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Analysis of UV-filters (and fragrances) used in cosmetics and textiles

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Summary

A screening study concerning mainly substances used as UV-filters in sunscreen lotions has been performed. The sampling program was focused on water and sediment from bathing sites and effluent and sludge from WWTPs. Biota samples (fish muscle) were also included. Chemical analysis was carried out for 22 UV-filters (of which 18 are used mainly in cosmetic products and 4 in textiles) and 2 substances used as fragrances. In surface water from a lake with a bathing site, sampled at noon on a popular day for swimming, 13 UV-filters were found in a summed concentration of 170 ng/l. Dominating compounds were OC, DBENZO, BP3, HMS, 3BC and ethylhexyl¬triazone. The dominating compounds in sediment close to the bathing site were OC, DBENZO, BP3, EHS and UV360.

In WWTP effluents, all investigated substances, with the exception of BMDM and UV327, were detected. Highest individual concentrations were found for bemotrizinol (2500 ng/l), UV360 (650 ng/l) and DBENZO (560 ng/l). OC, BP, and OMC were also important and frequently detected components in the effluents. In WWTP sludge, OC and UV360 were found in highest concentrations (44 000 and 21 000 ng/g dw respectively), the maximum summed concentration was 99 000 ng/g dw. This is in the same range as what is often found as a summed concentration for phthalate plasticizers in sludge. Sediment sampled downstream the discharge point of a WWTP did not show as high concentrations of UV-filters as was found in the vicinity of the investigated bathing site. The highest levels of the UV-filter BP3 measured in sediments indicates a possible environmental risk.

To the best of our knowledge, ethylhexyltriazone, bemotrizinol, UV360 and iscotrizinol have not been measured before in the Swedish environment and appears to be new important contaminants at bathing sites and in WWTPs.

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Summary

A screening study concerning mainly substances used as UV-filters in sunscreen lotions has been performed. The two main routes for these UV-filters to the aquatic environment after use are direct releases from the skin during bathing (outdoor) or during showering/bathing or washing of textiles. In the latter case the substances of concern will be transported with waste water to a waste water treatment plant (WWTP) and eventually to its receiving water. Thus, the sampling program was focused on water and sediment from bathing sites and effluent and sludge from WWTPs. Biota samples (fish muscle) were also included.

Chemical analysis was carried out for 22 UV-filters (of which 18 are used mainly in cosmetic products and 4 in textiles) and 2 substances used as fragrances.

In surface water from a lake with a bathing site, sampled at noon on a popular day for swimming, 13 UV-filters were found in a summed concentration of 170 ng/l. Dominating compounds were OC, DBENZO, BP3, HMS, 3BC and ethylhexyltriazone. Water samples taken at increasing distances from the bathing site showed an influence area on the km scale for many of the substances. In the morning and evening of the same day the number of detected substances and the summed concentrations were lower (approx. 50 ng/l). The concentrations on one day before and one day after the bathing season were even lower (approx 10 ng/l).

The dominating compounds in sediment close to the bathing site were OC, DBENZO, BP3, EHS and UV360. Sediment samples from increasing distances showed an impact from the bathing site for most substances, also for UV360 and iscotrizinol, which could not be seen in the water column. The concentrations in sediments during the season did not vary much, but UV360 showed an accumulation from May to October.

In WWTP effluents, all investigated substances, with the exception of BMDM and UV327, were detected. Highest individual concentrations were found for bemotrizinol (2500 ng/l), UV360 (650 ng/l) and DBENZO (560 ng/l). OC, BP, and OMC were also important and frequently detected components in the effluents.

In WWTP sludge, OC and UV360 were found in highest concentrations (44 000 and 21 000 ng/g dw respectively), the maximum summed concentration was 99 000 ng/g dw. This is in the same range as what is often found as a summed concentration for phthalate plasticizers in sludge.

Sediment sampled downstream the discharge point of a WWTP did not show as high concentrations of UV-filters as was found in the vicinity of the investigated bathing site. The highest levels of the UV-filter BP3 measured in sediments indicates a possible environmental risk.

To the best of our knowledge, ethylhexyltriazone, bemotrizinol, UV360 and iscotrizinol have not been measured before in the Swedish environment and appears to be new important contaminants at bathing sites and in WWTPs.

Sammanfattning

En screeningundersökning som huvudsakligen omfattat substanser som används som UV-filter i solskyddskrämer har utförts. Huvudsakliga vägar för dessa substanser att transporteras ut i miljön är genom direkt avnötning från huden vid badning i en sjö, eller via transport med tvättvatten till ett reningsverk och vidare till recipienten. Provtagningsprogrammet fokuserades på ytvatten och sediment från badsjöar och utgående vatten och slam från kommunala reningsverk. Biotaprover (fiskmuskel) ingick också.

Kemisk analys gjordes av 22 UV-filter (av vilka 18 huvudsakligen används i kosmetiska produkter och 4 i textilier) och två doftämnen.

I ytvatten från en badsjö provtaget mitt på dagen under en dag med många badande detekterades 13 UV-filter. Den sammanlagda koncentrationen var 170 ng/l. Dominerande föreningar var OC, DBENZO, BP3, HMS, 3BC och ethylhexyltriazone. Vattenprov insamlade på ökande avstånd från badplatsen visade ett påverkansområde i storleksordningen km för många av substanserna. Samma dags morgon och kväll var antalet substanser och även den sammanlagda koncentrationen lägre (ca 50 ng/l). Koncentrationer i prover tagna före och efter badsäsongen var ännu lägre (ca 10 ng/l).

Dominerande föreningar i sediment nära badplatsen var OC, DBENZO, BP3, EHS och UV360. Sediment från längre avstånd visade en påverkan från badplatsen för de flesta substanserna, även för UV360 och iscotrizinol, vilket inte kunde ses i vattenfasen. Sedimentkoncentrationen varierade inte så mycket under säsongen, men för UV360 sågs en ökning från maj till oktober.

I utgående vatten från reningsverk kunde alla substanser med undantag av BMDM och UV327 detekteras. Högsta koncentrationer uppmättes för bemotrizinol (2500 ng/l), UV360 (650 ng/l) and DBENZO (560 ng/l). OC, BP och OMC hittades också frekvent.

I reningsverksslam hittades OC och UV360 i högst koncentrationer (44 000 respektive 21 000 ng/g ts). den högsta sammanlagda koncentrationen var 99 000 ng/g ts. Detta är i samma storleksordning som vad som ofta uppmäts som sammanlagd koncentration av ftalater i reningsverksslam.

Sediment som provtagits nedströms utsläppspunkten för ett kommunalt reningsverk uppvisade inte lika höga koncentrationer av UV-filter som i sediment från badplatsen. De högsta koncentrationer som uppmätts av UV-filtret BP3 i sediment är tillräckligt höga för att utgöra en risk för miljöpåverkan.

Så vitt vi känner till har ethylhexyltriazone, bemotrizinol, UV360 och iscotrizinol inte tidigare påvisats som föroreningar i svensk miljö.

1 Introduction

As an assignment from the Swedish Environmental Protection Agency, a screening study concerning substances used as UV-filters or fragrances in cosmetic products has been performed. UV-filters are used in sunscreen lotions to protect the skin against sunburn and skin cancer caused by the UV (ultra violet) radiation from sunlight. Also some UV-filters used mainly in textile applications were included.

Two routes for UV-filters in cosmetics to the aquatic environment after use are direct releases from the skin during bathing (outdoor), during showering/bathing or washing of textiles. In the latter case the substances of concern will be transported with waste water to a waste water treatment plant (WWTP) and eventually to its receiving water.

The overall objectives of this screening were to assess the occurrence of selected substances in surface water and sediment and to identify the importance of the different pathways. This study is a follow up on a previous screening from 2009 (Remberger et al 2011).

The national sample program was supplemented with a regional program where six county administrative boards participated.

The substances included in the study are divided into three groups. The substances in the basic group, 14 UV-filters and two fragrances are listed in Table 1. The second group included three UV-filters of the triazine type and the high molecular weight benzotriazole UV360, Table 2. Finally, a third group consisted of four benzotriazoles, UV-filters mainly used in textile applications, Table 3. (In an early stage of the project the substance groups in Table 2 and Table 3 were referred to as Option 2 and Option 1 respectively.)

Triclosan (2,4,4'-Trichloro-2'-hydroxy diphenyl ether, CAS 3380-34-5) was included as a reference compound that can be used as a tracer for WWTP effluent.

Table 1 Group 1, Basic group, UV-filters and fragrances (DHI and PTML) used in cosmetics.

Substance	Structure
CAS 70356-09-1 Avobenzone Butylmethoxydibenzoyl methane BMDM	H ₃ C CH ₃ CH ₁
CAS 118-60-5 2-Ethylhexyl salicylate EHS	H ₃ C OH

CAS 52253-93-7, 118-56-9 Homomenthyl salicylate 3,3,5-Trimethyl cyclohexyl salicylate Homosalate HMS	OH O CH ₃
CAS 15087-24-8 3-Benzylidene camphor 3-BC	H ₃ C H ₃ C
CAS 36861-47-9, 38102-62-4 4-Methyl benzylidene camphor Eusolex 6300 4-MBC	H ₃ C CH ₃ CCH ₃
CAS 131-57-7 Oxybenzone Benzophenon-3 BP3	CH ₃
CAS 131-56-6 2,4-Dihydroxybensophenone DHB	HO OH O
CAS 131-53-3 2,2´-Dihydroxy-4-metoxybensophenone DHM B	он о
CAS 21245-02-3 Ethylhexyl p-dimethylaminobenzoat EPABA	CH ₃ CH ₃ CH ₃
CAS 302776-68-7 Diethylamino hydroxybenzoylhexyl benzoate DBENZO	OH O O
CAS 71617-10-2 2-Hydroxy-4-methoxybenzophenone Isoamyl p-methoxycinnamate IMC	H ₃ C O CH ₃ CH ₃
CAS 5466-77-3 2-Propenoic acid, 3-(4-methoxyphenyl)-, 2- ethylhexyl ester Ethylhexyl methoxycinnamate Octyl methoxycinnamate OMC	CH ₃ CH ₃ CH ₃
CAS 6197-30-4 Octocrylene OC	СН

CAS 119-61-9 Benzophenone BP	
CAS 17092-92-1 Dihydroactinidiolide DHI	H ₃ C CH ₃
CAS 3910-35-8 Benzene, 1,1'-(3,3-dimethyl-1-butenylidene)bis 1,1,3-Trimethyl-3-phenylindan PTML	CH ₃

Table 2 Group 2, UV-filters of the triazine type and UV360.

Compound	Structure
CAS 88122-99-0 Ethylhexyl triazone	
CAS 187393-00-6 Bemotrizinol	
CAS 103597-45-1 Bisoctriazol UV360	N OH N N N N N N N N N N N N N N N N N N
CAS 154702-15-5 Diethylhexyl butamidotriazone Iscotrizinol	

Table 3 Group 3, Benzotriazoles. UV-filters used in textile applications

Compound	Structure
CAS 25973-55-1 2-(2H-Benzotriazol-2-yl)-4,6-di-tert-pentylphenol UV 328	H ₃ C CH ₃ N HO CH ₃ CH ₃ CH ₃
CAS 3147-75-9 2-(2H-Benzotriazol-2-yl)-4-(1,1,3,3- tetramethylbutyl)phenol UV 329	HO CH ₃ CH ₃ CH ₃ CH ₃
CAS 3864-99-1 2,4-Di-tert-butyl-6-(5-chlorobenzotriazol-2-yl)phenol UV 327	H ₃ C CH ₃ OH N CH ₃ CH ₃
CAS 2440-22-4 2-(Benzotriazol-2-yl)-4-methylphenol UV P	HO CH ₃

2 Chemical properties and use

Chemical properties of the substances included in the screening are summarized in Table 4. UV-filters generally have low water solubility, low vapour pressure, and high log Kow. They absorb UV light in the wavelength range 280-400 nm. To be able to cover the entire UVB (280-320 nm) and UVA (320-400 nm) region sunscreen products often contain several filters.

Table 4. Physical and chemical properties.

