9:1) to remove the acetone. Prior to gas chromatography-mass spectrometry (GC-MS) analysis the extracts were cleaned-up on silica gel columns.

## 6.2.4 Fish samples

Fish samples (muscle, 10 g) were spiked with recovery standard (3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)propionic acid propyl ester) and homogenised in acetone:MTBE. The extracts were safeguarded and the samples were extracted once more with hexane:MTBE. Acetone was removed by shaking the combined organic phase with water. The final extracts were dried, concentrated and the solvent changed to hexane followed by clean-up on an alumina (neutral) column prior to GC-MS analysis.

## 6.2.5 Instrumentation

The extracts were analysed on a 6890N gas chromatograph connected to a 5973N mass selective detector (Agilent). The injection, 1 µl, was done in splitless mode at 240°C. The fused silica capillary column (VF-5MS 30 m × 0.25 mm i.d. × 0.25 µm film thickness, Varian) was held at 45°C for 1 min., ramped 15°C/min to 200°C, 5°C/min to 300°C, and held at 300°C for 9 min. Helium was used as carrier gas. The detector was used in selected ion monitoring mode (SIM) with electron ionisation energy of 70 eV. The analytes were identified by their characteristic retention times and one target ion used for quantification. One or two qualifier ions (Q-ion) were used to increase specificity of the detection, (Table 5). Quantification was based on comparison of the peak area to the known peak area of the standard analytes.

Table 5. Target ions (Tgt-ion) and qualifier ions (Q-ion) utilised in the MS-quantification

Compound	Tgt-ion	Q-ion 1	Q-ion 2
	m/z	m/z	m/z
3(3,5-di- <i>tert</i> -butyl-4-hydroxyphenyl)-propionic acid propyl ester <sup>a</sup>	305	320	-
3(3,5-di- <i>tert</i> -butyl-4-hydroxyphenyl)-propionic acid octyl ester <sup>b</sup>	530	531	515

Explanations: <sup>a</sup>recovery; standard <sup>b</sup>analyte; *m/z* the mass of the ion divided by its charge.

## 6.2.6 Quality control

The following quality criteria were used to ensure correct identification and quantification of the target compound: (a) the retention times should match those of the standard compounds within ± 0.05 min., (b) the intensity ratios (GC-MS) of the selected target- and qualifier ions should be within ± 15% of the expected / theoretical value (b) the signal-noise ratios should be greater than 3:1 (Haglund et al., 1993).

Internal standard (recovery standard added to the samples) approach were used for quantification, thus all reported values are recovery-corrected according to the recovery standard. Acceptable recovery rates for the added recovery standards were accepted if the recovery was >50 %. Field blanks were exposed to the environments at each sampling station (Keith, 1991). Method blanks and field blanks were included for each sample batch analysed to assess background interferences and possible contamination of the samples. Concentrations below field blank levels were treated as below limit of detection.

The limit of detection (LOD) was defined as 3 times the blank samples noise but in consideration of the actual sample noise in the target analytes “retention window” in the chromatograms. Possible background levels of analytes were subtracted from measured sample values (Keith 1991; Miller and Miller 1993).

## 7 Results and discussion

The results from the measurements of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate are given in Appendix 1 (national) and Appendix 2 (regional). Below, results from the national and the regional screening programmes are presented together.

### 7.1 Background areas

Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate was not detected in any of the samples (surface water, sediment, soil, fish) from the background areas. The detection limits for the different sample types are presented in Table 6.

Table 6. Detection limits for octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate in the different sample types analysed.

Sample type	Limit of detection
Soil	0.24 µg/kg dw
Waters	0.001 µg/l
Sediment	0.24 µg/kg dw
Fish	0.02 µg/kg ww

Explanations: dw = dry weight

### 7.2 Diffuse sources

#### 7.2.1 Sewage treatment plants (STPs)

Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate was found in all the included sludge samples from municipal sewage treatment plants (STPs), see Figure 3. Concentrations were in the range 0.62-17 µg/kg dw, with a mean concentration of 7.5 µg/kg dw. Sludge from Henriksdal and Käppala were sampled at two different occasions, values presented in figure 3 represents mean values.

