REPORT

Results from the Swedish National Screening Programme 2010

Subreport 2. Fluorescent Whitening Agents

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Title and subtitle of the report

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The concentration range of summed FWAs was 20 - 24 µg/l in three WWTP influents, 0.43 - 5.1 µg/l in nine WWTP effluents and 56 000 – 160 000 ng/g DW in nine WWTP sludges. DAS2, FB28 and, in some effluents, DAS1 was the dominating individual cmopounds. DAS2 is associated to paper applications rather than textile/detergent applications.

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Highest concentration in sediment, 7 200 ng/g DW, dominated by DAS1, was found near the discharge point of a WWTP. Further away, 1 km and 20 km from the effluent point, in the main direction of flow, concentrations were considerably lower, 69 and 150 ng/g DW. Sediments from Lake Mälaren showed summed concentrations of 92 – 900 ng/g DW. Relevant PNECs for evaluation of risks for sediment dwelling organisms were not available.

Analysis of FWAs extracted from toilet paper manufactured from recycled fibres (two different brands) showed that this may be a major source for FWAs in WWTP influents. The contribution from toilet paper made from virgin fibres (only one brand analysed) was more than 100-fold lower.

Keyword

Fluorescent whitening agents, FWA, fluorescent brighteners, FB, DSPB, DAS1, FB28, FB85 and DAS2, waste water treatment plant, WWTP, toilet paper

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Summary

A screening study has been performed concerning Fluorescent whitening agents (FWA). The term refers to a large group of chemicals used to increase the apparent whiteness of textiles and paper. The screening included five substances abbreviated DSPB, DAS1, FB28, FB85 and DAS2. The instrumental analytical method used (LC-MS-MS) is not based on the fluorescent properties of the compounds, thus FWAs other than the specific compounds analysed for was not detected.

The sampling programme was focused on diffuse emissions from urban areas reflected in samples from waste water treatment plants (WWTPs) and their receiving waters. Samples from potential industrial sources (effluent from paper and pulp industries) were also included. Emission from products was illustrated by analysing toilet paper.

The concentration range of summed FWAs was 20 - 24 µg/l in three WWTP influents, 0.43 - 5.1 µg/l in nine WWTP effluents and 56 000 – 160 000 ng/g DW in nine WWTP sludges. DAS2, FB28 and, in some effluents, DAS1 was the dominating individual compounds. DAS2 is associated to paper applications rather than textile/detergent applications.

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There is a general increase in the relative concentration of DAS1 from WWTP effluent via surface water to sediment.

In one out of seven effluents from paper and pulp industries the concentration was considerably higher (ten times) than in the highest WWTP effluent.

Analysis of FWAs extracted from toilet paper manufactured from recycled fibres (two different brands) showed that this may be a major source for FWAs in WWTP influents. The contribution from toilet paper made from virgin fibres (only one brand analysed) was more than 100-fold lower.

Sammanfattning

En screeningundersökning har utförts angående optiska vitmedel (OV). Optiska vitmedel är en stor grupp kemikalier som används för att öka den upplevda vitheten hos textilier och papper. Studien omfattade fem substanser förkortade DSPB, DAS1, FB28, FB 85 och DAS2. Den använda analytiska slutbestämningsmetoden (LC-MS-MS) bygger inte på föreningarnas fluorescensegenskaper. Därmed detekterades inte andra OV än de uppräknade.

Provtagningsprogrammet fokuserades på diffusa emissioner från urbana områden genom analys av prover från avloppsreningsverk (ARV) och deras recipienter. Prover från industriella källor (utgående vatten från skogsindustri) ingick också. Emissioner från produkter representerades av analys av toalettpapper.

Den summerade OV-koncentrationen var 20 - 24 µg/l i ingående vatten till tre ARV, 0.43 - 5.1 µg/l i utgående vatten från nio ARV och 56 000 – 160 000 ng/g TS i slam från nio ARV. De dominerande substanserna var DAS2, FB28 och, i några utgående vatten, DAS1. DAS2 är förknippat med pappers- snarare än textilapplikationer.

De högsta uppmätta koncentrationerna i ytvatten för DSPB, DAS1 och DAS2 var mer än 600 gånger lägre än beräknade PNEC-värden, d.v.s. koncentrationer där inga effekter på vattenmiljön av ämnet kan förväntas. Även utan utspädning var den högsta koncentrationen i utgående avloppsvatten mer än 50 gånger lägre än PNEC. Alltså bör dessa ämnen inte utgöra en risk för vattenlevande organismer. För de övriga substanserna fanns inga PNEC-värden att tillgå.

Högsta koncentrationen i sediment, 7 200 ng/g TS, dominerat av DAS1, uppmättes nära utsläppspunkten för ett avloppsreningsverk. På längre avstånd, 1 km resp. 20 km i den huvudsakliga flödesriktningen var koncentrationen lägre, 69 – 150 ng/g TS. I sediment från Mälaren var koncentrationen 92 – 900 ng/g TS. Relevanta PNEC-värden för bedömning av risk för sedimentlevande organismer saknas.

Man ser en generell ökning av den relativa koncentrationen av DAS1 från utgående avloppsvatten över ytvatten till sediment.

I ett av sju utgående vatten från skogsindustrier var koncentrationen förhöjd (10 gånger) jämfört med det högsta värdet från ett kommunalt reningsverk.

Analys av OV extraherat från toalettpapper tillverkat av återvunna fibrer (två fabrikat) visade att detta kan vara en viktig källa till OV-halten i ingående vatten till reningsverk. Bidraget från toalettpapper tillverkat av nya fibrer (ett fabrikat) var mer än hundra gånger lägre.

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1 Introduction

As an assignment from the Swedish Environmental Protection Agency, screening studies of Polychlorinated naphtalenes (PCN), Fluorescent whitening agents (FWA) and Pharmaceuticals have been performed during 2010/2011.

The overall objectives of the screening studies are to determine 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 presented in three separate reports according to Table 1.

Table 1. Substance groups included in the screening.

Substance group	Sub-report #
Polychlorinated naphtalenes (PCN)	1
Fluorescent whitening agents	2
Pharmaceuticals	3

The screening study has been carried out by Swedish Environmental Research Institute (IVL) together with Umeå University (UmU). The chemical analyses of the fluorescent whitening agents were undertaken at IVL, PCN and pharmaceuticals were analysed at UmU.

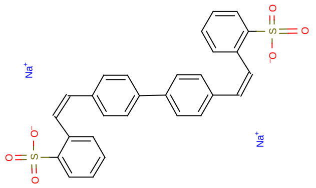
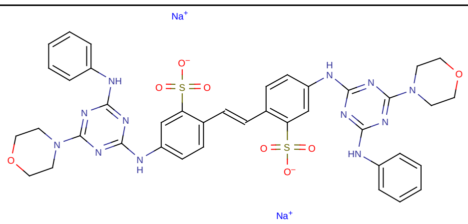
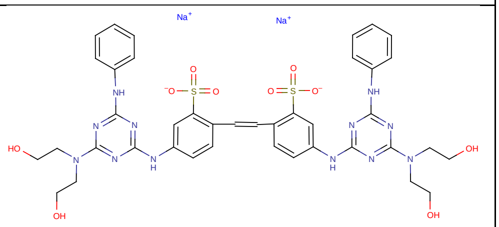
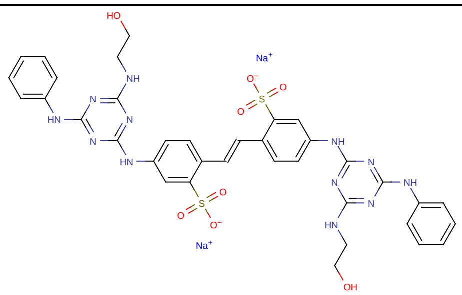
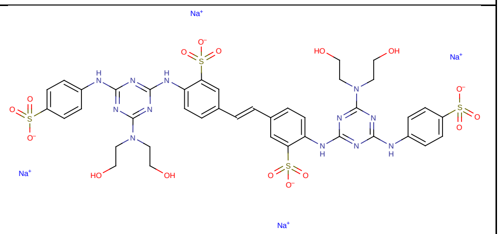
This sub-report concerns the screening of fluorescent whitening agents, FWA, also called fluorescent brighteners, FB. The term refers to a large group of chemicals used to increase the apparent whiteness of textiles and paper. The substances have the ability to absorb invisible ultraviolet radiation in the wavelength range 300-400 nm and emit visible blue light (400-500 nm) which makes surfaces look whiter.