Substance	Log Kow	pKa	Reference			
BMDM	4.51	9.74	Rodil et al 2008			
EHS	5.97	8.13	Rodil et al 2008			
HMS	6.16	8.09	Rodil et al 2008			
3-BC	5.3	-	Ramos et al., 2015			
4-MBC	5.47	-	Giokas et al 2005			
BP3	3.79; 3.52	7.56	Rodil et al 2008; Jeon et al. 2006			
DHB	2.96	-	ChemIDplus, (2015)			
DHMB	3.82	1	ChemIDplus, (2015)			
EPABA	6.15	2.39	Rodil et al 2008			
DBENZO	6.54	1	Affected by pH. QSAR in Wernersson 2008			
IMC	4.33	1	ChemIDplus (2015)			
OMC	5.80	1	ChemIDplus (2015)			
OC	6.88	1	ChemIDplus (2015)			
BP	3.18	1	ChemIDplus (2015)			
DHI	2.2	-	PubChem.ncbi.hlm,hih.gov			
PTML	5.9	1	ChemIDplus (2015)			
Ethylhexyl triazone	14.5	-	PubChem.ncbi.hlm,hih.gov			
Bemotrizinol	8.8	1	PubChem.ncbi.hlm,hih.gov			
UV360	12.46	7.62	Montesdeoca-Esponda et al. 2013			
Izcotriazol	11.8	1	PubChem.ncbi.hlm,hih.gov			
UV328	7.25; 8.85	8.28	ChemIDplus (2015) Carpinteiro et al. 2010			
UV329	6.2	1	Thomas et al., 2013			
UV327	9.41	7.81	Carpinteiro et al. 2010			
UV P	4.31	4.30	ChemIDplus (2015), Carpinteiro et al. 2010			

Group 1

BMDM is a sunscreen with good coverage of the UVA-spectrum and is often combined with UVB-filters in sunscreen products. It has complexing properties and metal ions *e.g.* Fe(III) may therefore induce photochemical degradation. BMDM degrades in the sunlight and loses effectiveness over time.

The salicylic acid based sunscreen filters (*EHS*, *HMS*) absorb ultraviolet rays with a wavelength from 295 nm to 315 nm (UVB). They do not protect against UVA and are relatively weak sunscreen filters. When exposed to sunlight HMS undergoes some degradation¹.

Benzylidene camphor substances, (*3-BC*, *4-MBC*) are agents that absorbs in the UVB range. All the camphor sunscreens are supposed to dissipate the photon energy by cistrans isomerisation (Buser et al., 2005).

UV-filters based on bensophenone (*BP3*, *DHB*, *DHMB*, *DBENZO*) are typical broad-band filters and absorb both in UVA and UVB. Benzophenone filters have high stability to UV-light (BASF, 1995).

EPABA is used as an UVB blocker in sunscreens.

¹ www.smartskincare.com

Cinnamic acid derivatives (*IMC*, *OMC*) absorb ultraviolet radiation in the UVB range. When exposed to sunlight OMC is converted into a less UV absorbing form, which reduces its effectiveness. This conversion can be partly prevented by certain other UV blockers ². OMC is the most common UV-filter in sun lotions, used in over 90% of those available in Europe (Thomas et al, 2014).

OC is a highly stable sunscreen filter and does not degrade in sunlight. It protects other UV blocking agents from UV-induced degradation. It absorbs UVB, short UVA (UVA-2) but not long UVA. It is a relatively weak sunscreen, inadequate when used alone ³.

BP has a broad use as UV adsorbent e.g as photo initiator in UV-curing applications such as inks, imaging, and as clear coatings in the printing industry. Benzophenone prevents UV light from damaging scents and colours in products such as perfumes and soaps. It can also be added to plastic packaging as an UV blocker to prevent photodegradation of the packaging polymers ⁴.

Group 2.

The triazines *ethylhexyl triazone*, *bemotrizinol* and *iscotrizinol* are used in cosmetic products. *UV360* is not a triazine but have similar properties and usage and is therefore mentioned in this context. The substances in this group are extremely lipophilic (Table 4) and are resistant to UV-degradation. They are therefore also used as scavengers to other more UV-labile filters.

Group 3.

Benzotriazoles (*UV328*, *UV329*, *UV327*, *UVP*) are used as UV stabilizers in coated textiles. Benzotriazoles may to a large extent enter Sweden through imported finished goods.

The use in Sweden according to the SPIN (Substances in preparations in the Nordic countries) database for BMDM, BP3, EPABA, OMC, OC, BP, UV328, UV329, UV327 and UV P is illustrated in Figure 1. EHS, DHB and PTML have registered annual amounts of zero or one tonne. Registered amounts of HMS, 4MBC and DBENZO are confidential. 3BC, DHMB, IMC and DHI were not registered in 2011. The amount of compounds that are imported as ingredients in finished cosmetic products is not included in the SPIN database, therefore the actual consumption is most likely higher than the figures presented.

³ www.smartskincare.com

² www.smartskincare.com

⁴ https://en.wikipedia.org/wiki/Benzophenone

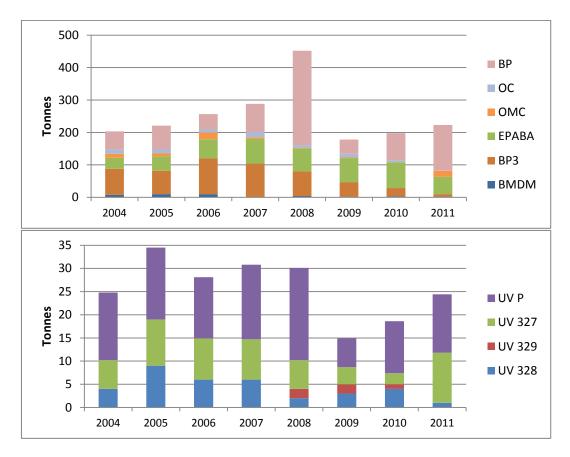


Figure 1 Use in Sweden for the years 1999 to 2011 according to the SPIN database (www.kemi.se).

3 Previous measurements in the environment

Many of the substances in group 1 were included in the previous screening by Remberger et al (2009), in some cases at the same or similar sampling spots as in the present study. Comparisons to the previous results are made in the results section.

The results will also be compared to a recent Norwegian study (Thomas et al 2014) where eight of the UV-filters (BP3, EPABA, OMC, OC, UV328, UV329, UV 327 and UV360) were determined.

4 Sampling program

The sampling program was focused on diffuse sources in urban environments. Effluent and sludge from Kungsängen WWTP, Uppsala were sampled together with surface water, sediment and fish from the receiving water, Fyrisån.

Surface water was sampled upstream (-1.7 km) and close to (5 m) the effluent discharge point. Samples were also taken 150 m downstream (ds 1) and 4.6 km downstream (ds 3) the discharge point. The annual average flow in Fyrisån is $8.6~m^3/s$ and the average effluent flow from Kungsängsverket is $2~200~m^3/h$ (Uppsala vatten, 2011), i.e. a mean dilution factor of 14.

Effluent and sludge from the following WWTPs were also included: Ön, Umeå, Henriksdal, Stockholm and Rya, Göteborg.

For sampling at, and in the vicinity of a bathing site, the Lake Rönningesjön, Täby (Figure 2) was selected. This lake has a length of approx. 2.2 km and a width of 0.2-0.3 km. The average and maximum depth is 3.4 and 4.7 m respectively. It has two popular bathing sites, one at "J", and one south west of "5". Surface water and sediment were sampled from a jetty (J) and from a boat in a gradient from the jetty (sampling points 1-6) during a popular day for swimming in August 2014. To get the daily variation, water samples from the jetty were collected in the morning and in the evening of the same day. Surface water and sediment were also sampled in May and October, before and after the bathing season.



Figure 2 Map showing Lake Rönningesjön with sampling points.

Background samples (surface water, sediment and fish) were taken from the Lake Tärnan, Vallentuna, within the framework of the national freshwater environmental monitoring and from Lake Tärnan which has no connection to effluents from WWTPs but may receive direct anthropogenic contamination from people in the region.

The sampling program is summarised in Table 5, and details such as sampling dates are given in Appendix 1.

Table 5 National sampling program

	WWTP Effluent	WWTP Sludge	Surface water	Sediment	Fish	Sum
Background						
Tärnan			1	1	1	3
Urban areas, WWTPs						
Kungsängen WWTP, Uppsala	1	1	4	4	3	13
Ön WWTP, Umeå	1	1				2
Henriksdal WWTP, Stockholm	1	1				2
Rya WWTP, Göteborg	1	1				2
Urban area, bathing site						
Varying distance from jetty			4	4		8
Different times of day			4			4
Different times of year			3	3		6
Sum	4	4	16	12	4	40

In addition to the national screening program Swedish county administrative boards had the opportunity to collect and send samples for analysis. Six counties participated with samples including influent, effluent and sludge from municipal WWTPs, surface water, sediment and fish. The number of samples is given in Table 6, details are given in Appendix 1. For some samples in the regional program analysis of the substances in groups 2 and 3 was not commissioned.

Table 6 Regional sampling program

	WWTP Influent	WWTP Effluent	WWTP Sludge	Surface water	Sediment	Fish	Sum
No of samples	2	6	4	7	4	4	27

5 Methods

5.1 Sampling

Surface water samples were collected using a Ruttner sampler and were transferred into 1 L pre-muffled glass bottles and acidified. In field and during the transport to the laboratory the samples were stored in darkness. At the laboratory the samples were stored in a fridge (4°C) until chemical analysis.

Surface **sediment** (0-2 cm) was collected using a Kajak sampler. The sediment was transferred into pre-muffled (400°C) glass jars and stored in a freezer (-18°C) until chemical analysis.

Fish from the background lake Tärnan (Perch; *Perca fluviatilis*) was collected by means of fishing net and dissected by the staff at Swedish Museum of Natural History. The muscle samples were stored at -18°C in pre-muffled (400°C) glass jars until chemical analysis.

The fish samples from River Fyrisån were collected using fishing net. From the total catch approximately 10 individuals of Perch (*Perca fluviatilis*) were selected, representing the second-fifth year classes. The fish samples were wrapped in pre-cleaned aluminium foil and stored in a freezer (-18°C) until dissection. Fish included in the regional sample program was delivered as whole fish. Fish muscle was dissected at IVL laboratory using solvent washed scalpels and were stored at -18°C in pre-muffled (400°C) glass jars until chemical analysis.

The staff at the different WWTPs collected **effluent water** (day samples). The samples were collected in 1 L glass bottles. When the samples arrived to the laboratory they were acidified and stored at -18° C until chemical analysis.

Sludge samples, from the anaerobic chambers, were taken by the staff at the different WWTPs. The sludge was transferred into muffled glass jars and stored at -18°C until analysed.

5.2 Chemical analysis

The substances in Table 1 and Table 3 were analysed by gas chromatography followed by tandem mass spectroscopy (GC-MS/MS), whereas the substances in Table 2 were analysed by liquid chromatography followed by tandem mass spectroscopy (LC-MS/MS).

5.2.1 GC-MS/MS analysis

5.2.1.1 Sample preparation

Water sample volumes varied between 200 and 1000 ml depending on sample type. The unfiltrated and acidified water was spiked with recovery standards and extracted using a solid phase extraction column (Isolut ENV+, 200 mg) with Filter aid 400 on the top (3.5 g). The cartridges were activated with toluene, methyl tert-butyl ether (MTBE), dichloromethane (DCM), methanol and Milli-Q water. Samples were applied to the SPE columns at a flow rate of 5 ml/min. Milli-Q water was used to wash the SPE column before eluting. The analytes were subsequently eluted with methanol, acetone, DCM and a mixture of hexane and MTBE (3:1). The final extract was diluted with acidified ultra-pure water and extracted with a mixture of toluene and MTBE (3:1).

Freeze dried sediment (2 g) or sludge (1 g) was acidified with phosphoric acid, spiked with recovery standard and extracted with 15 mL MTBE: acetonitrile(ACN):methanol (60:30:10) using Microwave-assisted extraction (MAE) with a time program of 10 minutes at 80°C. The methanol was washed away by shaking the extract with acidified water. The water was extracted twice with hexane:MTBE (1:1). The extracts were pooled and cleaned-up on "graphitised carbon black" (GCB) columns and subsequently fractionated on a silica gel column into two fractions F1 and F2 containing fragrance (DHI, PTML) and UV-filters respectively. The F1-fraction contained high amount of elementary sulphur which was removed with the aid of metallic cupper.