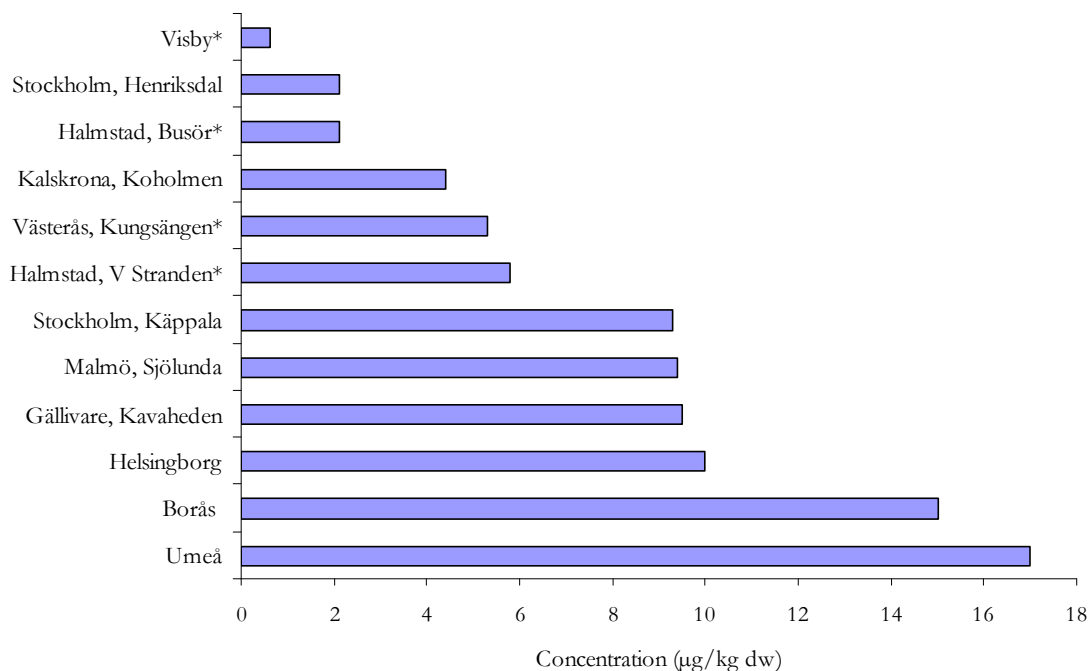


Figure 3. Concentrations of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate in sludge samples.  
\*Samples from the regional screening programme.

The substance was also detected in all the analysed STP influent water samples, whereas in effluent waters concentrations were below the limit of detection in 3 out of 7 samples, see Table 7. The concentration in effluent waters were, except Kungsängen (Västerås), lower than in influent water. Thus, removal of the substance from the water phase takes place during the waste water treatment process, plausible by absorption to sludge.

Table 7. Concentrations of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate in STP water samples.

STP	Influent (µg/l)	Effluent (µg/l)
Nyköping, Brandholmen <sup>a</sup>		0.0022
Halmstad, Busör <sup>a</sup>	0.0024	< 0.001
Stockholm, Henriksdal	0.011	0.0010
Västerås, Kungsängen <sup>a</sup>	0.0048	0.0071
Stockholm, Käppala	0.0037	0.0029
Umeå	0.016	< 0.001
Halmstad, V Stranden <sup>a</sup>	0.013	< 0.001

<sup>a</sup> Samples from the regional programme

## 7.2.2 Urban environment

The city of Stockholm was chosen for an extended screening for diffuse sources in the urban environment. Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate was found in all matrices from the studied Stockholm STPs, Figure 3 and Table 7. Sludge was sampled at two occasions and the variation in concentrations was found to be low; the concentrations in sludge samples from Henriksdal and from Käppala were 1.8 and 2.3 µg/kg dw, and 8.8 and 9.7 µg/kg dw, respectively.

Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate was also found in three out of four storm water samples, see Table 8. Concentrations were relatively high, in the same range or higher than for the STP influent waters.

Table 8. Concentrations of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate in storm water samples from the city of Stockholm

Sampling site	Concentration (µg/l)
Eugeniätunneln	< 0.001
Torstenssonsgatan	0.038
Starrängsringen	0.0053
Sveavägen	0.11

Leachage of the substance from materials and products in use in the urban environment are thus taking place.

Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate was also found, just above the detection limit, in two out of three fish samples, see Table 9. Biota is thus exposed to the antioxidant. In the other sampled matrices from the urban environment; surface water, sediment and soil, octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate above the detection limits could not be found. A water concentration of 0.05 ng/l can be estimated from a concentration of 0.07 µg/kg ww in fish tissue and the BCF 1473 found (OECD SIDS, 2007). This is 20 times lower than the detection limit for water samples. Thus among the matrices sampled analysis of fish samples seems to be the most sensitive method for the detection of this specific chemical in the environment.

Table 9. Concentrations of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate in fish samples from the city of Stockholm.

Sampling site	Concentration (µg/kg ww)
Årstaviken	<0.02
Stora Essingen	0.07
Riddarfjärden	0.04

Explanation: ww = wet weight.

## 7.3 Point sources

Leachate waters and sludge from waste treatment plants and landfills were analysed to study the potential leachage of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate from products during waste disposal. The substance was found in leachate waters from Högbytorp landfill, and in sludge from the Löt waste treatment plant, see Table 10.

Table 10. Concentrations of octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate in water and sludge from landfills and waste treatment plants.

Site	Leachate water ( $\mu\text{g/l}$ )	Sludge ( $\mu\text{g/kg}$ )
Björshult waste treatment plant*	< 0.001	-
Högbytorp landfill influent	0.0022	-
Högbytorp landfill effluent	0.0065	-
Kovik waste treatment plant	< 0.001	< 0.24
Löt waste treatment plant	< 0.001 (1)	3.1
	< 0.001 (2)	-
Slottsmöllan landfill*	< 0.001 (1)	-
	< 0.001 (2)	-

\*Samples from the regional program.

(1) Leachate from closed waste treatment plant/landfill.

(2) Leachate from waste treatment plant/landfill in use.

## 8 Conclusions

Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate occurred in all sludge samples included in the screening.

The substance was found in STP influent and effluent waters. The concentrations decreased during the sewage treatment process.

The substance was also found in storm waters, and in leachate waters and leachate water sludge from waste treatment plants and landfills. This indicates that emissions to the environment may occur through leachate from products and/or materials during use and disposal.

Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate was also detected, just above the detection limit, in fish samples from the urban environment. Biota is thus exposed to the antioxidant.

Using the bioconcentration factor found in the literature and the concentrations found in fish, a water concentration approximately 20 times lower than the detection limit can be estimated. Thus among the matrices sampled, analysis of fish samples seems to be the most sensitive method for the detection of this specific chemical in the environment.

Since octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate, and also other antioxidants of similar structure, are used in relatively large amounts, it may in the future be of interest to also study those similar substances, and also their degradation products.

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**Appendix 1: Information on samples and results from the National Screening Programme**