2 Chemical substances, properties and use

2.1 Substances included in the screening

The screening includes five substances which are listed in Table 2 were also CAS-numbers and chemical structures are given.

Table 2 Substances included in the screening

Abbreviation, CAS#	Structural formula
DSBP Distyrylbiphenylsulfonate 27344-41-8	
DAS1 16090-02-1	
FB28 4193-55-9	
FB85 12224-06-5	
DAS2 16470-24-9	

DAS1, FB28, FB85 and DAS2 all share a common triazinylaminostilbene structure with different groups attached to it. The compounds could be classified according to the number of sulphonic acid groups in the molecule. Thus DSBP, DAS1, FB28 and FB85 are disulphonic FWAs; DAS2 is a tetrasulphonic FWA.

The naming of FWAs is sometimes confusing. The numbering is not consistent and they are sold under a variety of innovative trade names.

2.2 Properties

FWAs generally have high water solubility and also a tendency to associate to organic surfaces. They are not readily biodegradable (Engström 2009).

During sewage treatment FWAs (DSPB and DAS1) are removed exclusively by adsorption to sludge. Biodegradation is not observed in either the aerobic biological stage or in the anaerobic sludge treatment (Poiger 1999).

The predicted no-effect concentration (PNEC) is the concentration below which exposure to a substance is not expected to cause adverse effects. Calculated PNECs in water for selected FWAs are presented in Table 3. PNECs for sediment living organisms were not available.

Table 3 Calculated PNECs in water for DSPB, DAS1 and DAS2 (Engström, 2009)

Substance	Used assessment factor	PNEC, µg/l
DSPB	50	20
DAS1	100	68.5
DAS2	50	200

The fluorescence of FWAs in aqueous solution decreases upon exposure to sunlight (photofading). This is due to isomerization from the fluorescent E-isomer to the non fluorescent Z-isomer, yielding a steady state between the two isomers. Photofading rates in river water exposed to natural sunlight has been reported as 7% for DSPB and 71% for DAS1 after 60 minutes. Further degradation is much slower. Effective half-lives in the environment are dependent on season, clouds and screening of light by natural waters in deeper layers. The degradation of DSPB will be approximately 3 times faster than for the DAS-type FWAs (Kramer 1996).

2.3 Production and use

The registered annual use of selected FWAs in Sweden is listed in Table 4 and illustrated in Figure 1.

Table 4 Registered use of selected FWAs in Sweden 1999-2009, tonnes (SPIN, 2011)

Year	DSBP	DAS1	FB28	DAS2	FB263
1999	0	0	5	1170	0
2000	0	0	17	1371	0
2001	1	1	8	801	0
2002	6	0	179	2141	1
2003	0	0	300	1642	35
2004	0	0	346	1658	138
2005	3	0	326	1759	96
2006	2	0	112	1263	197
2007	2	0	796	1314	333
2008	2	1	1241	1204	681
2009	3	15	1233	819	557

The annual use of DAS2 has been fairly constant during the period 1999 – 2009 but the use of FB28 and FB263 has increased. All three are now used in the 1 000 tonnes per year range. The use of DSBP and DAS1 is considerably lower (≤15 tonnes per year).

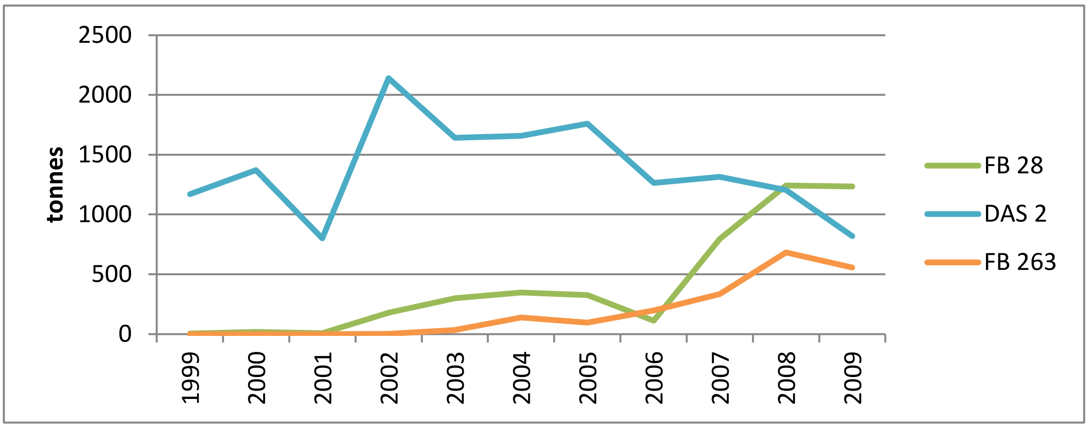


Figure 1 Annual use of selected FWAs in Sweden 1999 - 2009.

DSPB and DAS1 are mainly used in detergent applications while DAS2 has paper-related use (Kramer, 1996).

As it was not possible to obtain FB263 as an analytical standard this substance was not included among the analysed FWAs.

3 Previous measurements in the environment

A selection of previously reported environmental concentrations for DAS1 is cited in Table 6, for DSPB in Table 5 and for FB28 in Table 7. Data for FB85 or DAS2 was not found. Table 8 lists concentrations where FWAs are reported as a sum. The abbreviation WWTP refers to waste water treatment plant.

Table 5 Previously reported concentrations of DSPB in different matrices

Concentration	Matrix	Reference
185 ng/l	WWTP effluent, Taiwan, 2006	Chen H-C et al , 2006
3.3 – 8.9 µg/l	WWTP effluent, Switzerland, 1986	Kramer et al 1996 (citing a conf rep 1986)
0.04 – 0.6 µg/l	river water, Switzerland, 1994	Kramer et al 1996 (citing dissertation 1994)
12 ng/l	surface water, Lake Greifensee, Switzerland, 1995, july	Stoll & Giger 1997
71 ng/l	surface water, Lake Greifensee, Switzerland, 1995, january	Stoll & Giger 1997
19 – 434 ng/l	river water, Japan, 11 rivers, 2003,2005 summer	Hayakawa et al 2007
49 – 939 ng/l	river water, Japan, 11 rivers, 2002, winter	Hayakawa et al 2007
430 – 1140 µg/kg DW	sediment, Lake Greifensee, Switzerland, 1995	Stoll & Giger 1997

Table 6 Previously reported concentrations of DAS1 in different matrices.

Concentration	Matrix	Reference
42 ng/l	WWTP effluent, Taiwan, 2006	Chen H-C et al , 2006
2.6 – 4.5 µg/l	WWTP effluent, Switzerland, 1986	Kramer et al 1996 (citing a conf rep 1986)
0.04 – 0.4 µg/l	river water, Switzerland, 1994	Kramer et al 1996 (citing dissertation 1994)
53 ng/l	surface water, Lake Greifensee, Switzerland, 1995, july	Stoll & Giger 1997
98 ng/l	surface water, Lake Greifensee, Switzerland, 1995, january	Stoll & Giger 1997
28 – 661 ng/l	river water, Japan, 11 rivers, 2003,2005 summer	Hayakawa et al 2007
26 – 901 ng/l	river water, Japan, 11 rivers, 2002, winter	Hayakawa et al 2007
650 – 1420 µg/kg DW	sediment, Lake Greifensee, Switzerland, 1995	Stoll & Giger 1997

Table 7 Previously reported concentrations of FB28 in different matrices

Concentration	Matrix	Reference
1580 ng/l	WWTP effluent, Taiwan, 2006	Chen H-C et al , 2006
100 – 145 ng/l	river water, receiving untreated wastewater, Taiwan, 2006	Chen H-C et al , 2006

Table 8 Previously reported concentrations of summed optical whiteners

Concentration	Matrix	Reference
10 – 20 µg/l	WWTP influent, Switzerland 1994	Kramer et al 1996 (citing dissertation 1994)
82.7 mg/kg	WWTP sludge, Sweden. 1994	Stockholm Vatten, 2003
85 – 170 mg/kg DW	WWTP sludge, 9 WWTPs, Switzerland, 1993	Poiger 1994

4 Sampling strategy and study sites

4.1 Screening program

A sampling strategy was developed in order to determine concentrations of FWAs in the Swedish environment and to identify the important pathways. The sampling programme was focused on diffuse emissions from urban areas reflected in samples from waste water treatment plants (WWTPs) and their receiving waters. The individual samples are listed in Appendix 1.