Fish muscle (10 g) was placed in a PTFE bomb, spiked with recovery standard, homogenised in ACN and extracted using Microwave-assisted extraction (MAE) with a time program of 15 minutes at 85°C. The sample was transferred to a glass test tube and sodium sulphate and sodium chloride was added. The sample was mixed and centrifuged. The extraction with ACN

was repeated. The pooled extract was diluted with water and extracted with hexan:MTBE and hexan:DCM. The extract was dried over sodium sulphate, concentrated and subjected to clean-up on a PSA (ethylenediamine-N-propyl) column.

5.2.1.2 Derivatization and instrumental analysis

The extract was reacted with the silylating reagent MSTFA (*N*-methyl-*N*-trifluoroacetamide) and analysed for the substances in Table 1 and Table 3, with the exception of BMDM, using GC-MS/MS.

In a second step the MSTFA derivatised extracts were methylated according to Nagtegaal et al. (1997) with minor modifications. The extract was evaporated to dryness and re-dissolved in dry MTBE (dried over molecular sieve 5 Å). Methyl-iodide and NaH was added and the test tube was closed and shaken. The reaction was performed at 80°C for 2 hours. The test tube was cooled and water and hexane was added. After extraction the organic phase was dried, concentrated and transferred to a GC-vial and analysed for BMDM using GC-MS/MS analysis.

GC-MS/MS analysis was performed on a gas chromatograph (GC) coupled to a 7000A Triple Quad MS (Agilent Technologies, Inc. Santa Clara, CA, USA). The instrument was equipped with an auto injector (Agilent 7683B). The injection was in pulsed splitless mode (1 μ l) at 240°C. The detector was used in MRM mode with electron ionisation at energy of 70 eV. The fused silica capillary column (VF-5MS 30 m \times 0.25 mm i.d. \times 0.25 μ m 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 15°C/min to 325°C and held at 325°C for 2 min. Helium was used as carrier gas. The analytes were identified by their characteristic retention times and one product ion used for quantification. A second product ion was used as qualifier to increase specificity of the detection. Quantification was based on comparison of the peak area to the known peak area of the standard analytes.

5.2.2 LC-MS/MS analysis

5.2.2.1 Sample preparation

Water sample volumes varied between 200 and 1000 ml depending on sample type. The unfiltrated and acidified water was spiked with recovery standards and extracted using solid phase extraction (Isolute C18 –EC, 500 mg). The C18 cartridges were activated with DCM, acetone and Milli-Q water. Samples were applied to the SPE columns at a flow rate of 5 ml/min. Milli-Q water was used to wash the SPE column before eluting. The analytes were subsequently eluted with methanol, acetone, DCM and a mixture of hexane and MTBE (3:1). The final extract was diluted with acidified ultra-pure water and extracted with a mixture of toluene and MTBE (3:1). The solvent was changed to methanol prior to LC-MS analysis.

Freeze dried sediment (1 g d.w.) or sludge (0.5 g d.w) was acidified with phosphoric acid, spiked with recovery standard and extracted with 10 mL MTBE:DCM:methanol (45:45:10) using MAE with a time program of 15 minutes at 75°C. The methanol was washed away by shaking the extract with acidified water. The water was extracted twice with hexane:MTBE (1:1). The extracts were pooled and cleaned-up on PSA columns.

5.2.2.2 Instrumental analysis

LC-MS/MS analysis was performed using an UFLC liquid chromatography system (Shimadsu Scientific Instruments, Columbia, USA), equipped with an auto sampler, a quaternary pump, and on-line degassing system. The compound separation was performed with a reversed phase C8 column (Thermo Scientific, HvPURITY C8, 3.0 mm ID x 50 mm length, 5 μ m). Gradient elution was performed with 5 mM acetic acid and 5 mM ammonium acetate in water as solvent A and methanol as solvent B. The analytical detector was a triple quadrupole mass spectrometer API 4000 (Applied Biosystems, California, USA). The mass spectrometer interface was an electrospray ionization (EI) source. The instrument altered between positive and negative mode during detection. Instrument control and data processing were performed by the Analyst software.

5.2.3 Quality control

Possible memory effects were evaluated by a blank injection of hexane after standard samples of varying concentrations. Laboratory blank samples were included in each batch. Standards were analysed in a wide concentration range (5–500 ng/ml) and were used for evaluating the linearity, sensitivity - quantification limit (LOD) defined as 3 times the standard deviation of the blank, reproducibility of retention, precision as repeatability, and column stability. Method recoveries were determined by spiking the standard solution to matrix at the following concentration levels: Milli-Q water (100 ng/l), surface water (100 ng/l) and sewage effluent (1000 ng/l). Analyte addition was made with the criteria that the spiking would be at a level at least three times the original concentration in surface water and sewage effluent, respectively.

Samples were quantified using internal standard method. Several calibration standards covering the whole concentration range were measured before, in the middle and at the end of sample sequences. The maximum difference between results at quantification and qualification mass transition was set to 20% as criterion for positive identification.

UV-filters are additives used in personal care products which potentially may cause contamination of samples. Thus, these products were avoided when handling the samples. Furthermore, nitrile plastic gloves were used throughout the analytical work. The use of plastic materials was avoided. Glass equipment and silica gel was heated (400°C; 8 hours) prior to use. Using these precautions, we experienced no problems concerning background levels in analytical blank samples.

Sludge is a very complex matrix and contains a large number of (organic) substances. These "natural" organic substances may be present in very high concentrations which can interfere with the identification of the target substances. Therefore, both the extraction clean-up and instrumental analysis methods was adapted in order to address this problems especially with sludge samples. This worked well for all analytes determined with GC-MS/MS but not for the compounds in option 2 determined with LC-MS/MS. In this case the determination suffered serious matrix ionization suppression as a result of charge competition in the ESI source between compounds in the sample matrix and the target analytes. This effect resulted in diminished and irreproducible analytic response, but after dilution of the extracts we obtained reliable results.

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

 \pm 0.05 min., (b) the intensity ratios of the selected ions should be within \pm 15% of the expected / theoretical value and (c) the signal-noise ratios should be greater than 3:1 (Haglund et al., 1993).

Internal standard (known amount of a 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 were >50 %. Method blanks were included for each sample batch and analysed to assess background interferences and possible contamination of the samples.

The limit of detection (LOD) was defined as three times the blank samples noise but in consideration of the actual sample noise in the chromatogram retention windows of the target analytes. Possible background levels of analytes were subtracted.

6 Results and discussion

All individual results are tabulated in Appendix 2.

An overview of detection frequencies is given in Table 7 for substances in Group 1, in Table 8 for Group 2, and in Table 9 for Group 3.

Table 7 Detection frequencies in different sample types for substances in Group 1.

	Influent n=2	Effluent n=10	Sludge n=9	Surface w n=24	Sediment n=17	Fish muscle n=8
BMDM	50%	0%	0%	0%	0%	0%
EHS	100%	100%	100%	54%	41%	13%
HMS	100%	100%	100%	58%	35%	13%
3BC	100%	40%	0%	79%	0%	13%
4MBC	100%	30%	22%	29%	0%	0%
BP3	100%	100%	22%	54%	53%	13%
DHB	100%	100%	11%	25%	0%	0%
DHMB	100%	70%	0%	38%	0%	0%
EPABA	100%	40%	44%	33%	12%	0%
DEBENZO	100%	70%	56%	25%	24%	0%
IMC	50%	60%	0%	25%	0%	0%
OMC	50%	100%	100%	17%	35%	38%
OC	100%	100%	100%	67%	71%	38%
BP	100%	100%	100%	71%	88%	0%
DHI	100%	100%	100%	100%	100%	38%
PTML	100%	50%	100%	8%	47%	13%
Triclosan	100%	90%	100%	17%	6%	0%

Table 8 Detection frequencies in different sample types for substances in Group 2.

	Influent	Effluent	Sludge	Surface w	Sediment	Fish muscle
	n=0	n=6	n=6	n=21	n=15	n=0
Ethylhexyltriazone		33%	100%	19%	7%	
Bemotrizinol		83%	100%	43%	0%	
UV360		67%	100%	24%	53%	
Iscotrizinol		33%	100%	38%	67%	

Table 9 Detection frequencies in different sample types for substances in Group 3.

	Influent n=2	Effluent n=9	Sludge n=9	Surface w n=21	Sediment n=17	Fish muscle n=8
UV 328	100%	44%	100%	5%	12%	0%
UV 329	100%	44%	100%	38%	6%	0%
UV 327	0%	0%	100%	0%	0%	0%
UV P	100%	100%	89%	0%	29%	0%

6.1 Background

UV-filters were detected in low concentrations in surface water sampled in early September from the background lake Tärnan. 4MBC (13 ng/l), 3BC (5.3 ng/l) and OC (4.3 ng/l) accounted for 78 % of the summed concentration (29 ng/l). The lake is not totally unaffected by human activities; a few settlements exist within the drainage area. Another source could be air transport, but this has not been investigated. UV-filters were not detected in sediment or fish muscle from the lake.

6.2 Bathing sites

Water from Lake Rönningesjön was sampled at several occasions. Measured concentrations of UV-filters in water from the bathing site (taken from the jetty) in the morning, noon and evening on a popular day for swimming in August is illustrated in Figure 3, showing clearly higher concentrations at noon.

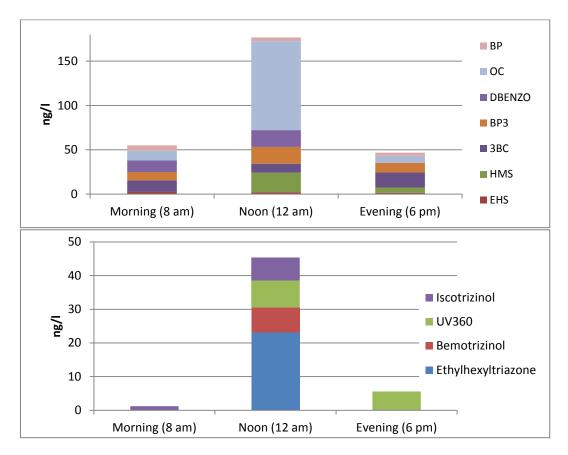


Figure 3 Lake Rönningesjön. Concentration in surface water at the jetty at different times on one day in August. Basic group (top), triazines (bottom).

Out of the 14 UV-filters in the basic group nine was found. Dominating substances were OC, DBENZO, BP3, HMS, 3BC and ethylhexyltriazone. All four substances in the triazine group (group 2) were detected. The diurnal variation indicates a quick turnover of the UV-filters in the water column presumable caused by wind powered mixing and/or degradation.

The noon sample from August is compared to samples from before and after the bathing season (May and October) in Figure 4. The concentrations in May and October were much lower, but 4MBC, EHS and BP could be detected before the bathing season in May. The extremely lipophilic UV360 and the triazines (Group 2) was not detected.

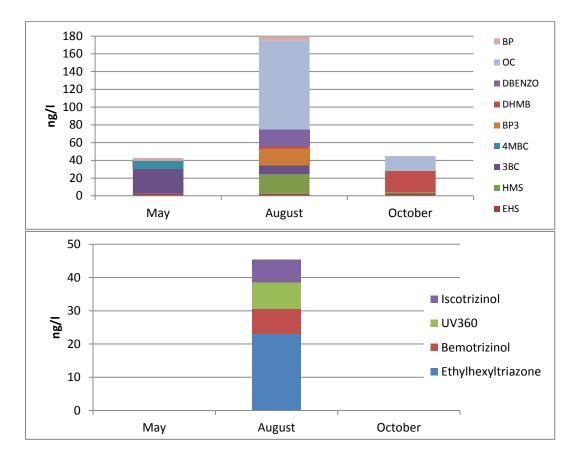


Figure 4 Lake Rönningesjön. Concentration in surface water at the jetty at different times of year. Basic group (top), triazines (bottom).

