Norwegian Screening 2016

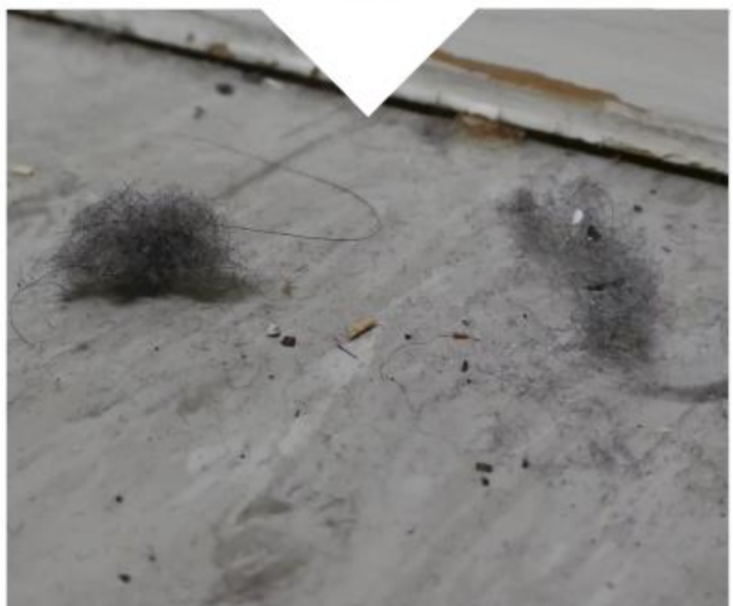
Martin Schlabach, NILU

ENVIRONMENTAL MONITORING

M-818 | 2017

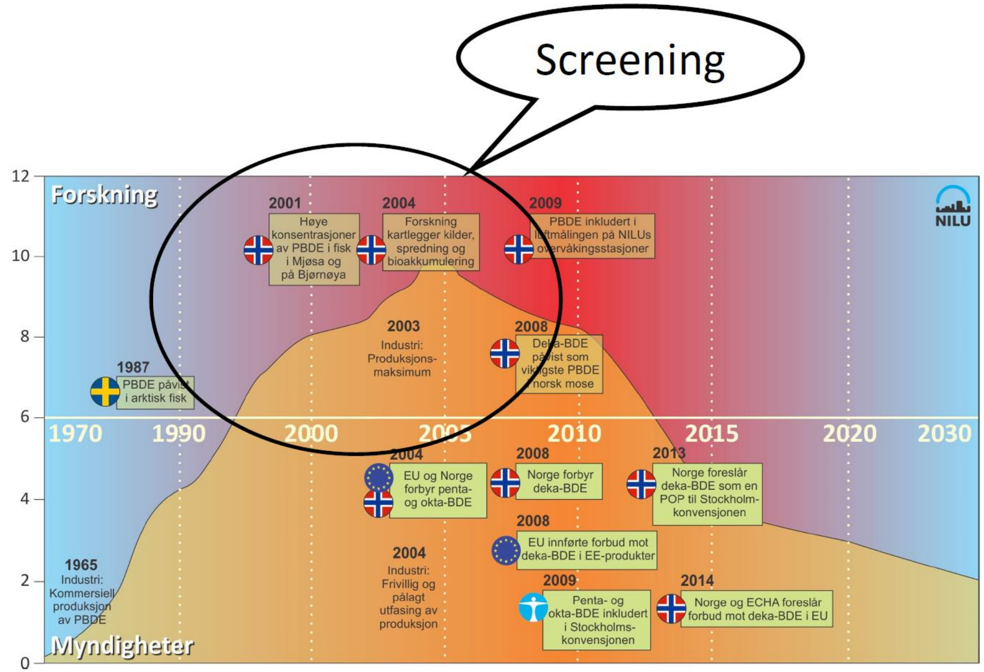
Screening programme 2016

Selected compounds with relevance for EU regulation



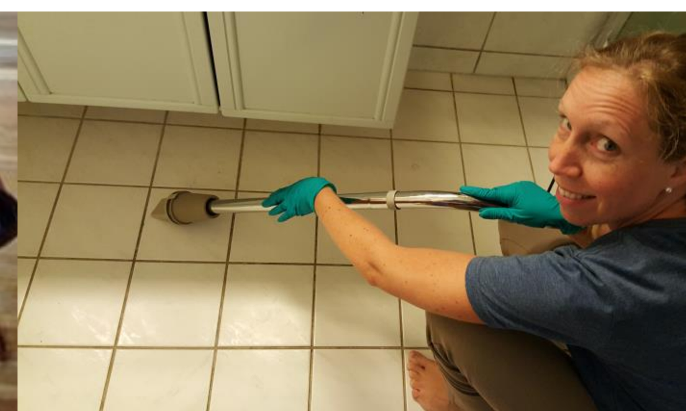
Project background

- Selected Compounds:
  - Potentially harmful to environment
  - Relevant for EU regulation
  - Earlier measurements
  - Tonnage used
- Sample types:
  - WWTP effluents and landfill leachate,
  - Recipient and biota
  - Indoor air and dust
- Motivation:
  - Relevant emissions in Norway???
  - Bioaccumulation???
  - Environmental risk???



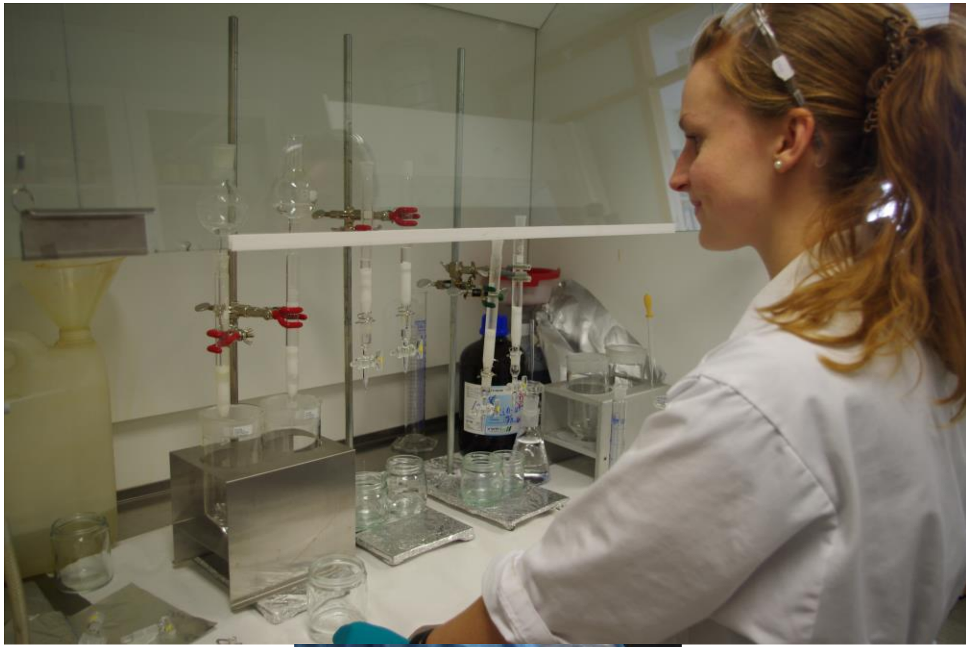
Sampling

Matriks	Lokaliteter	Prøvetype	Tilleggs-informasjon	Antall prøver
Utslipp fra renseanlegg	Oslo, Vestfjorden Avløpsselskap (VEAS), med avansert rensing (PE 700 000)	5 x 24 timers strøm-proporsjonal blandprøve	Vannføring	10