The measurements included influent, effluent and sludge from WWTPs. Surface water and sediment samples were collected both at background locations and in the urban area Stockholm. Surface water upstream and at several locations downstream the WWTP in Uppsala were analysed.

Samples from potential industrial sources (effluent from paper and pulp industries) were also included. Emission from products was illustrated by analysing toilet paper.

The sampling program is summarized in Table 9.

Table 9 National sampling program

Type	WWTP Influent	WWTP Effluent	WWTP Sludge	Surface water	Sediment	Other	Total
Background areas							
Lakes				2	2		4
Baltic Sea					2		2
Urban areas							
Stockholm	1	1	1	3	6		12
WWTP Uppsala	1	1	1	5			8
other WWTPs	1	5	5				11
Point source							
Effluent , paper and pulp industry						7	7
Products							
Toilet paper						3	
Total	3	7	7	10	10	10	47

In addition to the national screening program Swedish county administrative boards had the opportunity to collect and send samples for analysis. The administrative county board in Värmland participated with effluent and sludge from two municipal WWTPs, Table 10.

Table 10 Regional sample program

Type	WWTP Effluent	WWTP Sludge	Total
Urban areas			
WWTPs	2	2	4
Total	2	2	**4**

5 Methods

5.1 Sampling

Surface waters were sampled directly into 1 litre polyethylene bottles.

Surface sediments (0-2 cm) from shallow waters were collected by means of a Kajak sampler. The sediment was transferred into muffled (400 ºC) glass jars. Deep sea sediments were provided by SGU. Sediments were stored in a freezer (-20 ºC) until analysed.

The staff at the different WWTPs collected influent and effluent water samples in 1 litre PE bottles de-watered sludge samples from the anaerobic chambers into PE jars. The samples were stored frozen (-18°C) until analysis.

5.2 Analysis

The analytical method used was not based on the fluorescent properties of the compounds. FWAs other than the specific compounds analysed for was thus not detected. Conformational changes causing photofading (2.2) did not affect the results.

DAS2 was purchased as a solution (Blankopher phorwhite BBU, Fluorochem) with no declaration of water content. Drying at 50°C gave a solid residue of 21%. All calculated concentrations refer to this solid content. All other FWAs were purchased as solids and no correction for purity was done.

5.2.1 Sample preparation

The method was adopted from Chen H-C et al (2006) and Chen H-C, Ding W-H (2006). Water samples were solid phase extracted using Oasis WAX, 60 mg (Waters). The analytes were eluted with MeOH+ACN (1+4, v/v) containing 2% ammonium hydroxide, the extract evaporated to dryness and redissolved in water+ACN (1+1, v/v) containing the internal standard CI 248060, (CAS 5463-64-9).

Wet sludge, approximately 1 g, was diluted with 25 ml MilliQ water heated to 70°C for 1 h with occasional shaking. After centrifugation 2.5 ml of the water phase was safeguarded, the rest was discarded. 25 ml fresh water was added and the extraction repeated twice. The combined safeguarded water phases were analysed as described for water.

Wet sediment, approximately 4 g, was extracted with 25 ml MilliQ water heated to 70°C for 1 h with occasional shaking. After centrifugation the water phase was safeguarded. 25 ml fresh water was added and the extraction repeated three times. The combined safeguarded water phases were analysed as described for water.

To toilet paper, approximately 0.5 g, 50 ml water (MilliQ) was added. The mixture was heated to 80°C for 2 h with occasional shaking. After centrifugation 30 ml of the supernatant was analysed as described for water.

5.2.2 Instrumental analysis

Liquid chromatography was performed on a Prominence UFLC system (Shimadzu) with two pumps LC-20AD, degasser DGU-20A5, autosampler SIL-20ACHT and column oven CTO-20AC. The analytical column was an Thermo HyPurity C8 50 mm x 3 mm, particle size 5 µm (Dalco Chromtech). The mobile phase A was 5 mM di-n-hexylammonium acetate in water, the mobile phase B was methanol. A gradient from 40% to 100% B was run. The column temperature was 35°C and the flow rate 0.4 ml/min.

The effluent was directed to an API 4000 triple quadrupole mass spectrometer (Applied Biosystems). Electrospray ionisation in negative mode with MRMs according to Table 11 was used.

Table 11 list of used MRMs

	MRM1, quantifier	MRM2, qualifier
DSPB	517.2 > 437.1	517.2 > 79.5
DAS1	879.3 > 799.2	879.3 > 569.0
FB28	915.3 > 587.2	915.3 > 835.3
FB85	827.2 > 623.3	827.2 > 747.3
DAS2	1075.3 > 747.0	
IS	677.0> 597.0	

5.2.3 Quality control

Three different WWTP effluents were analysed in duplicate. Two different sample volumes (1 and 5 ml) were used for each sample. The resulting average coefficients of variation were 8.4, 7.5, 3.7, 15 and 22% for DSPB, DAS1, FB28, FB85 and DAS2 respectively.

Aliquots (50 ml) of a surface water (sample #8939) was analysed in duplicate without spiking. The same sample with spiking (1 ng per analyte, 2.1 ng for DA2) was analysed in pentaplicate. The average recovery was 111, 78, 88, 97 and 176% for DSBP; DAS1, FB28, FB85 and DAS2 respectively. Three times the standard deviation of the measured amounts for the spiked samples equalled 0.80, 0.67, 1.2, 0.62 and 4.2 ng for DSBP; DAS1, FB28, FB85 and DAS2 respectively. These results were used to calculate detection limits also when larger sample amounts were used.

6 Results and discussion

The results of the measurements of the fluorescent whitening agents are presented in detail in Appendix 2 where the concentrations of the individual substances are given. Results for different sample types are presented below.

6.1 Background areas

FWAs were not detected in surface water or sediment from freshwater background lakes. The detection limits were in the range 10 – 35 ng/l and 5 – 10 ng/g DW for individual compounds. Low concentrations of DAS1 and DAS2 were detected in brackish water deep sea sediments. The concentrations were 8.8 and 13 ng/g DW at Härnösandsdjupet and 25 and 20 ng/g DW at Landsortsdjupet (see 6.4, Figure 10). DSBP, FB28 or FB85 were not found.

6.2 Waste water treatment plants

6.2.1 WWTP influent

The FWA concentrations in influent water to three waste water treatment plants were measured at one occasion. The WWTPs are located in Uppsala (Kungsängsverket), Stockholm (Henriksdal) and Umeå (Öhn) and are all relatively large, treating water from approximately 160 000, 835 000 and 100 000 person equivalents respectively.

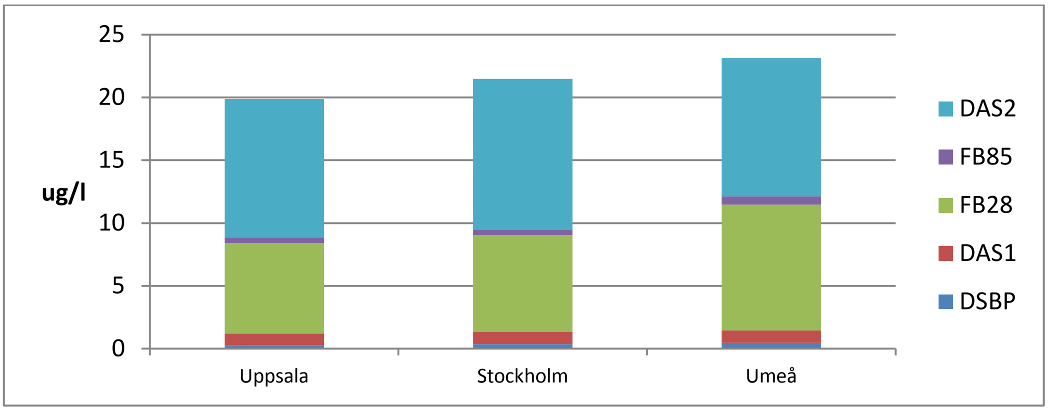


Figure 2 Concentration of FWAs in influent to WWTPs.

The FWA concentration in the three influents were very similar with a summed concentration for the five substances of 20 – 24 µg/l. DAS2 and FB28 dominated with concentrations around 10 µg/l, DAS1, FB85 and DSBP followed with concentrations around 1 µg/l or less.

The summed concentrations are is in agreement with what has been reported from Switzerland (Table 8).

6.2.2 WWTP effluent

The FWA concentrations in effluents from nine municipal waste water treatment plants were measured at one occasion. The sizes of the WWTPs are from medium to large.