Category	Sample ID	Matrix	Site	Notes	Sampling date	Conc	Unit	DW (%)	Lipid (%)
Background	6036	Soil	Gårdsjön		2007-09-13	<0.24	µg/kg DW	24.4	
Background	6226	Sediment	Bysjön, Årjäng		2007-09-06	<0.24	µg/kg DW	15.0	
Background	6227	Sediment	Spjutsjön, Falun		2007-09-03	<0.24	µg/kg DW	7.0	
Background	6228	Sediment	Gipsjön, Malung		2007-09-04	<0.24	µg/kg DW	5.5	
Background	6224	Surface water	Spjutsjön, Falun		2007-09-03	<0.001	µg/l		
Background	6225	Surface water	Gipsjön, Malung		2007-09-04	<0.001	µg/l		
Background	6223	Surface water	Bysjön, Årjäng		2007-09-06	<0.001	µg/l		
Background	6874	Fish	Spjutsjön, Falun	Perch	2007-09-03	<0.02	µg/kg ww		0.75
Background	6873	Fish	Gipsjön, Malung	Perch	2007-09-04	<0.02	µg/kg ww		0.61
Background	6872	Fish	Bysjön, Årjäng	Perch	2007-09-06	<0.02	µg/kg ww		0.79
Diffuse, STP	6896	Sludge	Umeå		2008-04-02	17	µg/kg DW	35.8	
Diffuse, STP	6608	Sludge	Malmö, Sjölanda		2007-11-01	9.4	µg/kg DW	25.7	
Diffuse, STP	6847	Sludge	Stockholm, Käppala		v2 2008	8.8	µg/kg DW	24.0	
Diffuse, STP	6844	Sludge	Stockholm, Käppala		2007-12-20	9.7	µg/kg DW	25.4	
Diffuse, STP	6249	Sludge	Karskrona, Koholmen		2007-11-18--25	4.4	µg/kg DW	18.7	
Diffuse, STP	6584	Sludge	Stockholm, Henriksdal		2007-10-25	1.8	µg/kg DW	99.7	
Diffuse, STP	6035	Sludge	Stockholm, Henriksdal		V36 2007	2.3	µg/kg DW	98.6	
Diffuse, STP	6596	Sludge	Helsingborg		2007-10-29-- 11-04	10	µg/kg DW	18.7	
Diffuse, STP	6231	Sludge	Gällivare, Kavahedens		2007-09-20--26	9.5	µg/kg DW	17.1	
Diffuse, STP	6909	Sludge	Borås		2008-03	15	µg/kg DW	21.3	
Diffuse, STP	6891	Influent	Umeå		2008-03-27	0.016	µg/l		
Diffuse, STP	6764	Influent	Käppala		2007-02-05	0.0037	µg/l		
Diffuse, STP	6560	Influent	Stockholm, Henriksdal		2007-10-25	0.011	µg/l		
Diffuse, STP	6892	Effluent	Umeå		2008-03-27	<0.001	µg/l		
Diffuse, STP	6765	Effluent	Stockholm, Käppala		2007-12-06	0.0029	µg/l		
Diffuse, STP	6563	Effluent	Stockholm, Henriksdal		2007-10-25	0.0010	µg/l		
Diffuse, urban	6360	Surface water	Stockholm Årstaviken		2007-10-03	<0.001	µg/l		