	Hamar (HIAS), med avansert rensing (PE 55 000)			
Innløpsvann til renseanlegg	Oslo, Vestfjorden Avløpsselskap (VEAS), med avansert rensing (PE 700 000)	5 x 24 timers strøm-proporsjonal blandprøve	Vannføring	10
	Hamar (HIAS), med avansert rensing (PE 55 000)			
Slam	Vestfjorden Avløpsselskap (VEAS), med avansert rensing (PE 700 000)	5 blandprøver	Vekt slam/dag	10
	Hamar (HIAS), med avansert rensing (PE 55 000)			
Sigevann	Lindum eller Yggestad	5 prøver	Vannføring	5
Overflatevann	Mjøsa	5 prøver		5
Sediment	Mjøsa	5 prøver		5
Ørret	Mjøsa	10 individer	Vekt, lengde, modningsgrad og kjønn	10
Rotter	Oslo/Akershus	Samleprøver av minst 3 individer		5
Innemiljø Luft	Oslo/Akershus		Spørreskjema	10
Innemiljø Støv	Oslo/Akershus		Spørreskjema	10



Sample clean-up

- Reducing risk of sample contamination
- No nitril-cloves during sampling and cleanup
- Restricted use of personal care products before sampling and cleanup
- Special routines for siloxanes