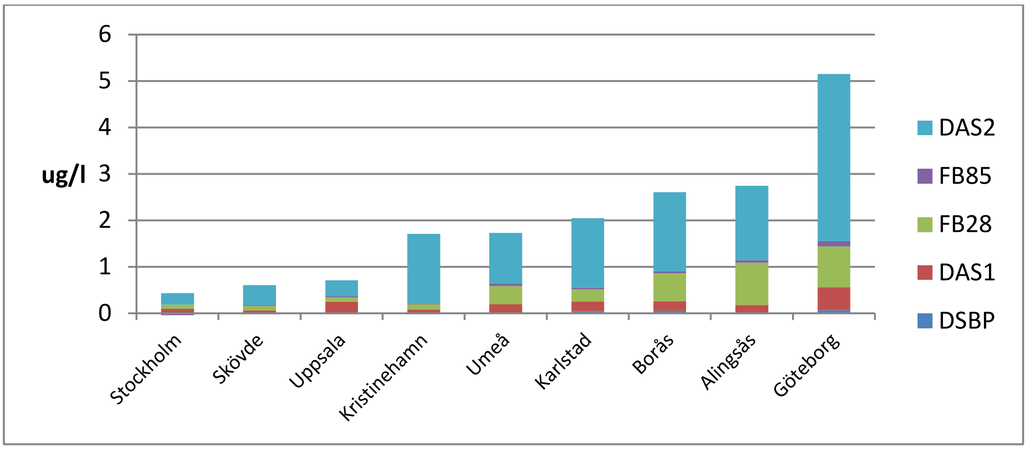


Figure 3 Concentration of FWAs in effluents from WWTPs.

The summed FWA concentration in the nine effluents varied from 0.43 ug/l (Stockholm) to 5.1 µg/l (Göteborg), Figure 3.

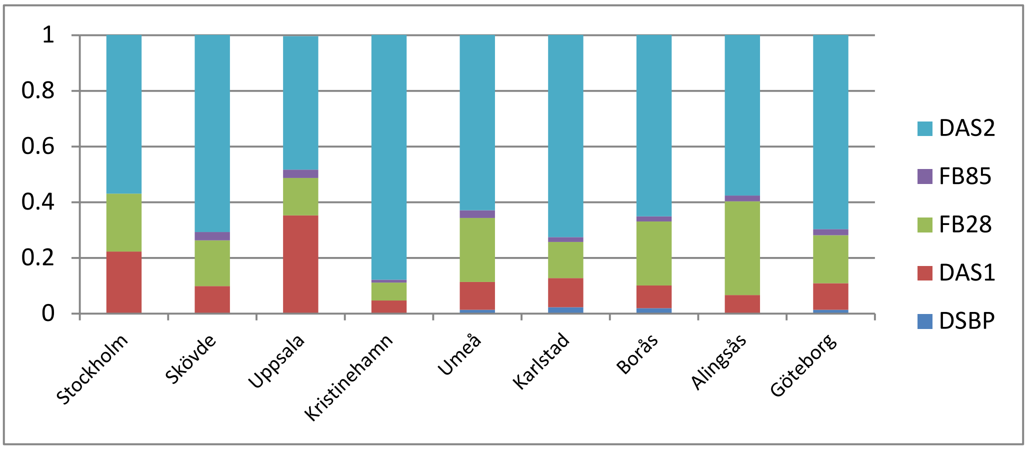


Figure 4 Relative concentration of FWAs in effluents from WWTPs.

DAS2 dominated in all samples (0.25 – 3.6 µg/l), making up 48% – 88% of the summed concentrations. FB28 (0.089 – 0.91 µg/l) and DAS1 (0.059 – 0.49 µg/l) followed next while FB85 and DSBP each made up less than 3% of the sum, Figure 4.

The concentrations of DSPB and FB28 are lower than published results for WWTP effluents from Switzerland and Taiwan (Table 5, Table 7). The concentrations of DAS1 are lower than published results from Switzerland but higher than one result from Taiwan (Table 6).

6.2.3 Removal efficiency

From the three WWTPs where both influent and effluent was analysed the removal efficiency ((Infl – Effl) / Infl) was calculated (Figure 5). The efficiency was 90% or higher in all cases with the exception of DAS1 in Uppsala and Umeå (73% and 83%).

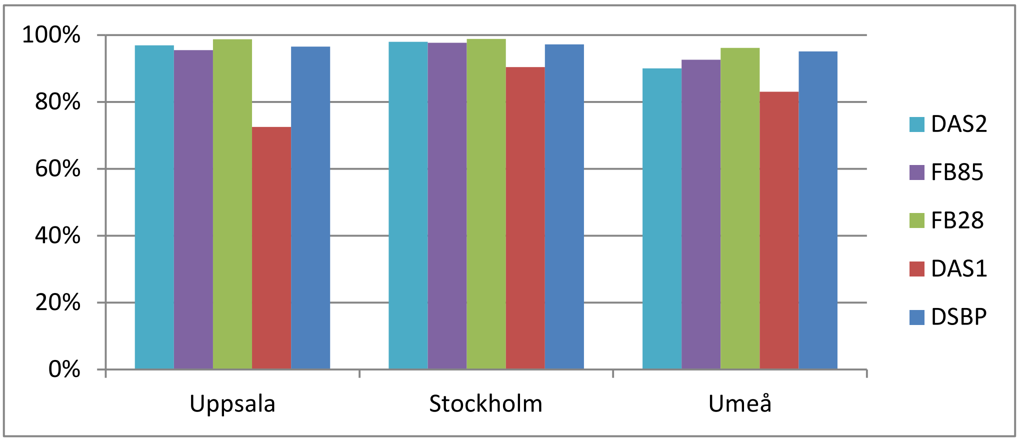


Figure 5 Removal efficiency

6.2.4 WWTP sludge

The FWA concentrations in sludge from the nine municipal waste water treatment plants sampled at one occasion are presented in Figure 6.

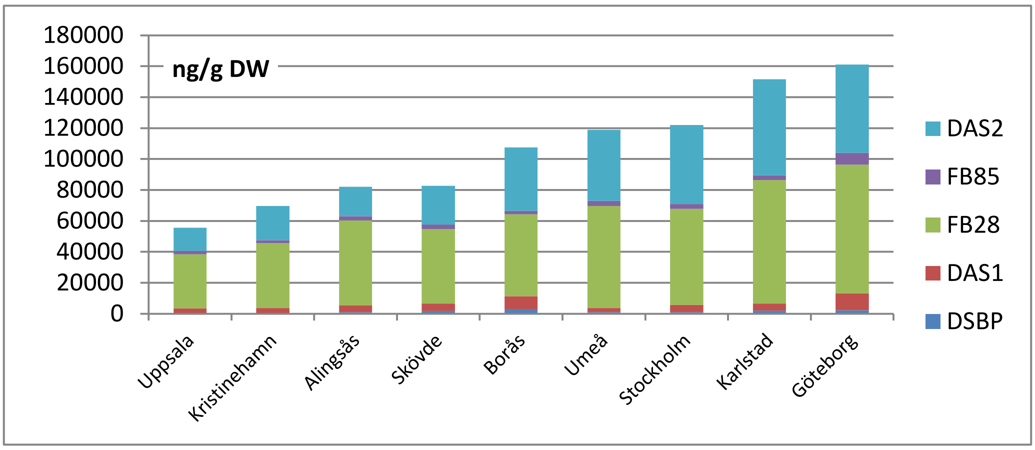


Figure 6 Concentration of FWAs in sludge from WWTPs.

The summed concentrations varied from 56 000 to 160 000 ng/g DW with a median of 110 000 ng/g DW. FB28 and DAS2 were the dominating FWA in all the sludge samples. The median, min and max concentrations are summarized in Table 12.

Table 12 Median, min and max concentrations of FWAs in sludge from WWTPs (n=9), ng/g DW.

	Median ng/g DW	Min ng/g DW	Max ng/g DW
Sum FWAs	110 000	56 000	160 000
FB28	55 000	35 000	83 000
DAS2	41 000	15 000	62 000
DAS1	4 500	2 800	11 000
FB85	2 900	1 900	7 700
DSBP	1 100	430	3 100

The results for summed FWAs are in good agreement with previously reported results (Table 8).