Category	Sample ID	Matrix	Site	Notes	Sampling date	Conc	Unit	DW (%)	Lipid (%)
Diffuse, urban	6357	Surface water	Stockholm Stora Essinge		2007-10-03	<0.001	µg/l		
Diffuse, urban	6363	Surface water	Stockholm Riddarfjärden		2007-10-03	<0.001	µg/l		
Diffuse, urban	6803	Storm water	Stockholm Torstenssonsgatan		2007-12-13	0.038	µg/l		
Diffuse, urban	6805	Storm water	Stockholm Sveavägen		2007-12-13	0.11	µg/l		
Diffuse, urban	6804	Storm water	Stockholm Starrängsringen		2007-12-13	0.0053	µg/l		
Diffuse, urban	6614	Storm water	Stockholm Eugeniattunneln		2007-11-07	<0.001	µg/l		
Diffuse, urban	6464	Soil	Årstafältet		2007-10-15	<0.24	µg/kg DW	70.0	
Diffuse, urban	6459	Soil	Vårberg		2007-10-12	<0.24	µg/kg DW	61.0	
Diffuse, urban	6493	Soil	Humlegården		2007-10-17	<0.24	µg/kg DW	77.3	
Diffuse, urban	6361	Sediment	Stockholm Årstaviken		2007-10-03	<0.24	µg/kg DW	16.1	
Diffuse, urban	6358	Sediment	Stockholm Stora Essingen		2007-10-03	<0.24	µg/kg DW	13.3	
Diffuse, urban	6364	Sediment	Stockholm Riddarfjärden		2007-10-03	<0.24	µg/kg DW	19.6	
Diffuse, urban	6362	Fish	Stockholm Årstaviken	Perch	2007-10-03	<0.02	µg/kg ww		0.74
Diffuse, urban	6359	Fish	Stockholm Stora Essinge	Perch	2007-10-03	0.07	µg/kg ww		0.87
Diffuse, urban	6365	Fish	Stockholm Riddarfjärden	Perch	2007-10-03	0.04	µg/kg ww		0.57
Point sources	6400	Leachate water sludge	Löt waste treatment plant Vallentuna	finished 1995	2007-10-09	3.1	µg/kg DW	8.6	
Point sources	6483	Leachate water sludge	Kovik waste treatment plant, Värmdö		2007-10-15	<0.24	µg/kg DW	11.8	
Point sources	6399	Leachate water	Löt waste treatment plant, Vallentuna	new	2007-10-09	<0.001	µg/l		
Point sources	6396	Leachate water	Löt waste treatment plant, Vallentuna	finished 1995	2007-10-09	<0.001	µg/l		
Point sources	6461	Leachate water	Kovik waste treatment plant, Värmdö		2007-10-15	<0.001	µg/l		
Point sources	6541	Leachate water	Landfill Högbytorp	effluent	2007-10-23	0.0065	µg/l		
Point sources	6539	Leachate water	Landfill Högbytorp	influent	2007-10-23	0.0022	µg/l		

**Appendix 2: Information on samples and results from the Regional Screening Programme**

Sample ID	County	City	Site	Matrix	Sampling date	Conc	Unit	DW (%)
6304	Gotland (I)	Gothem	Gothemsån	Sediment	2007-10-01	<0.24	µg/kg DW	69.2
6302	Gotland (I)	Visby	Visby STP	Sludge	2007-09-25 - 10-02	0.62	µg/kg DW	95.0
6204	Halland (N)	Halmstad	Slottsmöllan landfill, in use	Leachate water	2007-09-20	<0.001	µg/l	
6205	Halland (N)	Halmstad	Slottsmöllan landfill, discontinued	Leachate water	2007-09-20	<0.001	µg/l	
6318	Halland (N)	Halmstad	Busör STP	Influent	2007-09-24 -10-02	0.0024	µg/l	
6319	Halland (N)	Halmstad	Busör STP	Sludge	2007-09-24 -10-02	2.1	µg/kg DW	17.8
6320	Halland (N)	Halmstad	Busör STP	Effluent	2007-09-24 -10-02	<0.001	µg/l	
6328	Halland (N)	Halmstad	V Stranden STP	Influent	2007-09-24 -10-01	0.013	µg/l	
6329	Halland (N)	Halmstad	V Stranden STP	Sludge	2007-09-24 -10 01	5.8	µg/kg DW	29.1
6330	Halland (N)	Halmstad	V Stranden STP	Effluent	2007-09-24 -10 01	<0.001	µg/l	
6336	Södermanland (D)	Nyköping	Björshult waste treatment plant	Leachate water	2007-10-01	<0.001	µg/l	
6337	Södermanland (D)	Nyköping	Brandholmens STP	Effluent	2007-09-25 -10-01	0.0022	µg/l	
6368	Västmanland (U)	Västerås	Kungsängens STP	Influent	2007-10-03	0.0048	µg/l	
6369	Västmanland (U)	Västerås	Kungsängens STP	Effluent	2007-10-03	0.0071	µg/l	
6374	Västmanland (U)	Västerås	Kungsängens STP	Sludge	2007-10-03	5.3	µg/kg DW	22.8