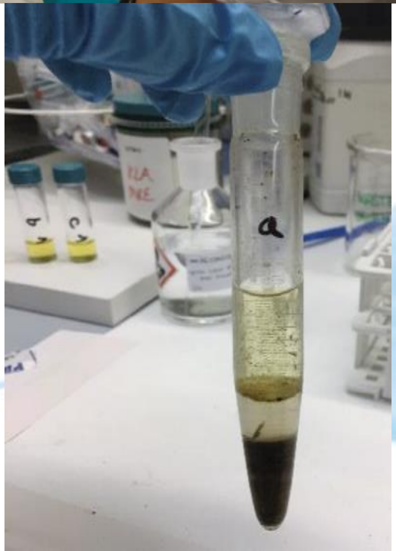


Table 1: Volatiles
Name, Acronym, CAS, Function, and Log Kow

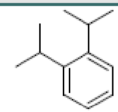
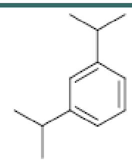
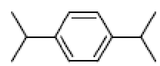
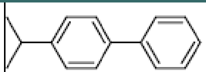
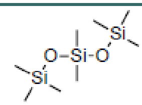
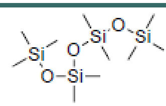
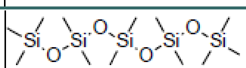
Name	Acronym	Structure	CAS	Function	Log Kow
1,2-Diisopropylbenzene			577-55-9	Solvent	4,9
1,3-Diisopropylbenzene			99-62-7	Solvent	4,9
1,4-Diisopropylbenzene			100-18-5	Solvent	4,9
4-Isopropyl-1,1'-biphenyl			25640-78-2	Solvent	5,14
Octamethyltri-siloxane	L3		107-51-7	Solvent/ Impurity	5,7
Decamethyltetra-siloxane	L4		141-62-8	Solvent/ Impurity	7,1
Dodecamethylpenta-siloxane	L5		141-63-9	Solvent/ Impurity	7,4

Table 2: Dechloranes

Name, Acronym, CAS, Function, and Log K_{ow}

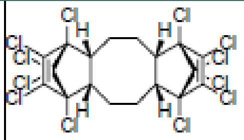
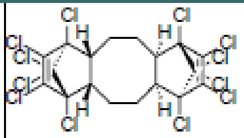
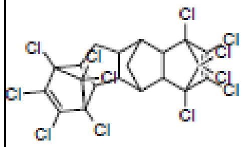
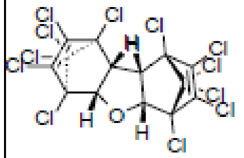
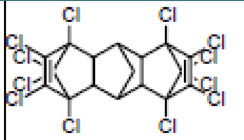
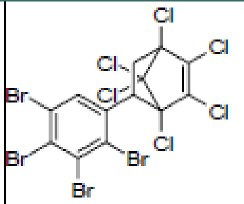
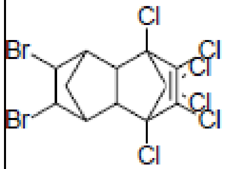
Name	Acronym	Structure	CAS	Function	Log K_{ow}
Dechlorane plus syn	synDP		135821-03-3	Flame retardant	8,85
Dechlorane plus anti	antiDP		135821-74-8	Flame retardant	8,85
Dechlorane 601	Dec601		13560-90-2	Flame retardant	9,22
Dechlorane 602	Dec602		31107-44-5	Flame retardant	7,37
Dechlorane 603	Dec603		13560-92-4	Flame retardant	8,24
Dechlorane 604	Dec604		34571-16-9	Flame retardant	8,84
Dibromoaldrin	DBALD		20389-65-5	Flame retardant	5,77

Table 3: Phenoles, bisphenoles and phenolic antioxidants

Name, Acronym, CAS, Function, and Log K_{ow}

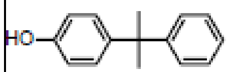
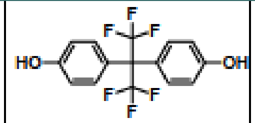
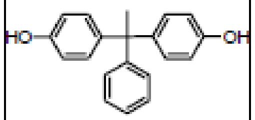
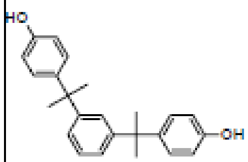
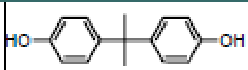
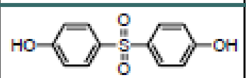
Name	Acronym	Structure	CAS	Function	Log K_{ow}
4-Cumylphenol or Hydroxydiphenyl-propane	HPP		599-64-4	Metabolite of detergents	3,88
Bisphenol AF	BPAF		1478-61-1	Monomer	4,52
Bisphenol AP	BPAP		1571-75-1	Monomer	3,99
Bisphenol M	BPM		13595-25-0	Monomer	5,49
Bisphenol A	BPA		80-05-7	Monomer	3,24
Bisphenol F	BPF		620-92-8	Monomer	2,57
Bisphenol S	BPS		80-09-1	Monomer	8,93