6.3 Surface water

The effluent from Kungsängsverket WWTP, Uppsala, flows into the streaming water Fyrisån. Surface water sampled upstream (1.7 km) and at four points downstream (5 m, 150 m, 3.5 km and 4.6 km) from the discharge point was analysed. The concentration of FWAs (DAS1, FB28 and DAS2) clearly increased in the first downstream sample and then sequentially decreased in the following samples (Figure 7). The annual average flow in Fyrisån is 8.6 m^3/s and the average effluent flow from Kungsängsverket WWTP is 2 200 m^3/h (Uppsala vatten, 2011). This gives a mean dilution factor of 14. The dilution factors for DAS1 and DAS2 obtained when the surface water concentrations are compared to the analysed effluent water sample (6.2.2) are illustrated in Figure 8. The sequentially decreasing concentrations in the surface water is probably mainly the result of improved mixing of the effluent into the streaming water.

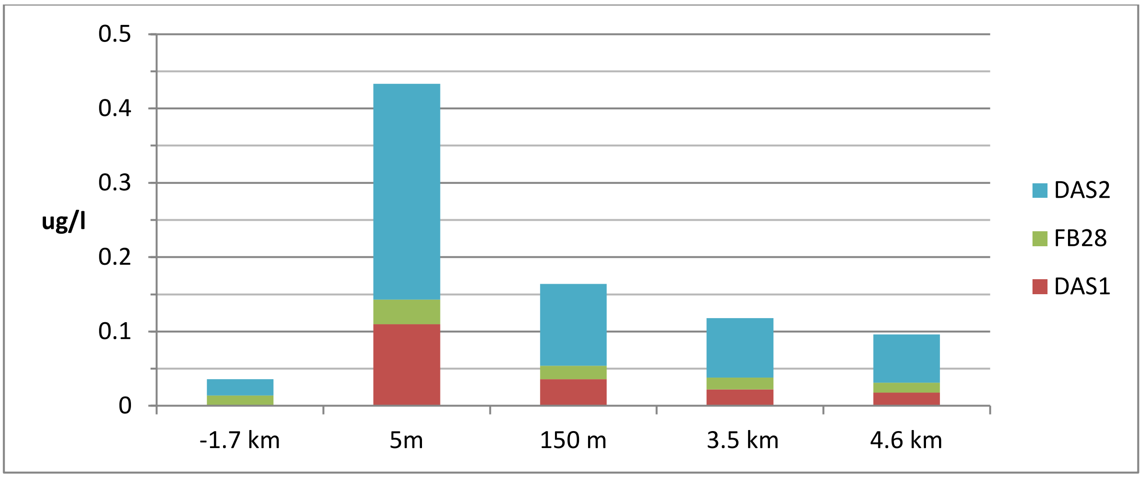


Figure 7 Concentration of FWAs in surface water downstream Kungsängsverket

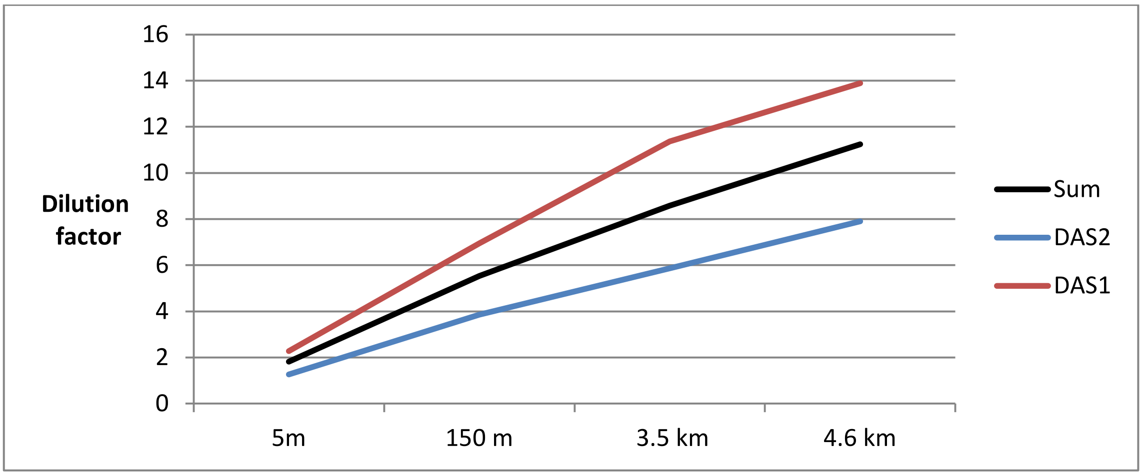


Figure 8 Dilution factor for DAS1 and DAS2 in surface water relative to WWTP effluent water downstream Kungsängsverket.

Surface water was sampled in Årstaviken and Riddarfjärden, both in Lake Mälaren in Stockholm, and at Blockhusudden which is in the inner archipelago downstream of the discharge point of Henriksdal WWTP. All concentrations are close to or lower than what was found in Fyrisån upstream Kungsängsverket WWTP (Figure 9).

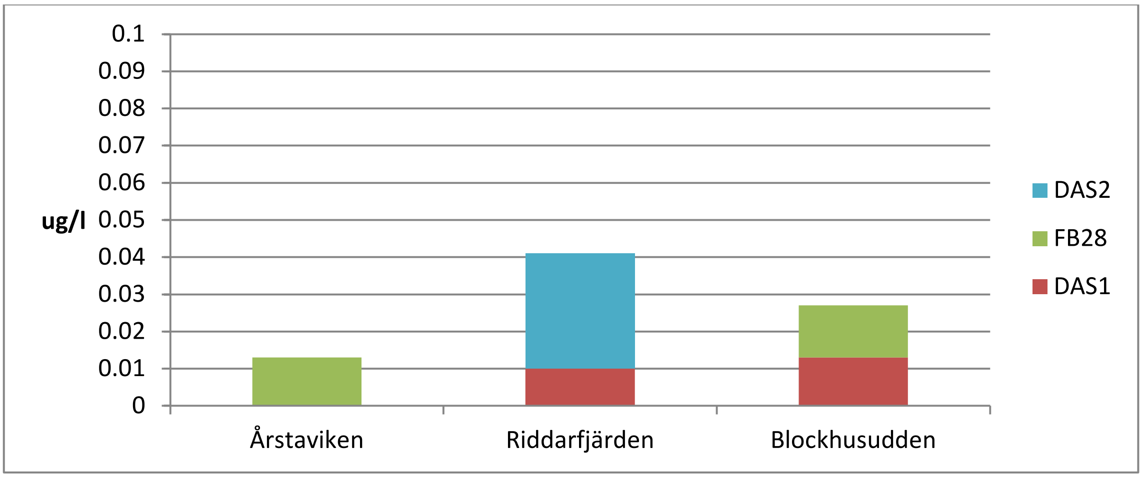


Figure 9 Concentration of FWAs in surface water, Stockholm (note the different scale from Figure 7)

6.4 Sediment

FWA concentrations in sediments are illustrated in Figure 10. Low concentrations of DAS1 and DAS2 were detected in brackish water deep sea sediments (6.1). DAS1, DAS2 and also FB28 in higher concentrations (26 – 430 ng/g DW per compound) were found in sediments from Lake Mälaren (St Essingen, Årstviken, Riddarfjärden) Stockholm. Highest concentrations (4 400, 1 600 and 1 200 ng/g DW for DAS1, DAS2 and FB28) were found at Valdemarsudde which is near the discharge point of Henriksdal WWTP. At 1 km (Biskoppsudden) and 20 km (Torsbyfjärden) away from the effluent point, in the main direction of flow, concentrations were in the low range of what was measured in Lake Mälaren.