The UV-filters in Group 1 were measured at the same jetty in the screening 2009 (Remberger 2011). The composition then was quite similar to what was determined now but the concentrations were higher in 2009.

Concentrations of the UV-filters in the water at varying distances from the jetty (Figure 2, point 1-6) is shown in Figure 5. The concentration shows a decrease to point 1 but thereafter an increase to point 5, probably due to a second bathing site south west of this point. Through the whole gradient OC dominates but, at point 4, 3BC showed the highest concentration. These results indicate a transport of the UV-filters over large parts of the lake. The triazines showed a more local presence and were detected only in low concentrations away from the jetty.

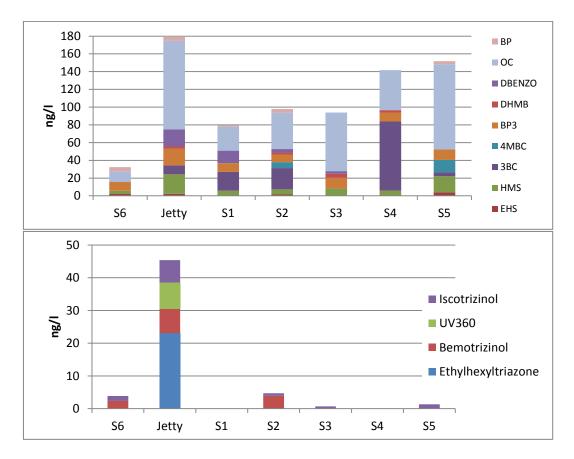


Figure 5 Lake Rönningesjön. Concentration in surface water in different areas of the lake in August. Basic group (top), triazines (bottom).

The fragrance DHI was detected in all water samples from Lake Rönningesjön. The concentrations were in the range 200-340 ng/l with a mean of 270 ng/l. The concentration was fairly equal throughout the lake and no difference during the day or seasons could be demonstrated. Contamination during the analytical procedures was suspected at first but could be ruled out since the analytical blanks could not support this theory. The fragrance PTML was only detected in one sample from Rönningesjön (22 ng/l).

The concentration of UV-filters in sediments from before, during and after the bathing season (May, August, October) are presented in Figure 6. The composition and concentration of UV-filters varied somewhat over the year but not in the same magnitude as in the water column. The dominating compounds were OC, DBENZO, BP3, EHS and UV360. While the concentration of the UV-filters (basic group) did not vary much during the season, UV360 showed a pronounced accumulation from May to October.

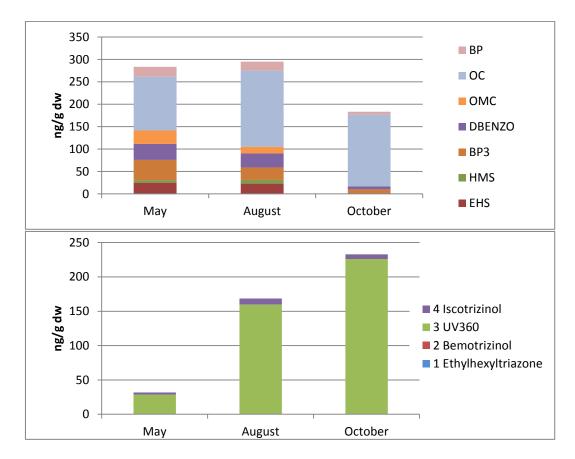


Figure 6 Lake Rönningesjön. Concentration in sediment at S2 at different times of year. Basic group (top), triazines (bottom).

Concentrations in the sediment at varying distances from the jetty (Figure 2, point S1–S5) are shown in Figure 7. Unlike the water gradient, the sediment gradient showed no pronounced impact from the second bathing site. Through the whole gradient OC dominated. BP3, DBENZO, EHS, BP, OMC and HMS were also detected in the whole gradient. The results indicate a transport of the UV-filters from the bathing sites (hot spots) over most of the lake. UV360 and, in lower concentration, iscotrizinol, were detected in the whole gradient unlike the situation in the water column.

Sediment concentrations were not measured in the previous screening concerning UV-filters in Lake Rönningesjön (Rembeger et al., 2011), so comparisons over time cannot be done.

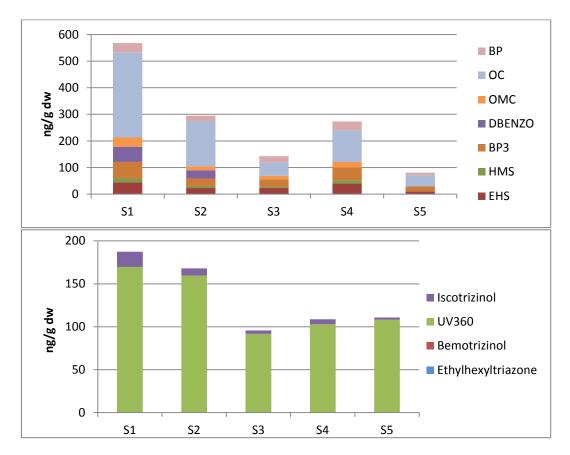


Figure 7 Lake Rönningesjön. Concentration in sediment in different areas of the lake in August. Basic group (top), triazines (bottom).

The fragrances DHI and PTML showed no clear concentration gradient in sediments from points S1–S5 in Lake Rönningesjön sampled in August. DHI was detected in all samples in the concentration range 220-420 ng/g dw (average 350 ng/g dw). The PTML concentration was significantly lower (4–21 ng/g dw).

The concentration of DHI in sediment from S2 was higher in October than in May and August (Figure 8). PTML showed low concentrations in May and August but was not detected in October.

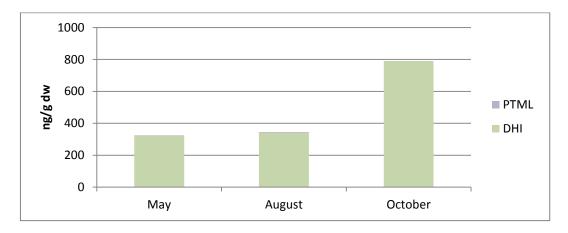


Figure 8 Lake Rönningesjön. Concentrations in sediment at S2 of fragrances at different times of year.

Concentrations of the UV-filters in surface water from the different bathing sites in the regional sampling program are illustrated in Figure 9. The concentrations were lower compared to the concentrations in Lake Rönningesjön during daytime in the bathing season (Figure 5) but in the same range as in the morning and evening samples (Figure 3). The composition was also different. OC was not a dominating component as in Lake Rönningesjön. Instead the more important UV-filters were 3BC, IMC and BP3.

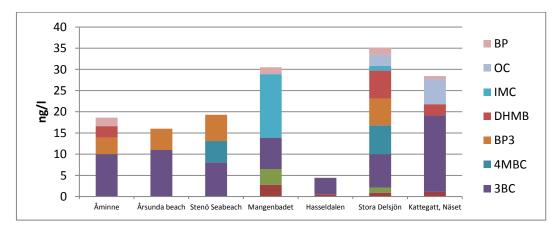


Figure 9 Concentrations of UV-filters in water from bathing sites in the regional sampling program.

Three out of four sediments from the regional program contained UV-filters in low concentrations: OC (21 ng/g dw) and BP (2.3-7.8 ng/g dw), Figure 10.

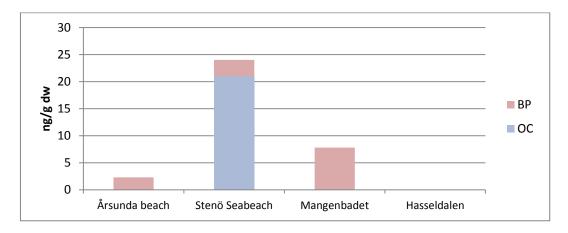


Figure 10 Concentrations of UV-filters in sediment from bathing sites in the regional sampling program.

6.3 WWTPs

Influent

Concentrations in influent waters to WWTPs were not a major priority for this project, but in the regional sample program two influents from medium size WWTPs were included. All UV-filters in the basic group were detected. The summed concentrations were 28 000 and 42 000 ng/l respectively in the two samples. Five substances: OC, 4MBC, HMS, EHS and BP3 together made up 97% and 92% of the summed concentration. The dominating substance in both cases was OC (Figure 11).

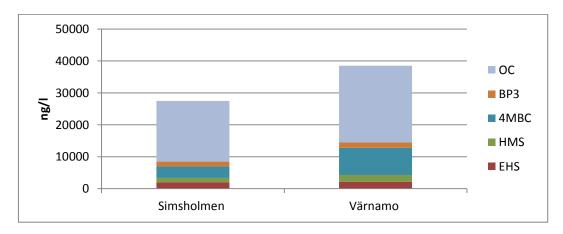


Figure 11 Concentration in WWTP influent waters

The summed concentrations in effluents from the same two WWTPs were 870 and 1 000 ng/l, respectively, which corresponds to summed removal efficiencies of 97 – 98%. For individual substances, calculated removal efficiencies were almost always above 90%.

Effluent

Concentrations of the UV-filters in the basic group in effluent waters from ten WWTPs from both the national and regional programs are shown in Figure 12. The summed concentrations were 220–1800 ng/l. Individual concentrations showed a large variation with a maximum of 560 ng/l. Dominating UV-filters were OC, BP, BP3, DEBENZO, 3BC and OMC.

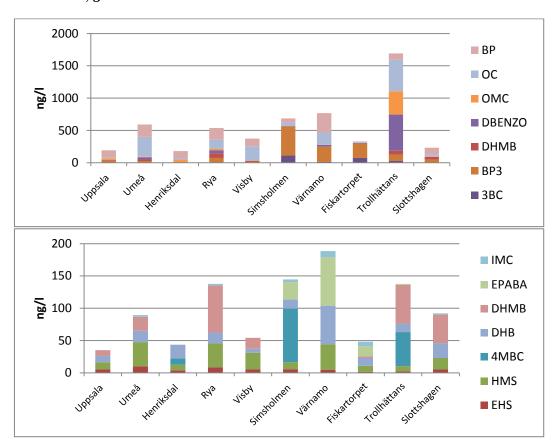


Figure 12 Concentration in WWTP effluent waters for UV-filters in the basic group. Substances with higher concentrations (>100 ng/l in at least one sample) are shown in the upper graph, substances with lower concentrations in the lower graph.

In the previous study (Remberger, 2011) effluents from eight WWTPs was included and summed concentrations ranged 23–1300 ng/l. In Norwegian effluents Tomas (2013) reported higher total concentrations, in the range 300–8900 ng/l; OC, BP3 and OMC dominated

The fragrance DHI was found in all effluents (24-73 ng/l) while PTML was found occasionally (<2-46 ng/l), Figure 13.

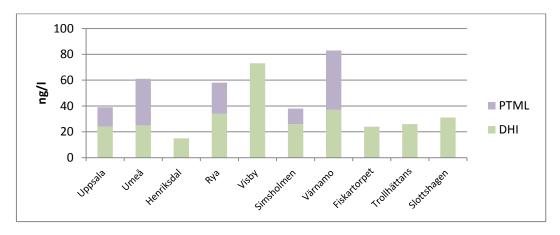


Figure 13 Concentrations of fragrances in WWTP effluents.

Concentrations of substances in the triazine group (group 2) in effluent waters from six WWTPs are shown in Figure 14. Two effluents, Uppsala and Umeå, contained increased concentrations of bemotrizinol and UV360.

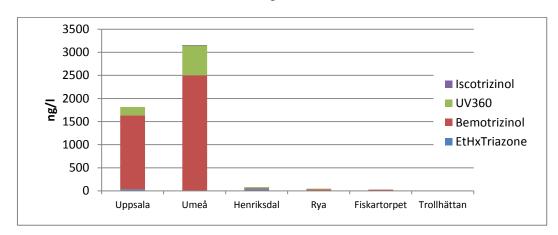


Figure 14 Concentration in WWTP effluent waters for substances in the triazine group.

The benzotriazoles (group 3, textile related UV-filters) were detected in low concentrations compared to the other UV filters and the triazines. UV P was detected in all nine samples (1.9–8.8 ng/l), UV329 was found in five (5–7 ng/l) and UV328 in three samples (0.7–5.6 ng/l). UV327 was not detected.