Table 3: Phenoles, bisphenoles and phenolic antioxidants

Name, Acronym, CAS, Function, and Log K_{ow}

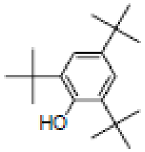
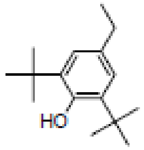
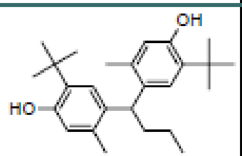
Name	Acronym	Structure	CAS	Function	Log K_{ow}
2,4,6-Tris(tert-butyl)phenol	TTBP or AO246		732-26-3	Antioxidant	5,93
2,6-Di-tert-butyl-4-ethylphenol	DBEP or AO		4130-42-1	Antioxidant	5,24
4,4'-Butane-1,1-diylbis(2-tert-butyl-5-methylphenol	BBM or AO44B25		85-60-9	Antioxidant	7,43

Table 4: BADGE and BFDGE

Name, Acronym, CAS, Function, and Log K_{ow}

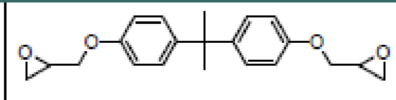
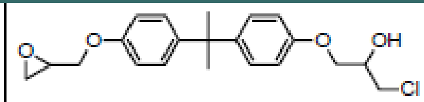
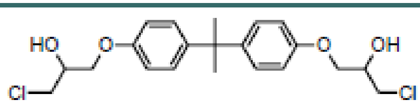
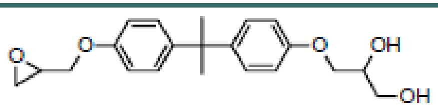
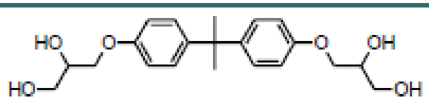
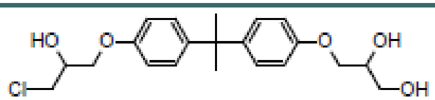
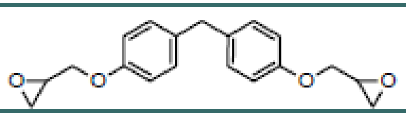
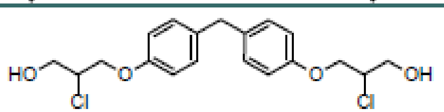
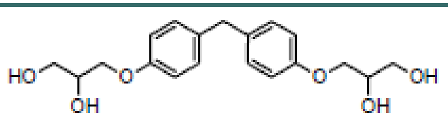
Name	Structure	CAS	Log K_{ow}
BADGE		1675-54-3	3,43
BADGE-HCl		13836-48-1	3,66
BADGE-2HCl		4809-35-2	4,01
BADGE-H_2O		76002-91-0	2,96
BADGE-2H_2O		5581-32-8	1,86
BADGE-HCl-H_2O		227947-06-0	2,24
BFDGE		2095-03-6	2,49
BFDGE-2HCl		374772-79-9	2,97
BFDGE-2H_2O		72406-26-9	1,47

Table 5: Pesticides, POPs, PFAS, phosphor based antioxidants and phosphor flame-retardants