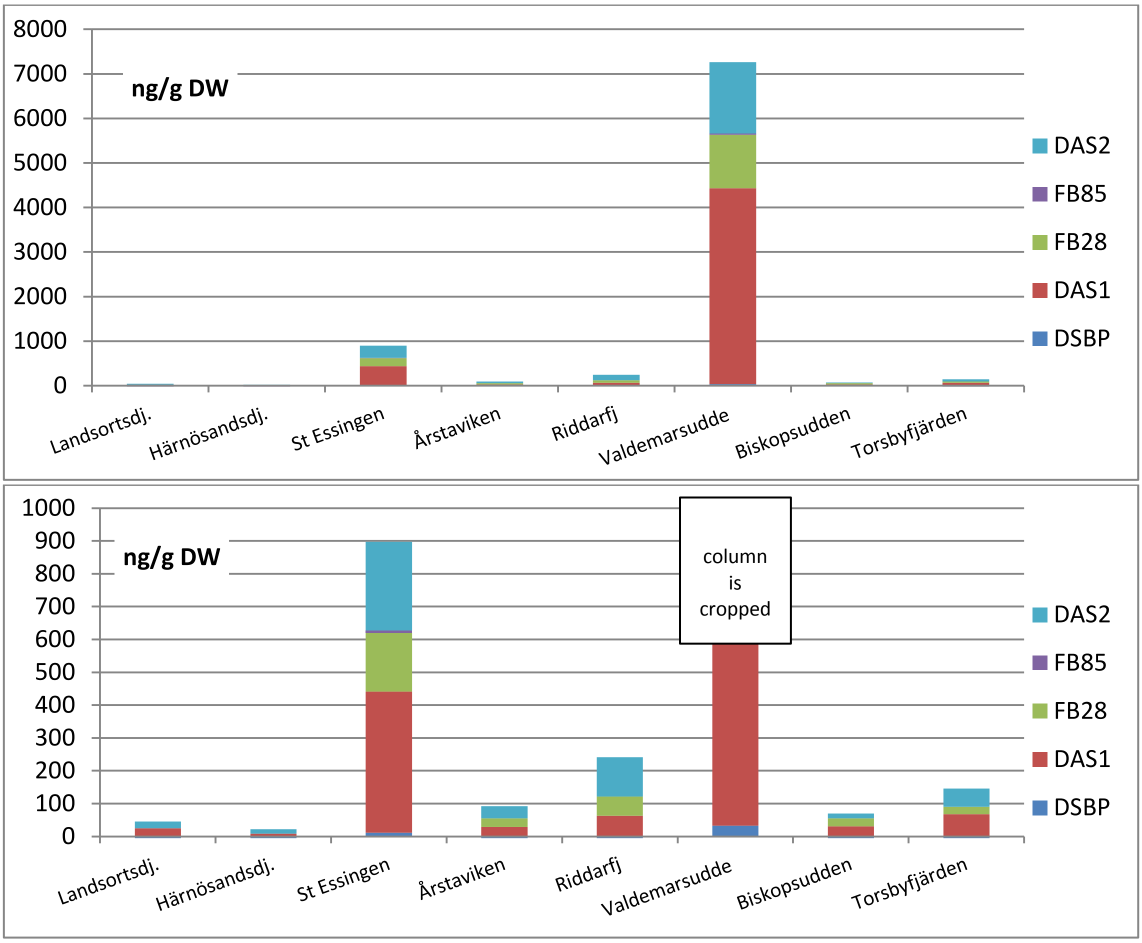


Figure 10 Concentration of FWAs in sediments. Both graphs show the same data but to different scales.

The relative concentrations of DAS1, FB28 and DAS2 in the sediments are illustrated in Figure 11.

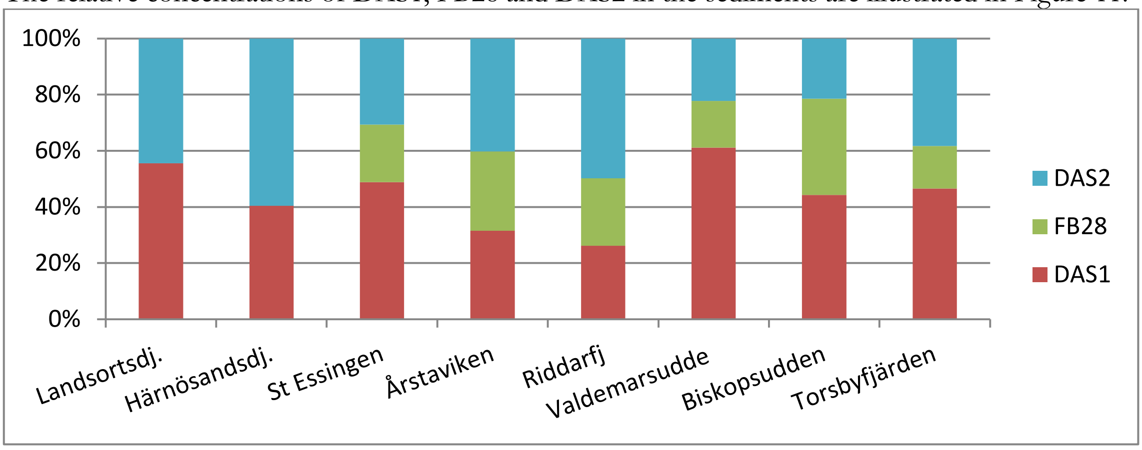


Figure 11 Relative concentration of DAS1, FB28 and DAS2 in sediments.

There is an increase in the relative concentration of DAS1 (median of all measured values) from WWTP effluent (11 %) via surface water (23 %) to sediment (45 %).

7 Effluent from pulp and paper industries

Information of use of specific FWAs in different industries was not easily obtained. Thus combined effluents from seven pulp and paper industries available to the laboratory for other purposes were analysed. DSPB, DAS1, FB28 and FB85 concentrations were all <1 µg/l. DAS2 was 39 µg/l in one case (industry 6) and <4 in the others.

In two cases (not industry 6) influents to an internal treatment facility were also available. In one of those samples the concentrations of FB28 and DAS2 were 6.8 µg/l and 13 µg/l. All other concentrations were <1 µg/l (DSPB, DAS1, FB28 and FB85) or <4 µg/l (DAS2).

8 Toilet paper

To illustrate potential emissions from consumer products a small study on toilet paper was made. Three brands of household toilet paper were purchased in the same grocery store in Stockholm. They were labelled TP1, TP2 and TP3. Some of the text on the packages are summarised in Table 13. All three carried the Nordic Ecolabel.

Table 13 List of toilet papers

	Text
TP1	100 % recycled fibre, made in Scandinavia by white recycled fibre
TP2	100 % new fibre, virgin fibre, this product does not contain any re-cycled paper
TP3	100 % recycled fibre, produced in the Nordic countries

The results of analysis for FWAs are listed in Table 14. The dominating FWAs are DAS2 and FB28. The concentrations in TP1 and TP3 (recycled fibre) are more than 100 times higher than in TP2 (virgin fibre). The difference is striking and may perhaps be interpreted as a general difference between virgin and recycled fibres.

Table 14 Result of analysis of toilet papers

		DSPB, µg/g	DAS1, µg/g	FB28, µg/g	FB85, µg/g	DAS2, µg/g	Sum, µg/g
TP1	recycled fibre, white	0.18	0.031	120	16	470	610
TP2	virgin fibre	<0.01	<0.01	0.96	0.082	1.4	2.4
TP3	recycled fibre	0.23	<0.01	36	25	310	370

Most toilet paper will ultimately end up as part of the influent to a WWTP. Do FWAs originating from toilet paper contribute significantly to the concentration in the influent? The annual consumption of toilet paper in Sweden is around 15 kilo per person (Icakuriren 2010). The annual effluent volume per person could be estimated to 130 m^3 (calculated from Stockholm Vatten 2010). This equals 0.12 g toilet paper per litre effluent. The concentration obtained of dissolving the amount of FWAs found in 0.12 g of TP1, TP2 and TP3 respectively in one litre of water is illustrated in Figure 12. The leftmost bar shows the average concentration measured in influent waters (6.2.1).

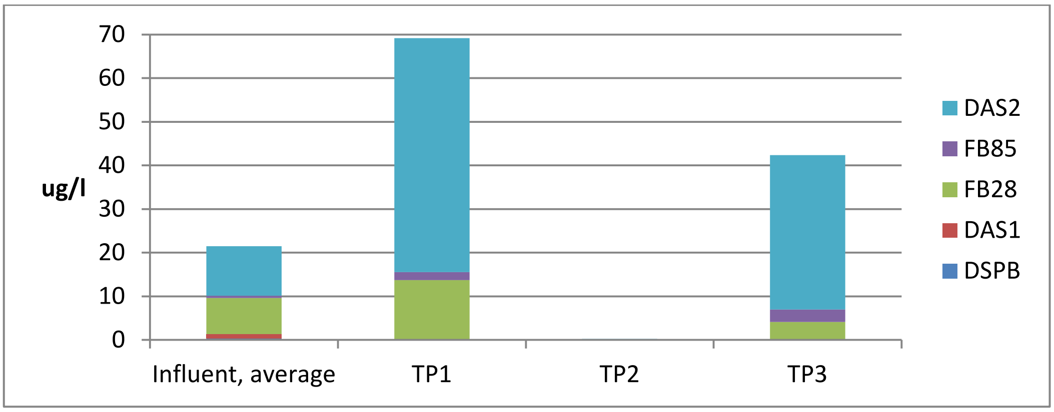


Figure 12 Average concentration in WWTP influents compared to estimated concentration obtained from toilet papers TP1, TP2 and TP3 (see text).