Sludge

Sludge from eight WWTPs were analysed for the substances in the basic group, Figure 15. Summed concentrations of the UV-filters ranged from 11 000 to 54 000 ng/g dw. All substances with the exception of 3BC, DHMB and IMC were detected in at least one sludge sample.

OC dominated in all samples. This may be explained by the fact that the compound is strongly lipofilic (Table 4) and thus will be concentrated in the sludge phase. The accumulation in sludge may also indicate a high resistance against microbial degradation.

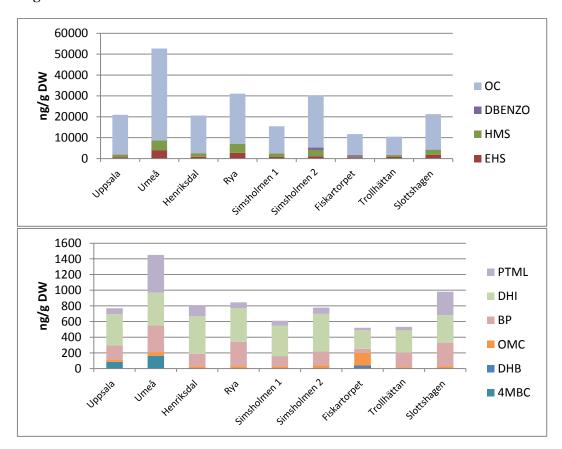


Figure 15 Concentration in sludge for substances in the basic group. Substances with higher concentrations (above), with lower concentrations (below),

One WWTP, Simsholmen, Jönköping, delivered two samples, one from the beginning of July, representing inflow from a cool period, and one from August when the temperature was higher, and presumably, so was the use of sunscreen products. The summed concentration in the sample from August was approximately doubled compared to the one from July, which is in line with that assumption.

The fragrance DHI was detected in the range 240-480 ng/g dw. PTML showed a greater concentration range (27-480 ng/g dw).

Six of the sludges were analysed for the triazine substances. All compounds were found in all sludge samples; UV360 showed the highest concentration in most of the samples (Figure 16).

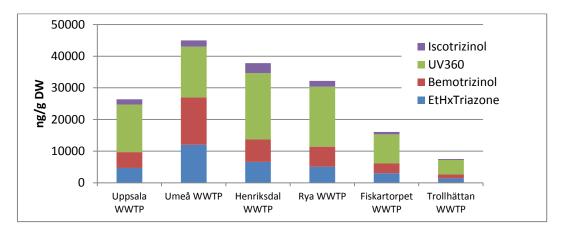


Figure 16 Concentration in sludge for substances in the triazine group.

The sludge samples were also analysed for group 3, the benzotriazoles (Figure 17). UV 328 was the dominating compound. The concentrations in the two samples from Simsholmen were fairly equal, probably reflecting the fact that these compounds are not primarily used in sunscreen products but in textiles.

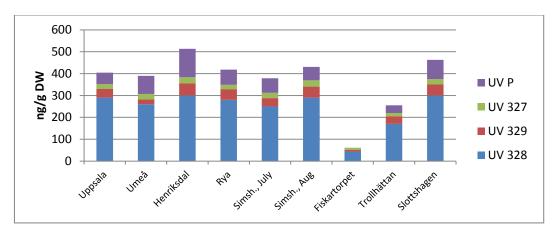


Figure 17 Concentration in sludge for substances in the benzotriazole group.

In Figure 18 the summed concentrations of the three compound groups are shown together. The concentration of triazines were in the same magnitude as the the UV-filters in basic group (mostly OC); the concentration of benzotriazols were much lower.

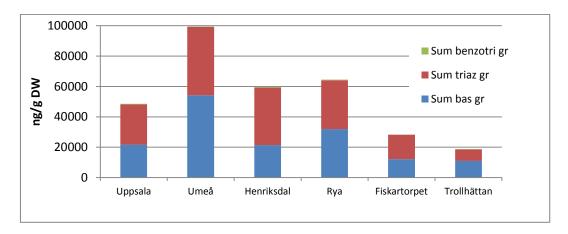


Figure 18 Concentration in sludge. Summed concentrations for the basic group, the triazine group and the benzotriazole group, respectively.

From three of the WWTPs, sludge was sampled and analysed for most of the UV-filters in the basic group also in 2009 (Remberger et al 2011). The concentrations of HMS, OC and EHS were higher in 2014 than in 2009, while the concentrations of DBENZO and 4-MBC were lower (Figure 19).

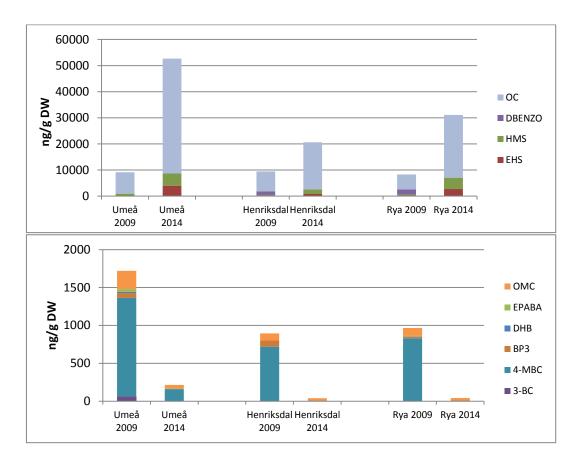


Figure 19 Concentration of UV-filters in the basic group in sludge from three WWTPs 2009 and 2014.

The results are in good agreement with Norwegian data. Thomas (2013) found that OC was the dominating compound in sludge constituting 77–90 % of the total UV-filters, followed by EHS and HMS. Among the benzotriazoles UV 328 was the dominating compound in Sweden but UV329 in Norway. UV360 was not detected in Norwegian sludge.

Receiving water

UV-filters were measured both in effluent from Kungsängsverket WWTP, Uppsala and in the receiving water Fyrisån, see 4. The results are given in Figure 20.

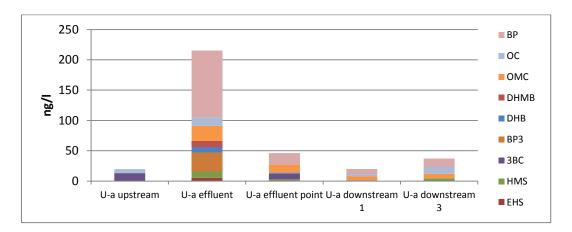


Figure 20 Concentrations in surface water from River Fyrisån upstream the effluent discharge point, in the effluent from Kungsängen WWTP, in surface water close to, and at two distances (150 m, 4.6 km) downstream the discharge point.

3BC, OC and DHB were detected upstream the effluent discharge point, but in low concentrations. The dominating UV-filters BP and OMC (and also DHB) in the effluent were clearly detected in the river water at the discharge point and at the two points downstream, showing a dilution factor of about six at all points. 3BC was not present in the effluent, but detected at the discharge point in about the same concentration as in the upstream water.

Bemotrizinol was detected in the effluent (Figure 14) in high concentrations but did not occur in the receiving water or sediment.

Sediment

Concentrations of UV-filters (and also the fragrance PTML) in sediments from River Fyrisån are shown in Figure 21. The sediment concentration is higher at the site "downstream 1" than close to the effluent discharge point, indicating that the conditions for sedimentation are more favourable at this point.

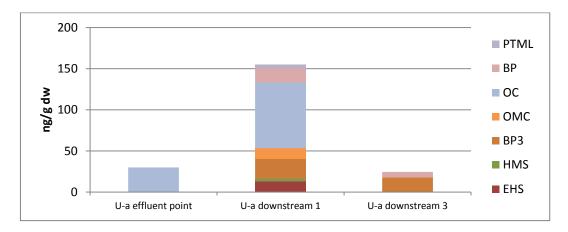


Figure 21 Concentrations in sediment from River Fyrisan close to, and at two distances (150m, 4.6 km) downstream the discharge point.

It can be noticed that the sediments from Lake Rönningesjön, influenced by bathing sites, generally showed higher concentrations of UV-filters than what was found downstream Uppsala WWTP.

Tomas (2014) reported data on eight UV-filters in sediments collected from Oslofjord and Lake Mjøsa in Norway. In sediments from Oslofjord OC (<0.7-82 ng/g dw), UV328 (3-25 ng/g dw) and EHS (8-16 ng/g dw) was detected. In sediments from Lake Mjøsa only EHS was detected (10-20 ng/g dw). UV360, that was measured in significant concentrations (<20-240 ng/g dw) in the present study, was not detected in the Norwegian study.

6.4 Fish

Fish muscle was analysed for the substances in Group 1 and Group 3. None of the substances were detected in the sample from the background lake Tärnan. Fish from sites related to Uppsala WWTP (regional reference lake Valloxen, Fyrisån close to the discharge point and Downstream 3) contained OC (all sites), OMC, HMS and EHS in the concentration range 0.6-7.5 ng/g fw (Figure 22).

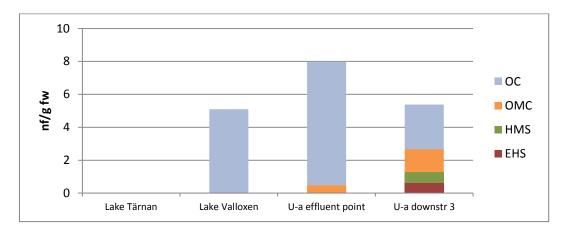


Figure 22 Fish (perch muscle) from Lake Tärnan, Lake Valloxen and two sites in River Fyrisån.

Fish from the regional program contained three UV-filters, 3BC, BP3 and OMC, just above the LOD in the concentration range 0.33-3.6 ng/g fw (Figure 23).

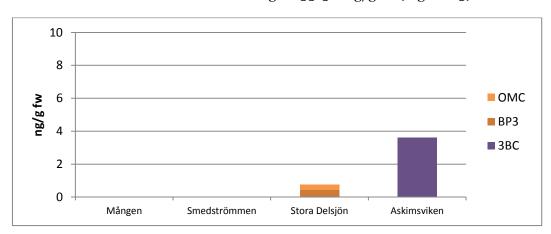


Figure 23 Fish muscle (Askimsviken sea trout, all others perch) from the regional sample program

Remberger (2011) detected two UV-filters (OC, OMC) in fish from the Stockholm area in the same concentration range as in the present study.

6.5 Simplified risk assessment

In a review concerning environmental concentrations and toxicity of personal care products in the aquatic environment (Braush & Rand. 2011) it was pointed out that UV filters are persistent in the environment while being continually replenished through normal usage. The main concerns are their ability to bioaccumulate and their potential for estrogenic activity. It was concluded that shortage of data on environmental concentrations and on toxic effects did not allow for a comprehensive risk profile to be developed.

Table 10 gives the Predicted No Effect Concentrations (PNEC) that are available from the ECHA (European Chemicals Agency) database (ECHA 2015), from a recently published report from the development of the first EU watch list under the Environmental Quality Standards Directive (Carvalho et al , 2015) and from a recent paper by Paredes et al (2015).

Table 10 PNEC values

	PNEC aqua fresh	PNEC aqua marine	PNEC sed fresh	PNEC sed marine	Ref
4-MBC		0.054 ug/l (AF 100)b			Paredes et al 2015
BP3	0.67 ug/l (AF 1000)	0.067 ug/l (AF 10000)	0.066 mg/kg dw	0.0066 mg/kg dw	ECHA 2015
BP3		0.0369 ug/l (AF 100)b			Paredes et al 2015
OMC			0.2 mg/kg (AF 10) a	0.2 mg/kg (AF 10) a	Carvalho et al 2015
OMC		0.488 ug/l (AF 100)c			Paredes et al 2015
OC	0.023 mg/l	0.023 mg/l	4110 mg/kg dw	411 mg/kg dw	ECHA 2015

Endpoints:

Measured environmental concentrations (MEC), median and maximum, for each substance and sample type were calculated. The investigated surface waters were all fresh with the exception of one brackish and one marine water. MEC/PNEC ratios for the fresh surface waters are presented in Table 11. $PNEC_{aqua\ fresh}$ was used when available.