Name, CAS, Function, and Log Kow

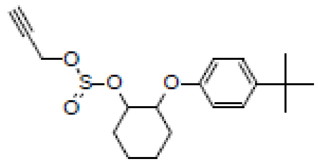
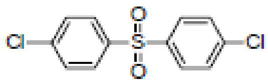
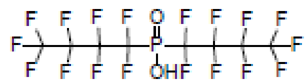
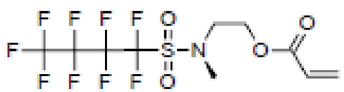
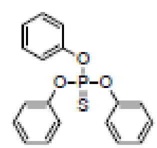
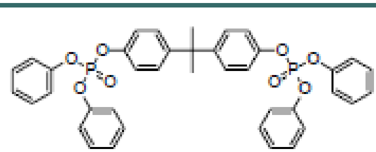
Name	Structure	CAS	Function	Log K_{ow}
Propargite		2312-35-8	Pesticide	5,0
Bis(4-chlorophenyl) sulfone (BCPS)		80-07-9	Monomer	3,9
Phosphinic acid, bis(nonafluorobutyl) (PFPiA)		52299-25-9	Intermediate Short chain PFAS	5,3
2-[methyl(1,1,2,2,3,3,4,4,4-nonafluorobutylsulfonyl)amino]ethyl prop-2-enoate (NFacrylat)		67584-55-8	Coating, repellent, dye, monomer	3,2
O,O,O-Triphenyl phosphorothioate (TPPT)		597-82-0	Lubricant, corrosion inhibitor, antioxidant	6,3
Bisphenol A bis(bisphenyl-phosphate) (BPA-BDPP)		5945-33-5	Flame retardant	10,8

Table 6: Synthetic musk, plasticizers, organo metals and UV compounds

Name, CAS, Function, and Log Kow

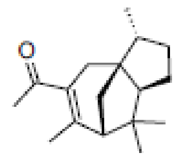
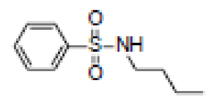
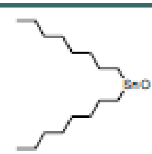
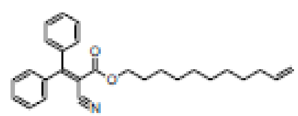
Name	Structure	CAS	Function	Log K_{ow}
[3R-(3α,3aβ,7β,8aα)]-1-(2,3,4,7,8,8a-hexahydro-3,6,8,8-tetramethyl-1H-3a,7-methanoazulen-5-yl)ethan-1-one (Methyl Cedryl ketone)		32388-55-9	Musk	5,02
n-Butylbenzene sulphonamide (NBBSulfone)		3622-84-2	Plasticiser	2,1
Di-n-octyltin oxide		870-08-6	Stabilizer	-
10-undecenyl 2-cyano-3,3-diphenylpropenoate		947701-81-6	UV-filter	4,0

Table 9: Detection frequency for all studied compounds

Compound	Emission					Recipient			
	WWTP Effluent	Leachate water	Sludge	House dust	Indoor air	Surface watera.	Sediment	Rat	Brown trout
L3	na	na	4	4	4	na	na	0	0
L4	na	4	4	4	4	na	na	0	0
L5	na	4	4	4	4	na	na	0	3
Di-isopropylbenzenes	na	0	3	1	4	0	na	0	1
4-Isopropyl-1,1'-biphenyl	na	0	3	4	0	0	na	0	0
Dibromoaldrin	0	0	0	0	na	na	0	0	0
Dec 601	0	0	0	0	na	na	0	0	0
Dec 602	0	4	0	0	na	na	0	0	4
Dec 603	0	4	0	1	na	na	0	0	0
Dec 604	0	0	3	3	na	na	0	0	0
DP syn	0	4	4	3	na	na	0	4	0
DP anti	1	4	4	2	na	na	0	3	0
PCB-153	0	4	4	4	na	na	3	4	4
HPP	0	0	3	0	0	0	0	0	0
TTBP	1	0	0	0	0	0	0	0	0
BBM	1	0	2	0	0	0	0	0	0
DTEB	0	0	0	0	0	0	0	0	0
BPS	4	4	3	4	na	0	0	0	0
BPF	1	3	3	4	na	0	0	0	0
BPA	2	4	4	4	na	3	0	0	0
BPAF	0	0	0	1	na	0	0	0	0
BPAP	0	0	0	0	na	0	0	0	0
BPM	0	0	2	0	na	0	0	0	0

na): not Analysed or not possible to Analyse
0): not detected (0 %)
1): rarely detected (< 10 %)
2): detected from time to time (10 – 49 %)
3): detected frequently (50 – 89 %)
4): detected in all samples (> 90%)