The figure shows that if all toilet paper used was of type TP1 or TP3 this would be more than enough as a source to the DAS2 and FB85 measured in the average influent. In fact it would be sufficient if 25-30 % would be of this type. It would also be an important source for FB28 but not for DAS1 or DSPB. On the other hand, even if all toilet paper was of the TP2 type it would only give a minor contribution to the concentration of FWAs in the influent.

9 Conclusions

All analysed FWAs (DSPB, DAS1, FB28, FB85 and DAS2) were detected in WWTP influent, effluent and sludge. The dominating FWA (DAS2) is associated to paper applications rather than textile/detergent applications.

The removal efficiency is generally > 90%. The removal mechanism is, according to the literature, exclusively adsorption to sludge.

The concentration in sludge (median of summed FWAs) was roughly ten times higher than previously found concentrations of siloxanes (D4-D6, Kaj et al 2005) but six times lower than previously found concentrations of LAS (Kaj et al 2008).

Toilet paper can be a considerable source for FWAs entering WWTPs.

In only one out of seven effluents from paper and pulp industries the concentration was considerably higher (ten times) than in the highest WWTP effluent.

The highest measured concentrations in surface water for DSPB, DAS1 and DAS2 were more than 600 times lower than the calculated PNECs (Table 3). Even without dilution the highest measured concentration in WWTP effluent were more than 50 times lower than the calculated PNECs. Thus there should be no risk for aquatic organisms due to those substances. For the remaining substances PNECs were not available.

Relevant PNECs for evaluation of risks for sediment dwelling organisms were not available.

10 Acknowledgement

The staffs at the municipal sewage treatment plants are acknowledged for their help during sampling. Also Tomas Viktor and Mikael Remberger, IVL, contributed with sampling. Karl Lilja, IVL, reviewed the sections on eco-toxicity.

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(SPIN, 2011)

Appendix 1 Sample table

Sample#	Nat/ County	Type	Municipality	Site	Matrix	Sampling date	Coord. RT90	
8712	Nat	Backgr	Katrineholm	Älgsjön	Surface water	2010-09-08	6552912	1532301
9069	Nat	Backgr	Vallentuna	Tärnan	Surface water	2011-02-13	6608668	1644632
8713	Nat	Backgr	Katrineholm	Älgsjön	Sediment	2010-09-08	6552912	1532301
8714	Nat	Backgr	Vallentuna	Tärnan	Sediment	2010-09-07	6608668	1644632
7743	Nat	Backgr		Deep sea sed., Landsortsdjupet	Sediment	Sept 2008	6508995	1648002
7737	Nat	Backgr		Deep sea sed., Härnösandsdjupet	Sediment	Sept 2008	6956505	1663305
8869	Nat	Diffuse	Stockholm	Årstaviken	Surface water	2010-10-30	6578916	1627145
7960	Nat	Diffuse	Stockholm	Årstaviken	Sediment	2009-09-16	6578147	1628330
8870	Nat	Diffuse	Stockholm	Riddarfjärden	Surface water	2010-10-30	6580129	1626978
7968	Nat	Diffuse	Stockholm	Riddarfjärden	Sediment	2009-09-16	6580141	1627276
7962	Nat	Diffuse	Stockholm	St Essingen	Sediment	2009-09-16	6579241	1623643
8955	Nat	Diffuse	Stockholm	Henriksdal WWTP	Influent	2010-11-29		
8958	Nat	Diffuse	Stockholm	Henriksdal WWTP	Effluent	2010-11-29		
8961	Nat	Diffuse	Stockholm	Henriksdal WWTP	Sludge	2010-12-01		
8874	Nat	Diffuse	Stockholm	Blockhusudden	Surface water	2010-11-04	6580307	1633813
7964	Nat	Diffuse	Stockholm	(Gr1) Valdemarsudde	Sediment	2009-09-16	6579672	1631232
7966	Nat	Diffuse	Stockholm	(Gr2) Biskopsudden	Sediment	2009-09-16	6579872	1632622
7986	Nat	Diffuse	Stockholm	(Gr3) Torsbyfjärden	Sediment	2009-09-17	6584391	1649834
8964	Nat	Diffuse	Uppsala	Kungsängsverket WWTP	Influent	2010-10-28		
8967	Nat	Diffuse	Uppsala	Kungsängsverket WWTP	Effluent	2010-10-28		
8970	Nat	Diffuse	Uppsala	Kungsängsverket WWTP	Sludge	2010-10-29		
8939	Nat	Diffuse	Uppsala	upstream Kungsängsv. WWTP, Islandsfallet, -1.7 km	Surface water	2010-10-26	6638712	1602921
8940	Nat	Diffuse	Uppsala	downstream Kungsängsv. WWTP 5 m	Surface water	2010-10-26	6637312	1603770
8941	Nat	Diffuse	Uppsala	downstream Kungsängsv. WWTP 150 m	Surface water	2010-10-26	6637256	1603806
8942	Nat	Diffuse	Uppsala	downstream Kungsängsv. WWTP, Ultuna, 3.5 km	Surface water	2010-10-26	6634102	1604680
8943	Nat	Diffuse	Uppsala	downstream Kungsängsv. WWTP, Flottsund, 4.6 km	Surface water	2010-10-26	6631161	1604268
8888	Nat	Diffuse	Umeå	Öhn WWTP	Influent	2010-10-19 - 20		
8889	Nat	Diffuse	Umeå	Öhn WWTP	Effluent	2010-10-19 - 20		
8890	Nat	Diffuse	Umeå	Öhn WWTP	Sludge	2010-10-21		
8882	Nat	Diffuse	Borås	Gässlösa WWTP	Effluent	2010 v. 38		
8883	Nat	Diffuse	Borås	Gässlösa WWTP	Sludge	2010-09-23		
8886	Nat	Diffuse	Alingsås	Nolhaga WWTP	Effluent	2010 v. 38		
8887	Nat	Diffuse	Alingsås	Nolhaga WWTP	Sludge	2010-09-23 - 24		
8884	Nat	Diffuse	Göteborg	Ryaverken WWTP	Effluent	2010-10-04 - 11		
8885	Nat	Diffuse	Göteborg	Ryaverken WWTP	Sludge	2010-10-06		
8929	Nat	Diffuse	Skövde	Skövde WWTP	Effluent	2010-11-16		
8938	Nat	Diffuse	Skövde	Skövde WWTP	Sludge	2010-11-17		
781	Nat	Point source		Industry 1	Effluent	2011-Mar		
787	Nat	Point source		Industry 2	Effluent	2010-Dec		
824	Nat	Point source		Industry 3	Effluent	2011-Mar		
825	Nat	Point source		Industry 4, influent to internal treatment	Influent	2011-Mar		
826	Nat	Point source		Industry 4	Effluent	2011-Mar		
828	Nat	Point source		Industry 5, influent to internal treatment	Influent	2011-Mar		

Sample#	**Nat/ County**	**Type**	**Municipality**	**Site**	**Matrix**	**Sampling date**	**Coord. RT90**	
829	Nat	Point source		Industry 5	Effluent	2011-Mar		
830	Nat	Point source		Industry 6	Effluent	2011-Mar		
831	Nat	Point source		Industry 7	Effluent	2011-Mar		
TP1	Nat	Diffuse		TP1, see table 13	Toilet paper	2011 Aug		
TP2	Nat	Diffuse		TP2, see table 13	Toilet paper	2011 Aug		
TP3	Nat	Diffuse		TP3, see table 13	Toilet paper	2011 Aug		
8810	Värmland	Diffuse	Karlstad	Sjöstadsverket WWTP	Effluent	2010-10-10		
8811	Värmland	Diffuse	Karlstad	Sjöstadsverket WWTP	Sludge	2010-10-10		
8790	Värmland	Diffuse	Kristinehamn	Fiskartorpet WWTP	Effluent	2010-09-30		
8791	Värmland	Diffuse	Kristinehamn	Fiskartorpet WWTP	Sludge	2010-09-30		