Table 11 MEC/PNEC ratios, fresh surface waters

	MEC _{median} ng/I	MEC _{max} ng/l	PNEC ng/l	MEC _{median} /PNEC	MEC _{max} /PNEC
4MBC	<0.5	14	54 m	< 0.009	0.26
BP3	4.5	19	670	0.007	0.028
OMC	<1	13	488 m	< 0.002	0.03
OC	7.0	100	23000	0.0003	0.004

m following a PNEC value indicates marine environment

MEC/PNEC ratios for the brackish and marine surface waters are presented in Table 12. PNEC_{marine} was used.

Table 12 MEC/PNEC ratios, brackish and marine waters

	MEC _{brackish} ng/I	MEC _{marine} ng/l	PNEC ng/l	MEC _{brackish} /PNEC	MEC _{marine} /PNEC
4MBC	5	<0.5	54 m	0.093	< 0.0093
BP3	6.2	<3	37 m	0.17	<0.081
OMC	<1	<1	488 m	< 0.002	< 0.002
OC	<2	5.9	23000	<0.0001	0.0003

m following a PNEC value indicates marine environment

a Melanoides tuberculata, freshwater snail, 28 d, sediment toxicity

b Isochrysis galbana, microalgae

c Paracentrotus lividus, sea urchin larvae

MEC/PNEC ratios for effluent water after tenfold dilution are presented in Table 13. The receiving waters were all fresh, PNEC_{aqua fresh} were used when available.

Table 13 MEC/PNEC ratios, effluent water after tenfold dilution

	MEC _{median} ng/I	MEC _{max} ng/I	PNEC ng/l	MEC _{median} /PNEC	MEC _{max} /PNEC
4MBC	<1.5	8.3	54 m	< 0.03	0.15
BP3	6.0	45	670	0.009	0.067
OMC	0.82	36	488 m	0.002	0.07
OC	9.4	49	23000	0.0004	0.002

m following a PNEC value indicates marine environment

A MEC/PNEC ratio above 1 indicates a potential environmental risk. The highest ratio for fresh surface water, 0.26, was seen for the maximum concentration measured for 4MBC. The PNEC used is for a marine environment and would probably be higher (the MEC/PNEC ratio lower) for fresh water.

The MEC/PNEC ratios obtained for effluent water after tenfold dilution do not indicate an environmental risk for recieving waters with low salinity. For a marine receiving water MEC_{max}/PNEC for BP3 would have been close to 1 (0.67).

MEC/PNEC_{fresh} ratios for sediments are presented in Table 14.

Table 14 MEC/PNEC ratios, sediment

	MEC _{median} ng/g dw	MEC _{max} ng/g dw	PNEC ng/g dw	MEC _{median} /PNEC	MEC _{max} /PNEC
BP3	15	62	66	0.22	0.94
OMC	<7	35	200	< 0.04	0.18
OC	47	320	4110000	0.00001	0.0008

For BP3 the ratio is close to 1 for the highest concentration measured, which was in a sediment from the bathing site in Rönningesjön sampled in August.

It was recently pointed out (Downs, 2015) that the local use of BP3 in sunscreen lotions poses a hazard to coral reef conservation and threatens the resiliency of coral reefs to climate change.

For the rest of the investigated substances no PNEC values were available. ECHA has however identified UV-328 as a SVHC (Substance of Very High Concern) due to its PBT/vPvB properties (PBT: persistent, bio-accumulative and toxic; vPvB: very persistent and very bio-accumulative). The motivation is available in a SVHC Support document (ECHA 2014a) but the report states that "no data relevant for assessing the T-criterion can be reported".

UV-320 has also been identified as a SVHC due to its PBT/vPvB properties (ECHA 2014b). Also for this substance it is stated that "no data relevant for assessing the T-criterion can be reported". UV-328 and UV-320 were in December 2014 included on the Candidate List for eventual inclusion in Annex XIV to REACH (ECHA 2014c). UV-

320 was not included in this screening but the structure of UV-320 is derived from the structure of UV-327 by replacing the chlorine atom by a hydrogen. For UV-327 no decision on SVHC status has been made but a SVHC draft support document is available (ECHA 2013). This document states about toxicity: "The available studies show that UV-327 is not acutely toxic for aquatic organisms. There is no information on the long-term toxicity of UV-327. Based on the currently available data it was concluded that UV-327 does not fulfil the T-criterion."

7 Conclusions

Bathing sites

- UV-filters are transported from the bathing sites to more unaffected parts in a lake.
- There is a diurnal turnover of the UV filters in the water column as a result of mixing and/or degradation (photo- and microbial degradation).
- The UV filter concentrations are reduced in water after the bathing season but low amounts could be detected in spring before the next bathing season.
- The levels in the sediments indicate a possible accumulation of UV-filters. This is especially clear for UV360.
- Sediment may be a more reliable matrix than water for environmental assessments of UV-filters. Water samples give only a snapshot of the situation.
- The highest levels of the UV-filter BP3 measured in sediments indicates a possible environmental risk.

Waste water treatment plants and receiving waters

- In WWTPs about 97-98 % of the UV-filters are removed from the waste water.
- The concentration in sludge of OC, HMS and EHS has increased between 2009 and 2015.
- Two our knowledge ethylhexyltriazone, bemotrizinol, UV360 and iscotrizinol (the compounds in the option 2 group) have not been measured before in the Swedish environment and appears to be important new contaminants at bathing sites and in WWTPs.
- The UV-filters mainly used in textile applications (option 1 group) were frequently detected in sludge and effluent water but more sparingly in surface water and sediment.

There is no obvious correlation between use in Sweden according to SPIN and the detected concentrations in the environment and effluents. For example, OC is the dominant UV filter in surface water, sediments and effluents but a minor component in SPIN. But, as already pointed out (Chapter 2), UV-filters contained in finished cosmetic products are not included in SPIN. On the other hand, BP is used in great amounts (SPIN-data) but is detected at low concentrations in environmental samples as well as in WWTP effluents. The explanation may be that OC and BP have different stability to photochemical and/or microbial degradation. The high concentration of OC in solid samples (sediment and sludge) can probably be attributed to the high K_{OW} (Table 4).

BMDM (avobenzone) is a good UV-filter in cosmetic products (broad spectral range; effective UV-scavenger) but is susceptible to UV-degradation⁵. This fact may explanation why BMDM was detected in only one sample in this investigation. This is also valid for previous investigations: BMDM was rarely detected, and then in low concentrations, even in UV-contaminated environmental samples from bathing sites (Poiger et al., 2004; Giokas et al., 2004; Rodil et al., 2008; Remberger et al. 2011). Rodil et al. (2009), however, measured a quite high concentration (2431 ng/l) in lake water collected from a bathing site a hot summer day.

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⁵ http://www.smartskincare.com

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Appendix 1 Sample list

ID	Nat./County	Municipallity	Site	Matrix	Sampling date	RT90 X	RT90 Y
3379	National	Vallentuna	Tärnan	Surface water	2014-09-10	6608704	164490
3380	National	Vallentuna	Tärnan	Sediment	2014-09-10	6608704	164490
3734	National	Vallentuna	Tärnan	Fish muscle, perch	2014-09-10	6608704	164490
3363	National	Uppsala	Uppsala WWTP	Effluent	2014-09-04	6637475	1603969
3364	National	Uppsala	Uppsala WWTP	Sludge	2014-09-03	6637475	1603969
3330	National	Uppsala	U-a upstream	Surface water	2014-08-28	6638717	1602928
3331	National	Uppsala	U-a effluent point	Surface water	2014-08-28	6637288	1603795
3332	National	Uppsala	U-a downstream 1 (150 m)	Surface water	2014-08-28	6637143	1603849
3334	National	Uppsala	U-a downstream 3 (4.6 km)	Surface water	2014-08-28	6634020	1604660
3349	National	Uppsala	U-a effluent point	Sediment	2014-08-29	6637288	1603795
3350	National	Uppsala	U-a downstream 1 (150 m)	Sediment	2014-08-29	6637143	1603849
3351	National	Uppsala	U-a downstream 3 (4.6 km)	Sediment	2014-08-29	6634020	1604660
3720	National	Uppsala	U-a Valloxen, reg backgr	Fish muscle, perch	2014-08-29	6624246	1612705
3721	National	Uppsala	U-a effluent point	Fish muscle, perch	2014-08-29	6637288	1603795
3722	National	Uppsala	U-a downstream 3 (4.6 km)	Fish muscle, perch	2014-08-29	6634020	1604660
3328	National	Umeå	Umeå WWTP	Effluent	2014-08-20		
3329	National	Umeå	Umeå WWTP	Sludge	2014-08-20		
3369	National	Stockholm	Henriksdal WWTP	Effluent	2014-09-03		
3372	National	Stockholm	Henriksdal WWTP	Sludge	2014-09-04		
3212	National	Göteborg	Rya WWTP	Effluent	2014-07-30		
3213	National	Göteborg	Rya WWTP	Sludge	2014-07-30		
3150	National	Täby	Rönningesjön, jetty May	Surface water	2014-05-23		
3151	National	Täby	Rönningesjön S5 May	Surface water	2014-05-23		
3242	National	Täby	Rönningesjön, jetty, morning	Surface water	2014-08-08	6596355	1630476
3249	National	Täby	Rönningesjön, noon, jetty	Surface water	2014-08-08	6596355	1630476
3241	National	Täby	Rönningesjön, jetty, evening	Surface water	2014-08-07	6596355	1630476
3243	National	Täby	Rönningesjön August S1	Surface water	2014-08-08	6596313	1630428

ID	Nat./County	Municipallity	Site	Matrix	Sampling date	RT90 X	RT90 Y
3244	National	Täby	Rönningesjön August S2	Surface water	2014-08-08	6596250	1630359
3245	National	Täby	Rönningesjön August S3	Surface water	2014-08-08	6595948	1630288
3246	National	Täby	Rönningesjön August S4	Surface water	2014-08-08	6595767	1630343
3247	National	Täby	Rönningesjön August S5	Surface water	2014-08-08	6595586	1630391
3248	National	Täby	Rönningesjön August S6	Surface water	2014-08-08	6596453	1630320
3464	National	Täby	Rönningesjön October S1	Surface water	2014-10-01	6596279	1630409
3467	National	Täby	Rönningesjön October S5	Sediment	2014-10-01	6595586	1630391
3152	National	Täby	Rönningesjön May S2	Sediment	2014-05-27	6596307	1630430
3250	National	Täby	Rönningesjön August S1	Sediment	2014-08-08	6596313	1630428
3251	National	Täby	Rönningesjön August S2	Sediment	2014-08-08	6596250	1630359
3252	National	Täby	Rönningesjön August S3	Sediment	2014-08-08	6595948	1630288
3253	National	Täby	Rönningesjön August S4	Sediment	2014-08-08	6595767	1630343
3254	National	Täby	Rönningesjön August S5	Sediment	2014-08-08	6595586	1630391
3465	National	Täby	Rönningesjön October S2	Sediment	2014-10-01	6596279	1630409
3346	Gotland	Gotland	Visby WWTP	Effluent	2014-07-22		
3347	Gotland	Gotland	Åminne	Surface water	2014-07-22	6391405	1676247
3306	Gävleborg	Sandviken	Årsunda beach Storsjön	Surface water	2014-08-15	6712248	1550825
3307	Gävleborg	Sandviken	Årsunda beach Storsjön	Sediment	2014-08-15	6712248	1550825
3304	Gävleborg	Söderhamn	Stenö Seabeach Söderhamnsfjärden	Surface water	2014-08-15	6793115	1574527
3305	Gävleborg	Söderhamn	Stenö Seabeach Söderhamnsfjärden	Sediment	2014-08-15	6793115	1574527
3308	Jönköping	Jönköping	Simsholmen WWTP	Influent	2014-07-10		
3309	Jönköping	Jönköping	Simsholmen WWTP	Effluent	2014-07-10		
3310, 3311	Jönköping	Jönköping	Simsholmen WWTP	Sludge	2014-07-09 and 2014-08-19		
3276	Jönköping	Värnamo	Värnamo WWTP	Influent	2014-08-12		
3277	Jönköping	Värnamo	Värnamo WWTP	Effluent	2014-08-12		
3255	Värmland	Karlstad	Mången (Mangenbadet)	Surface water	2014-08-11	6612371	1391184
3296	Värmland	Karlstad	Mången (Mangenbadet)	Fish muscle, perch	2014-08-11	6612653	1390590
3256	Värmland	Grums	Långsjön (Hasseldalen)	Surface water	2014-08-11	6591605	1345308
3224	Värmland	Grums	Långsjön (Hasseldalen)	Sediment	2014-07-31	6591657	1345271
3223	Värmland	Karlstad	Mången (Mangenbadet)	Sediment	2014-07-31	6612425	1391189