Table 9: Detection frequency for all studied compounds

Compound	Emission					Recipient			
	WWTP Effluent	Leachate water	Sludge	House dust	Indoor air	Surface watera.	Sediment	Rat	Brown trout
BADGE	na	na	na	na	na	na	na	na	na
BADGE-HCL	0	0	1	3	na	0	1	0	0
BADGE-2HCL	0	0	0	3	na	0	0	0	0
BADGE-H20	0	0	1	2	na	0	0	0	0
BADGE-2H20	0	1	0	3	na	0	1	0	0
BADGE-HCL-H2O	0	0	0	4	na	0	3	0	0
BFDGE	0	0	0	1	na	0	0	0	0
BFDGE-2HCL	0	0	0	1	na	0	0	0	0
BFDGE-2H20	0	0	0	0	na	0	0	0	0
Propargite	0	0	0	0	0	0	0	0	1
NBBSulfone	0	4	0	0	0	0	0	0	0
OTP	0	0	3	0	0	0	0	0	0
BPA-BDPP	0	0	0	2	0	0	0	0	0
TPPT	0	0	0	0	na	0	0	0	na
NFacrylat	0	0	0	0	na	0	0	0	na
Di-n-octyltin	0	0	0	4	na	0	0	0	0
BCPS	0	0	0	0	0	0	0	0	0
Methyl-cedryl-ketone	4	0	4	3	3	0	2	0	0
Undecenyl crylene	0	0	0	1	na	0	0	0	0
Octocrylene	4	4	4	4	na	4	4	na	0

na): not Analysed or not possible to Analyse
0): not detected (0 %)
1): rarely detected (< 10 %)
2): detected from time to time (10 – 49 %)
3): detected frequently (50 – 89 %)
4): detected in all samples (> 90%)

Table 10: Volatile concentrations in sludge samples from VEAS and HIAS WWTP						
	L3	L4	L5	1,3-di-isopropyl-benzene	1,4-di-isopropyl-benzene	4-Isopropyl-1,1'-biphenyl
Sample type	(Min - max) Average* Detection frequency			ng/g d.w.		
VEAS Sludge	(2,2 - 3,1) 2,5 100 %	(16 - 18) 15 100 %	(366 - 405) 377 100 %	(1,7 - 2,0) 1,8 100 %	(<0,2 - 2,5) 1,2 60 %	(1,6 - 2,2) 1,9 100 %
HIAS Sludge	(5,8 - 7,3) 6,5 100 %	(30 - 33) 32 100 %	(350 - 402) 383 100 %	(<2,0 - 2,1) 1,7 60 %	(1,1 - 2,3) 1,7 80 %	(<0,13 - 0,39) 0,25 60 %

*): For the non-detects LoD/2 was used, when calculating the average.

- Linear siloxanes in emission samples and indoor air
- Probably lower exposure via indoor air compared to application of personal care products
- ECHA estimates similar consumption of L3 og L4, and lower for L5. Our findings do not support ECHA's estimate. (Possible explanation: insufficient consumption data or unidentified sources)

Table 11: Volatile concentrations in dust and indoor air samples from the Oslo area						
	L3	L4	L5	1,3-di-isopropyl-benzene	1,4-di-isopropyl-benzene	4-Isopropyl-1,1'-biphenyl
Sample type	(Min - max) Average* Detection frequency			ng/g and ng/m^3		
House dust	(0,23 - 1,3) 0,46 100 %	(<0,2 - 1,6) 0,64 89 %	(<10 - 464) 98 55 %	(<0,5 - 9,2) 1,3 22 %	(<0,6 - 8,0) 1,3 11 %	(0,25 - 15) 2,3 100 %
Indoor air	(1,6 - 743) 88 100 %	(1,1 - 37) 14 100 %	(5,6 - 1460) 195 100 %	(0,45 - 4,7) 2,1 100 %	(0,51 - 3,6) 1,8 100 %	(<0,2 - <1,1) 0,22 100 %

*): For the non-detects LoD/2 was used, when calculating the average.

Di-isopropylbenzenes and 4-Isopropyl-1,1'-biphenyl are frequently found in sewage sludge and indoor samples, giving indications for frequent use of these compounds.

Table 12: Dechlorane concentrations in influent, effluent, and sludge samples from VEAS and HIAS WWTP