Appendix 2 Results table

Sample #	Nat/ County	Type	Municipality	Site	Matrix	Unit	DSBP	DAS1	FB28	FB85	DAS2	Sum	Karbamazepin
8712	Nat	Backgr	Katrineholm	Älgsjön	Surface water	ug/l	<0.019	<0.013	<0.013	<0.015	<0.035	<0.095	0.012
9069	Nat	Backgr	Vallentuna	Tärnan	Surface water	ug/l	<0.016	<0.011	<0.01	<0.012	<0.024	<0.073	9.4
8713	Nat	Backgr	Katrineholm	Älgsjön	Sediment	ng/g DW	<10	<5	<5	<5	<10	<35	
8714	Nat	Backgr	Vallentuna	Tärnan	Sediment	ng/g DW	<10	<5	<5	<5	<10	<35	
7743	Nat	Backgr		Deep sea sed., Landsortsdjupet	Sediment	ng/g DW	<10	25	<5	<5	20	45	
7737	Nat	Backgr		Deep sea sed., Härnösandsdjupet	Sediment	ng/g DW	<10	8.8	<5	<5	13	22	
8869	Nat	Diffuse	Stockholm	Årstaviken	Surface water	ug/l	<0.011	<0.008	0.013	<0.008	<0.02	0.013	
7960	Nat	Diffuse	Stockholm	Årstaviken	Sediment	ng/g DW	<10	29	26	<5	37	92	
8870	Nat	Diffuse	Stockholm	Riddarfjärden	Surface water	ug/l	<0.011	0.01	<0.01	<0.008	0.031	0.041	
7968	Nat	Diffuse	Stockholm	Riddarfjärden	Sediment	ng/g DW	<10	63	58	<5	120	250	
7962	Nat	Diffuse	Stockholm	St Essingen	Sediment	ng/g DW	11	430	180	6.4	270	900	
8955	Nat	Diffuse	Stockholm	Henriksdal WWTP	Influent	ug/l	0.35	1	7.7	0.42	12	22	0.37
8958	Nat	Diffuse	Stockholm	Henriksdal WWTP	Effluent	ug/l	<0.02	0.096	0.089	<0.02	0.25	0.43	0.56
8961	Nat	Diffuse	Stockholm	Henriksdal WWTP	Sludge	ng/g DW	1100	4700	62000	3200	51000	120000	200
8874	Nat	Diffuse	Stockholm	Blockhusudden	Surface water	ug/l	<0.011	0.013	0.014	<0.008	<0.02	0.027	
7964	Nat	Diffuse	Stockholm	(Gr1) Valdemarsudde	Sediment	ng/g DW	33	4400	1200	26	1600	7200	
7966	Nat	Diffuse	Stockholm	(Gr2) Biskopsudden	Sediment	ng/g DW	<10	31	24	<5	15	69	
7986	Nat	Diffuse	Stockholm	(Gr3) Torsbyfjärden	Sediment	ng/g DW	<10	68	22	<10	56	150	
8964	Nat	Diffuse	Uppsala	Kungsängsverket WWTP	Influent	ug/l	0.29	0.91	7.2	0.46	11	20	2.6
8967	Nat	Diffuse	Uppsala	Kungsängsverket WWTP	Effluent	ug/l	<0.02	0.25	0.096	0.021	0.34	0.71	1.1
8970	Nat	Diffuse	Uppsala	Kungsängsverket WWTP	Sludge	ng/g DW	430	3000	35000	2200	15000	56000	87
8939	Nat	Diffuse	Uppsala	upstream Kungsängsv. WWTP, Islandsfallet, -1.7 km	Surface water	ug/l	<0.011	<0.008	0.014	<0.008	0.022	0.037	0.029
8940	Nat	Diffuse	Uppsala	downstream Kungsängsv. WWTP 5 m	Surface water	ug/l	<0.011	0.11	0.033	<0.008	0.29	0.44	0.76
8941	Nat	Diffuse	Uppsala	downstream Kungsängsv. WWTP 150 m	Surface water	ug/l	<0.011	0.036	0.018	<0.008	0.11	0.16	0.19
8942	Nat	Diffuse	Uppsala	downstream Kungsängsv. WWTP, Ultuna, 3.5 km	Surface water	ug/l	<0.011	0.022	0.016	<0.008	0.08	0.12	0.11
8943	Nat	Diffuse	Uppsala	downstream Kungsängsv. WWTP, Flottsund, 4.6 km	Surface water	ug/l	<0.011	0.018	0.013	<0.008	0.065	0.096	0.087
8888	Nat	Diffuse	Umeå	Öhn WWTP	Influent	ug/l	0.47	1	10	0.65	11	24	1.6
8889	Nat	Diffuse	Umeå	Öhn WWTP	Effluent	ug/l	0.023	0.17	0.39	0.048	1.1	1.7	1.1
8890	Nat	Diffuse	Umeå	Öhn WWTP	Sludge	ng/g DW	940	2800	66000	3200	46000	120000	120
8882	Nat	Diffuse	Borås	Gässlösa WWTP	Effluent	ug/l	0.051	0.21	0.6	0.046	1.7	2.6	
8883	Nat	Diffuse	Borås	Gässlösa WWTP	Sludge	ng/g DW	3100	8200	53000	2300	41000	110000	
8886	Nat	Diffuse	Alingsås	Nolhaga WWTP	Effluent	ug/l	<0.02	0.18	0.91	0.054	1.6	2.7	
8887	Nat	Diffuse	Alingsås	Nolhaga WWTP	Sludge	ng/g DW	940	4500	55000	2600	19000	83000	
8884	Nat	Diffuse	Göteborg	Ryaverken WWTP	Effluent	ug/l	0.067	0.49	0.88	0.11	3.6	5.1	
8885	Nat	Diffuse	Göteborg	Ryaverken WWTP	Sludge	ng/g DW	2300	11000	83000	7700	57000	160000	
8929	Nat	Diffuse	Skövde	Skövde WWTP	Effluent	ug/l	<0.02	0.059	0.099	0.018	0.43	0.6	0.47

Sample #	Nat/ County	Type	Municipality	Site	Matrix	Unit	DSBP	DAS1	FB28	FB85	DAS2	Sum	Karbamazepin
8938	Nat	Diffuse	Skövde	Skövde WWTP	Sludge	ng/g DW	1800	4900	48000	2900	25000	83000	190
781	Nat	Point source		Industry 1	Effluent	ug/l	<0.8	<0.7	<1.2	<0.6	<4	<0.8	
787	Nat	Point source		Industry 2	Effluent	ug/l	<0.8	<0.7	<1.2	<0.6	<4	<0.8	
824	Nat	Point source		Industry 3	Effluent	ug/l	<0.8	<0.7	<1.2	<0.6	<4	<0.8	
825	Nat	Point source		Industry 4, influent to internal treatment	Influent	ug/l	<0.8	<0.7	6.8	<0.6	13	<0.8	
826	Nat	Point source		Industry 4	Effluent	ug/l	<0.8	<0.7	<1.2	<0.6	1.7	<0.8	
828	Nat	Point source		Industry 5, influent to internal treatment	Influent	ug/l	<0.8	<0.7	<1.2	<0.6	<4	<0.8	
829	Nat	Point source		Industry 5	Effluent	ug/l	<0.8	<0.7	<1.2	<0.6	<4	<0.8	
830	Nat	Point source		Industry 6	Effluent	ug/l	<0.8	<0.7	<1.2	<0.6	39	<0.8	
831	Nat	Point source		Industry 7	Effluent	ug/l	<0.8	<0.7	<1.2	<0.6	<4	<0.8	
TP1	Nat	Diffuse		TP1, see table 13	Toilet paper	µg/g	0.18	0.031	120	16	470	610	
TP2	Nat	Diffuse		TP2, see table 13	Toilet paper	µg/g	<0.01	<0.01	0.96	0.082	1.4	2.4	
TP3	Nat	Diffuse		TP3, see table 13	Toilet paper	µg/g	0.23	<0.01	36	25	310	370	
8810	Värmland	Diffuse	Karlstad	Sjöstadsverket WWTP	Effluent	ug/l	0.045	0.21	0.26	0.033	1.5	2	
8811	Värmland	Diffuse	Karlstad	Sjöstadsverket WWTP	Sludge	ng/g DW	2100	4400	80000	3100	62000	150000	
8790	Värmland	Diffuse	Kristinehamn	Fiskartorpet WWTP	Effluent	ug/l	<0.02	0.079	0.11	0.018	1.5	1.7	
8791	Värmland	Diffuse	Kristinehamn	Fiskartorpet WWTP	Sludge	ng/g DW	490	3200	42000	1900	22000	70000	