ID	Nat./County	Municipallity	Site	Matrix	Sampling date	RT90 X	RT90 Y
3198	Värmland	Kristinehamn	Fiskartorpet WWTP	Effluent	2014-07-22		
3199	Värmland	Kristinehamn	Fiskartorpet WWTP	Sludge	2014-07-22		
3492	V Götaland	Trollhättan	Trollhättan WWTP	Effluent	2014-10-14		
3493	V Götaland	Trollhättan	Trollhättan WWTP	Sludge	2014-10-14		
3681	V Götaland	Trollhättan	Göta älv, Smedströmmen, Stallbackabron	Fish muscle, perch	2014-09-27	6470175	1294349
3494	V Götaland	Göteborg	Stora Delsjön	Surface water	2014-09-01	6402258	1275231
3682	V Götaland	Göteborg	Stora Delsjön	Fish muscle, perch	2014-09-26	6402233	1275283
3495	V Götaland	Göteborg	Kattegatt, Näset beach	Surface water	2014-09-04	6394511	1266587
3683	V Götaland	Göteborg	Kattegatt, Saltsjönäs, Askimsviken	Fish muscle, Sea trout	2014-09-29	6394618	1267049
3299	Östergötland	Norrköping	Slottshagen WWTP	Effluent	2014-08-13		
3300	Östergötland	Norrköping	Slottshagen WWTP	Sludge	2014-08-18		

Appendix 2 Results

Results for substances in Group 1, Basic group.

Matrix	ID	Site	Unit	BMDM	EHS	HMS	звс	4MBC	BP3	DHB	DHMB	EPABA	DBENZO	IMC	ОМС	ос	ВР	DHI	PTML	Triklosan
Surface w	3379	Tärnan	ng/l	<1	0.6	2.2	5.3	13	<3	<0.5	<1	0.86	<3	1.2	<1	4.3	1.8	160	<1.6	<0.6
Surface w	3330	U-a upsteam	ng/l	<1	<0.5	<2	13	<0.5	<3	1	<1	<0.5	<2	1.5	<1	6	<0.6	21	<1.6	1.0
Surface w	3331	U-a effluent point	ng/l	<1	<0.5	3	9.1	<0.5	<3	2	<1	<0.5	<2	<0.5	13	<2	19	19	<1.6	2.3
Surface w	3332	U-a downstream 1	ng/l	<1	<0.5	<2	<0.5	<0.5	<3	1	<1	0.55	<2	<0.5	6.9	3.6	8.4	14	<1.6	1.4
Surface w	3334	U-a downstream 3	ng/l	<1	<0.5	4.7	<0.5	<0.5	<3	<0.5	<1	<0.5	3.1	<0.5	7.4	11	14	26	<1.6	<0.6
Surface w	3150	Rönningesjön, jetty, spring	ng/l	<1	2.4	<3	28	9.0	<5	<0.5	<2	<1	<2	<0.6	<2	<2	3	340	<3	<1
Surface w	3151	Rönningesjön S5, spring	ng/l	<1	<1	<3	<0.5	13	<5	1	<2	1.2	<2	<0.6	<2	<2	<1	310	<3	1.4
Surface w	3242	Rönningesjön, jetty, morning	ng/l	<1	2.6	<3	13	<1	9.4	<0.5	<2	1.5	13	1.1	<2	11	6.0	220	<3	<1
Surface w	3249	Rönningesjön, jetty, noon	ng/l	<1	2.3	22	10	<0.5	19	<0.5	2.4	2.0	19	<0.3	<1	100	4.3	220	<1.6	<0.6
Surface w	3241	Rönningesjön, jetty, evening	ng/l	<1	1.5	5.9	17	<1	11	<0.5	3.7	1.4	<1	<0.6	<2	8	3.4	300	<3	<1
Surface w	3243	Rönningesjön S1	ng/l	<1	<1	5.9	21	<1	10	<0.5	<2	2.2	14	<0.6	<2	27	1.4	250	<3	<1
Surface w	3244	Rönningesjön S2	ng/l	<1	1.6	5.7	24	6.7	8.6	<0.5	2.1	<1	4	2.0	<2	41	4.1	290	<3	<1
Surface w	3245	Rönningesjön S3	ng/l	<1	<1	8.4	<1	<1	12	1	4.4	<1	3	<0.6	3	66	<1	270	22	<1
Surface w	3246	Rönningesjön S4	ng/l	<1	<1	5.8	78	<1	10	0	2.8	<1	<2	<0.6	<2	45	<1	280	<3	<1
Surface w	3247	Rönningesjön S5	ng/l	<1	4.0	18	4.4	14	12	<0.5	<2	2.3	<2	<0.6	<3	96	3.4	310	<3	<1
Surface w	3248	Rönningesjön S6	ng/l	<1	2.0	3.6	<0.5	<1	10	<0.5	<2	<1	<2	<0.6	<4	12	4.8	240	<3	<1
Surface w	3464	Rönningesjön S1, autumn	ng/l	<1	2.6	1.6	<0.5	<0.5	<3	<0.5	24	<0.5	<2	<0.5	<1	16	0.8	250	<1.6	<0.6
Surface w	3347	Åminne	ng/l	<1	<0.5	<2	10	<0.5	3.9	<0.5	2.7	<0.5	<2	<0.5	<1	<2	2.0	89	<1.6	<0.6
Surface w	3306	Årsunda beach Storsjön	ng/l	<1	<0.5	<2	11	<0.5	5.0	<0.5	<1	<0.5	<2	<0.5	<1	<2	<0.6	100	<1.6	<0.6
Surface w	3304	Stenö Seabeach Söderhamnsfjärden	ng/l	<1	<0.5	<2	8.1	5	6.2	<0.5	<1	<0.5	<2	<0.5	<1	<2	<0.6	63	<1.6	<0.6
Surface w	3255	Mången (Mangenbadet)	ng/l	<1	2.8	3.7	7.4	<0.5	<3	<0.5	<1	<0.5	<2	15	<1	<2	1.6	44	<1.6	<0.6
Surface w	3256	Långsjön (Hasseldalen)	ng/l	<1	0.5	<2	3.9	<0.5	<3	<0.5	<1	<0.5	<2	<0.5	<1	<2	<0.6	40	<1.6	<0.6
Surface w	3494	Stora Delsjön	ng/l	<1	0.9	1.2	7.9	7	6.3	<0.5	6.6	<0.5	<2	1.1	<1	2.6	1.7	44	18	<0.6
Surface w	3495	Kattegatt, Näsets beach	ng/l	<1	1.1	<1.5	18	<0.5	<3	<0.5	2.7	<0.5	<2	<0.5	<1	5.9	0.7	40	<1.6	<0.6

Matrix	ID	Site	Unit	BMDM	EHS	HMS	звс	4MBC	BP3	DHB	DHMB	EPABA	DBENZO	IMC	омс	ос	ВР	DHI	PTML	Triklosan
Effluent	3363	Uppsala	ng/l	<3	5.3	11	<1	<15	31	10	9.1	<1	4	<1	25	14	110	24	15	10
Effluent	3328	Umeå	ng/l	<3	10	37	<1	<15	27	18	22	<1	32	2.3	9.1	310	190	25	36	18
Effluent	3369	Henriksdal	ng/l	<3	3.9	8.6	<1	10	10	21	<2	<1	<3	<1	30	19	120	15	<1.6	21
Effluent	3212	Rya	ng/l	<3	8.4	37	<1	<15	73	17	73	<1	47	2.2	24	140	180	34	24	17
Effluent	3346	Visby	ng/l	<3	5.1	26	<1	<15	11	6	17	<1	<2	<1	5.5	220	120	73	<1.6	6.3
Influent	3308	Simsholmen	ng/l	<6	2000	1400	27	3600	1500	230	6.2	6.1	110	11	<2	19000	290	190	15	99
Effluent	3309	Simsholmen	ng/l	<3	6	11	110	83	450	14	<2	27	6	4.2	5	47	66	26	12	1.3
Influent	3276	Värnamo	ng/l	290	2100	2200	45	8500	1700	550	39	26	380	<0.3	540	24000	870	410	43	830
Effluent	3277	Värnamo	ng/l	<3	4.9	39	12	<15	240	60	<2	75	21	9.6	4.4	190	300	37	46	53
Effluent	3198	Fiskartorpet	ng/l	<1	1.4	10	74	<15	220	12	2.4	16	9	6.8	3.0	11	8.8	24	<3	14
Effluent	3492	Trollhättans	ng/l	<3	2.3	7.9	26	53	100	13	60	1.4	560	<1	360	490	95	26	<1.6	<1
Effluent	3299	Slottshagen	ng/l	<3	5.0	18	<1	<15	46	23	44	<1	<3	2.0	7.2	34	100	31	<1.6	23
Sediment	3380	Tärnan	ng/g dw	<4	<7	<2	<2	<2	<13	<3	<6	<1	<3	<0.2	<7	<6	5.9	170	<3	<2
Sediment	3349	U-a effluent point	ng/g dw	<4	<7	<2	<2	<2	<13	<3	<6	<1	<3	<0.2	<7	30	<2	25	<3	<2
Sediment	3350	U-a downstream 1	ng/g dw	<4	13	3.3	<2	<2	24	<3	<6	<1	<3	<0.2	13	80	17	190	4.7	10
Sediment	3351	U-a downstream 3	ng/g dw	<4	<7	<2	<2	<2	18	<3	<6	<1	<3	<0.2	<7	<6	6.6	58	<3	<2
Sediment	3152	Rönningesjön May S2	ng/g dw	<4	25	6	<2	<2	45	<3	<6	1.2	36	<0.2	30	120	22	320	3.7	<2
Sediment	3250	Rönningesjön August S1	ng/g dw	<4	44	15	<2	<2	62	<3	<6	<1	57	<0.2	35	320	35	420	7.6	<2
Sediment	3251	Rönningesjön August S2	ng/g dw	<4	23	8.0	<2	<2	28	<3	<6	<1	31	<0.2	15	170	20	340	4.8	<2
Sediment	3252	Rönningesjön August S3	ng/g dw	<4	23	5.8	<2	<2	27	<3	<6	<1	<3	<0.2	13	51	23	410	5.9	<2
Sediment	3253	Rönningesjön August S4	ng/g dw	<4	40	12	<2	<2	48	<3	<6	1.2	<3	<0.2	21	120	32	390	20	<2
Sediment	3254	Rönningesjön August S5	ng/g dw	<4	10	<2	<2	<2	19	<3	<6	<1	<3	<0.2	<7	42	10	220	<3	<2
Sediment	3465	Rönningesjön October S2	ng/g dw	<4	<7	<2	<2	<2	10	<3	<6	<1	6.4	<0.2	<7	160	6.8	790	<3	<2
Sediment	3467	Rönningesjön October S5	ng/g dw	<4	<7	<2	<2	<2	<13	<3	<6	<1	<3	<0.2	<7	110	6.9	330	<3	<2
Sediment	3307	Årsunda beach Storsjön	ng/g dw	<4	<7	<2	<2	<2	<13	<3	<6	<1	<3	<0.2	<7	<6	2.3	18	15	<2
Sediment	3305	Stenö beach Söderhamnsfjärden	ng/g dw	<4	<7	<2	<2	<2	<13	<3	<6	<1	<3	<0.2	<7	21	3.0	57	<3	<2
Sediment	3224	Långsjön (Hasseldalen)	ng/g dw	<4	<7	<2	<2	<2	<13	<3	<6	<1	<3	<0.2	<7	<6	<2	10	3.8	<2
Sediment	3223	Mången (Mangenbadet)	ng/g dw	<4	<7	<2	<2	<2	<13	<3	<6	<1	<3	<0.2	<7	<6	7.8	11	<3	<2