Sample type	Dec 602	Dec 603	Dec 604	DPsyn	DPanti	PCB-153
	(Min - max) Average* Detection frequency			ng/L or ng/g d.w.		
VEAS Influent	(<0,1 - <0,1) **<0,05** 0 %	(<0,1 - <0,1) **<0,05** 0 %	(<0,2 - <0,2) **<0,1** 0 %	(<0,1 - 0,73) **0,24** 60 %	(<0,4 - 4,09) **1,59** 60 %	(<0,2 - 0,58) **0,28** 40 %
VEAS Effluent	(<0,1 - <0,1) **<0,05** 0 %	(<0,1 - <0,1) **<0,05** 0 %	(<0,2 - <0,2) **<0,1** 0 %	(<0,1 - 0,16) **0,07** 20 %	(<0,4 - 0,53) **0,32** 40 %	(<0,2 - <0,2) **<0,1** 0 %
VEAS Sludge	(<0,1 - <0,1) **<0,05** 0 %	(<0,1 - <0,1) **<0,05** 0 %	(0,88 - 1,69) **1,4** 100 %	(0,80 - 0,96) **0,91** 100 %	(2,22 - 3,55) **3,0** 100 %	(2,23 - 18,4) **5,7** 100 %
HIAS Influent	(<0,1 - <0,1) **<0,05** 0 %	(<0,1 - <0,1) **<0,05** 0 %	(<0,2 - <0,2) **<0,1** 0 %	(<0,1 - 1,1) **0,79** 80 %	(<0,4 - 4,8) **2,7** 80 %	(<0,1 - 1,1) **0,62** 80 %
HIAS Effluent	(<0,1 - <0,1) **<0,05** 0 %	(<0,1 - <0,1) **<0,05** 0 %	(<0,2 - <0,2) **<0,1** 0 %	(<0,1 - <0,1) **<0,05** 0 %	(<0,4 - <0,4) **<0,2** 0 %	(<0,2 - <0,2) **<0,1** 0 %
HIAS Sludge	(<0,1 - <0,1) **<0,05** 0 %	(<0,1 - <0,1) **<0,05** 0 %	(<0,05 - 1,3) **0,71** 60 %	(0,89 - 1,6) **1,1** 100 %	(2,3 - 6,9) **3,6** 100 %	(0,97 - 3,7) **2,4** 100 %
Leachate Lindum	(2,6 - 5,2) **3,8** 100 %	(0,05 - 0,06) **0,05** 100 %	(<0,2 - <0,2) **<0,1** 0 %	(101 - 127) **113** 100 %	(198 - 290) **262** 100 %	(137 - 218) **175** 100 %

*): For the non-detects LoD/2 was used, when calculating the average.

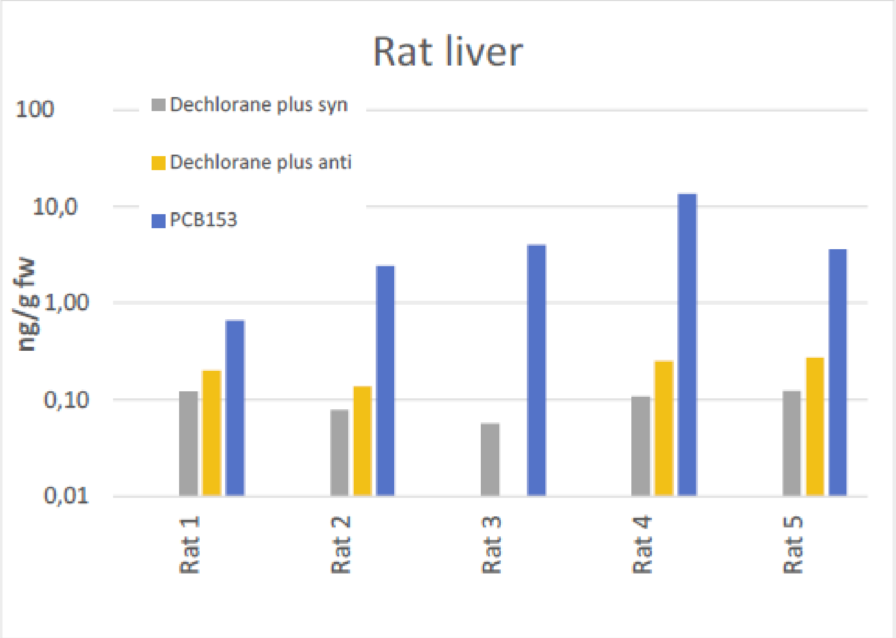


Figure 5: Concentration of DPsyn, DPanti, and PCB-153 in rat liver samples. Concentration given in ng/g, linear scale top figure, logarithmic scale bottom figure. House dust samples.

Table 13: Dechlorane concentrations in house dust samples

Sample type	Dec 603	Dec 604	DPsyn	DPanti	PCB-153
	(Min - max) Average* Detection frequency			ng/g	
House dust	(<0,05 - 0,06) **0,03** 11 %	(<0,1 - 4,6) **1,3** 78 %	(<1 - 77) **12** 78 %	(<1 - 56) **14** 78 %	(0,06 - 10) **1,5** 100 %

*): For the non-detects LoD/2 was used, when calculating the average.