Matrix	ID	Site	Unit	BMDM	EHS	нмѕ	звс	4MBC	BP3	DHB	DHMB	EPABA	DBENZO	IMC	омс	ос	ВР	DHI	PTML	Triklosan
Sludge	3364	Uppsala	ng/g dw	<5	660	1200	<30	86	<10	<5	<1	<3	140	<1	27	19000	180	400	76	420
Sludge	3329	Umeå	ng/g dw	<5	4000	4700	<30	160	<10	<5	<1	6.4	<20	<1	49	44000	340	420	480	370
Sludge	3372	Henriksdal	ng/g dw	<5	800	1800	<30	<4	12	<5	<1	<3	<20	<1	28	18000	160	480	140	720
Sludge	3213	Rya	ng/g dw	<5	2700	4400	<30	<4	11	<5	<1	<3	<20	<1	31	24000	310	430	74	520
Sludge	3310	Simsholmen	ng/g dw	<5	780	1700	<30	<4	<10	<5	<1	4.7	<20	<1	28	13000	130	390	62	300
Sludge	3311	Simsholmen	ng/g dw	<5	1100	3000	<30	<4	<10	<5	<1	<3	1200	<1	38	25000	180	480	80	310
Sludge	3199	Fiskartorpet	ng/g dw	<5	590	420	<30	<4	<10	38	<1	6.4	650	<1	160	10000	54	240	29	120
Sludge	3493	Trollhättan	ng/g dw	<5	720	930	<30	<4	<10	<5	<1	<3	47	<1	18	8800	190	280	44	200
Sludge	3300	Slottshagen	ng/g dw	<5	1700	2400	<30	<4	<10	<5	<1	36	190	<1	31	17000	300	350	300	730
Fish muscle	3734	Tärnan	ng/g fw	<1	<0.5	<0.3	<3	<0.1	<0.4	<2	<2	<1	<1	<0.1	<0.3	<2	<4	<0.5	<1	<0.3
Fish muscle	3720	U-a Valloxen, reg backgr	ng/g fw	<1	<0.5	<0.3	<3	<0.1	<0.4	<2	<2	<1	<1	<0.1	<0.3	5.1	<4	<0.5	<1	<0.3
Fish muscle	3721	U-a effluent point	ng/g fw	<1	<0.5	<0.3	<3	<0.1	<0.4	<2	<2	<1	<1	<0.1	0.49	7.5	<4	0.6	<1	<0.3
Fish muscle	3722	U-a downstream 3 (4.6 km)	ng/g fw	<1	0.63	0.65	<3	<0.1	<0.4	<2	<2	<1	<1	<0.1	1.4	2.7	<4	1.2	<1	<0.3
Fish muscle	3296	Mången (Mangenbadet)	ng/g fw	<1	<0.5	<0.3	<3	<0.1	<0.4	<2	<2	<1	<1	<0.1	<0.3	<2	<4	1.4	<1	<0.3
Fish muscle	3681	Göta älv, Smedströmmen, Stallbackabron	ng/g fw	<1	<0.5	<0.3	<3	<0.1	<0.4	<2	<2	<1	<1	<0.1	<0.3	<2	<4	<0.5	<1	<0.3
Fish muscle	3682	Stora Delsjön	ng/g fw	<1	<0.5	<0.3	<3	<0.1	0.44	<2	<2	<1	<1	<0.1	0.33	<2	<4	<0.5	3.8	<0.3
Fish muscle	3683	Kattegatt, Saltsjönäs, Askimsviken	ng/g fw	<1	<0.5	<0.3	3.6	<0.1	<0.4	<2	<2	<1	<1	<0.1	<0.3	<2	<4	<0.5	<1	<0.3

Results for substances in Group 2 (option 2) and Group 3 (option 1).

(Results were not commissioned for all samples, indicated by grey cells).

Matrix	ID	Site	Unit	UV 328	UV 329	UV 327	UV P	Ethylhexyl- triazone	Bemo- trizinol	UV360	Iscotrizinol
Surface w	3379	Tärnan	ng/l	<0.5	<1	<3	<0.5	<12	<2	<4	<1
Surface w	3330	U-a upsteam	ng/l	<0.5	2.1	<3	<0.5	<12	3.5	5.6	<1
Surface w	3331	U-a effluent point	ng/l	<0.5	1.5	<3	<0.5	<12	3.1	<4	<1
Surface w	3332	U-a downstream 1	ng/l	<0.5	1.5	<3	<0.5	<12	3.3	<4	<1
Surface w	3334	U-a downstream 3	ng/l	<0.5	3.5	<3	<0.5	<12	<2	<4	<1
Surface w	3150	Rönningesjön, jetty, spring	ng/l	<1	<1	<3	<1	<12	<2	<4	<1
Surface w	3151	Rönningesjön S5, spring	ng/l	<1	<1	<3	<1	<12	2.0	5.0	<1
Surface w	3242	Rönningesjön, jetty, morning	ng/l	<1	2.8	<3	<1	<12	<2	<4	1.2
Surface w	3249	Rönningesjön, jetty, noon	ng/l	<0.5	<1	<3	<0.5	23	7.5	8.0	6.8
Surface w	3241	Rönningesjön, jetty, evening	ng/l	<1	<1	<3	<1	<12	<2	5.6	<1
Surface w	3243	Rönningesjön S1	ng/l	<1	<1	<3	<1	<12	<2	<4	<1
Surface w	3244	Rönningesjön S2	ng/l	<1	2.7	<3	<1	<12	4.0	<4	0.74
Surface w	3245	Rönningesjön S3	ng/l	<1	<1	<3	<1	<12	<2	<4	0.70
Surface w	3246	Rönningesjön S4	ng/l	<1	4.9	<3	<1	<12	<2	<4	<1
Surface w	3247	Rönningesjön S5	ng/l	<1	<1	<3	<1	<12	<2	<4	1.4
Surface w	3248	Rönningesjön S6	ng/l	<1	<1	<3	<1	<12	2.4	<4	1.5
Surface w	3464	Rönningesjön S1, autumn	ng/l	<0.5	<1	<3	<0.5	<12	<2	<4	<1
Surface w	3347	Åminne	ng/l								
Surface w	3306	Årsunda beach Storsjön	ng/l	<0.5	<1	<3	<0.5	<12	<2	<4	<1
Surface w	3304	Stenö Seabeach Söderhamnsfjärden	ng/l	<0.5	<1	<3	<0.5	12	<2	<4	<1
Surface w	3255	Mången (Mangenbadet)	ng/l	0.8	2.1	<3	<0.5				
Surface w	3256	Långsjön (Hasseldalen)	ng/l	<0.5	<1	<3	<0.5				
Surface w	3494	Stora Delsjön	ng/l					19	6.4	<4	0.82

Matrix	ID	Site	Unit	UV 328	UV 329	UV 327	UV P	Ethylhexyl- triazone	Bemo- trizinol	UV360	Iscotrizinol
Surface w	3495	Kattegatt, Näsets beach	ng/l					14	16	18	1.6
Effluent	3363	Uppsala	ng/l	<1	6.7	<5	3.4	35	1600	180	<2
Effluent	3328	Umeå	ng/l	<1	7.0	<5	8.0	<30	2500	650	10
Effluent	3369	Henriksdal	ng/l	0.7	<2	<5	1.9	39	18	27	<2
Effluent	3212	Rya	ng/l	<1	<2	<5	7.3	<30	33	14	6
Effluent	3346	Visby	ng/l								
Influent	3308	Simsholmen	ng/l	48	6	<5	26				
Effluent	3309	Simsholmen	ng/l	<1	<2	<5	8.6				
Influent	3276	Värnamo	ng/l	95	17	<5	70				
Effluent	3277	Värnamo	ng/l	5.6	<2	<5	8.8				
Effluent	3198	Fiskartorpet	ng/l	<1	<2	<5	2.6	<30	26.0	<9	<2
Effluent	3492	Trollhättans	ng/l	4.7	5	<5	5.5	<30	<6	<9	<2
Effluent	3299	Slottshagen	ng/l	1.2	6	<5	5.1				
Sediment	3380	Tärnan	ng/g dw	<6	<3	<5	<4	<70	<40	<20	<1
Sediment	3349	U-a effluent point	ng/g dw	<6	<3	<5	<4	<70	<40	<20	<1
Sediment	3350	U-a downstream 1	ng/g dw	10	5.4	<5	6.3	<70	<40	<20	<1
Sediment	3351	U-a downstream 3	ng/g dw	<6	<3	<5	<4	<70	<40	<20	6.5
Sediment	3152	Rönningesjön S2	ng/g dw	<6	<3	<5	4.5	<70	<40	29	3.0
Sediment	3250	Rönningesjön S1	ng/g dw	<6	<3	<5	6.4	73.0	<40	170	18
Sediment	3251	Rönningesjön S2	ng/g dw	<6	<3	<5	4.1	<70	<40	160	8.5
Sediment	3252	Rönningesjön S3	ng/g dw	<6	<3	<5	<4	<70	<40	92	3.9
Sediment	3253	Rönningesjön S4	ng/g dw	7.1	<3	<5	5.2	<70	<40	100	5.8
Sediment	3254	Rönningesjön S5	ng/g dw	<6	<3	<5	<4	<70	<40	110	2.6
Sediment	3465	Rönningesjön autumn S1	ng/g dw	<6	<3	<5	<4	<70	<40	220	5.8
Sediment	3467	Rönningesjön autumn S5	ng/g dw	<6	<3	<5	<4	<70	<40	240	8.1
Sediment	3307	Årsunda beach Storsjön	ng/g dw	<6	<3	<5	<4	<70	<40	<20	<1
Sediment	3305	Stenö beach Söderhamnsfjärden	ng/g dw	<6	<3	<5	<4	<70	<40	<20	1.4

Matrix	ID	Site	Unit	UV 328	UV 329	UV 327	UV P	Ethylhexyl- triazone	Bemo- trizinol	UV360	Iscotrizinol
Sediment	3224	Långsjön (Hasseldalen)	ng/g dw	<6	<3	<5	<4				
Sediment	3223	Mången (Mangenbadet)	ng/g dw	<6	<3	<5	<4				
Sludge	3364	Uppsala	ng/g dw	290	40	22	52	4700	5000	15000	1700
Sludge	3329	Umeå	ng/g dw	260	22	25	83	12000	15000	16000	2000
Sludge	3372	Henriksdal	ng/g dw	300	56	27	130	6600	7100	21000	3100
Sludge	3213	Rya	ng/g dw	280	49	20	69	5100	6300	19000	1800
Sludge	3310	Simsholmen	ng/g dw	250	38	24	67				
Sludge	3311	Simsholmen	ng/g dw	290	51	28	62				
Sludge	3199	Fiskartorpet	ng/g dw	44	7.2	10	<10	3000	3100	9200	750
Sludge	3493	Trollhättan	ng/g dw	170	35	15	35	1400	1200	4600	300
Sludge	3300	Slottshagen	ng/g dw	300	50	25	88				
Fish muscle	3734	Tärnan	ng/g fw	<0.3	<0.3	<0.3	<0.2				
Fish muscle	3720	U-a Valloxen, reg backgr	ng/g fw	<0.3	<0.3	<0.3	<0.2				
Fish muscle	3721	U-a effluent point	ng/g fw	<0.3	<0.3	<0.3	<0.2				
Fish muscle	3722	U-a downstream 3 (4.6 km)	ng/g fw	<0.3	<0.3	<0.3	<0.2				
Fish muscle	3296	Mången (Mangenbadet)	ng/g fw	<0.3	<0.3	<0.3	<0.2				
Fish muscle	3681	Göta älv, Smedströmmen nedan Stallbackabron	ng/g fw	<0.3	<0.3	<0.3	<0.2				
Fish muscle	3682	Stora Delsjön	ng/g fw	<0.3	<0.3	<0.3	<0.2				
Fish muscle	3683	Kattegatt, Saltsjönäs, Askimsviken	ng/g fw	<0.3	<0.3	<0.3	<0.2	_			

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