Most dechloranes found in in all sample types including house.

Frequently findings in biota indicates persistency and bioaccumulation, which require indepth follow-up..

Table 15: Concentration of biphenols in influent, effluent, and sludge samples from VEAS and HIAS WWTP, leachate from Lindum landfill

Sample	BPS	BPF	BPA	BPM
	(Min - max) Average* Detection frequency		ng/L or ng/g d.w.	
VEAS Influent	(370 - 496) 440 100 %	(<90 - 111) 69 40 %	(<450**) - 536) 318 40 %	(<2 - 17) 6 20 %
VEAS Effluent	(221 - 354) 271 100 %	(<15 - <15) 8 0 %	(<30 - <30) 15 0 %	(<5 - <5) 2,5 0 %
VEAS Sludge	(3 - 10) 6 100 %	(<20 - <20) 10 0 %	(79 - 157) 131 100 %	(<0,5 - 193) 40 80 %
HIAS Influent	(481 - 602) 543 100 %	(<90 - 120) 89 80 %	(1 480 - 4 080) 2 454 100 %	(<2 - 4) 2 20 %
HIAS Effluent	(221 - 354) 271 100 %	(<15 - <15) 8 0 %	(<30 - <30) 15 100 %	(<5 - <5) 2,5 0 %
HIAS Sludge	(<2 - 41) 31 80 %	(26 - 68) 42 100 %	(1 500 - 1 610) 1 563 100 %	(<0,5 - <0,5) 0,3 0 %
Leachate Lindum	(3 600 - 6 320) 4 608 100 %	(<200 - 395) 265 60 %	(82 600 - 317 000) 147 400 100 %	(<5 - <5) 3 0 %

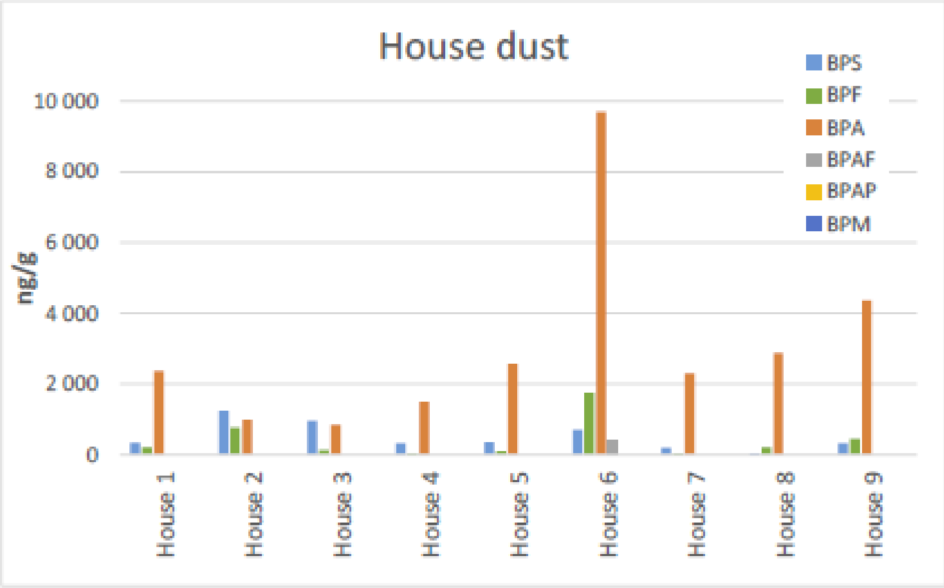
*): For the non-detects LoD/2 was used, when calculating the average.

**): As the analytical method was adjusted for best sensitivity of the main target compounds BPAP and BPM, it was not possible to achieve optimal sensitivity for BPA for this study.

Table 16: Concentration of bisphenols in house dust samples from Oslo area

Sample	BPS	BPF	BPA	BPAF
	(Min - max) Average* Detection frequency		ng/g	
House dust	(23 - 1 230) 488 100 %	(22 - 1 740) 401 100 %	(839 - 9 690) 3 050 100 %	(<10 - 490) 50 0 %

*): For the non-detects LoD/2 was used, when calculating the average.



- BPAF og BPM only found in some emission and dust samples .
- BPAP not found at all.
- The reference compounds BPA, BPS, og BPF found in nearly all emissions samples and qualify for routine monitroing.
- Intake of bisphenols by ingestion of house dust can be as high as via food.

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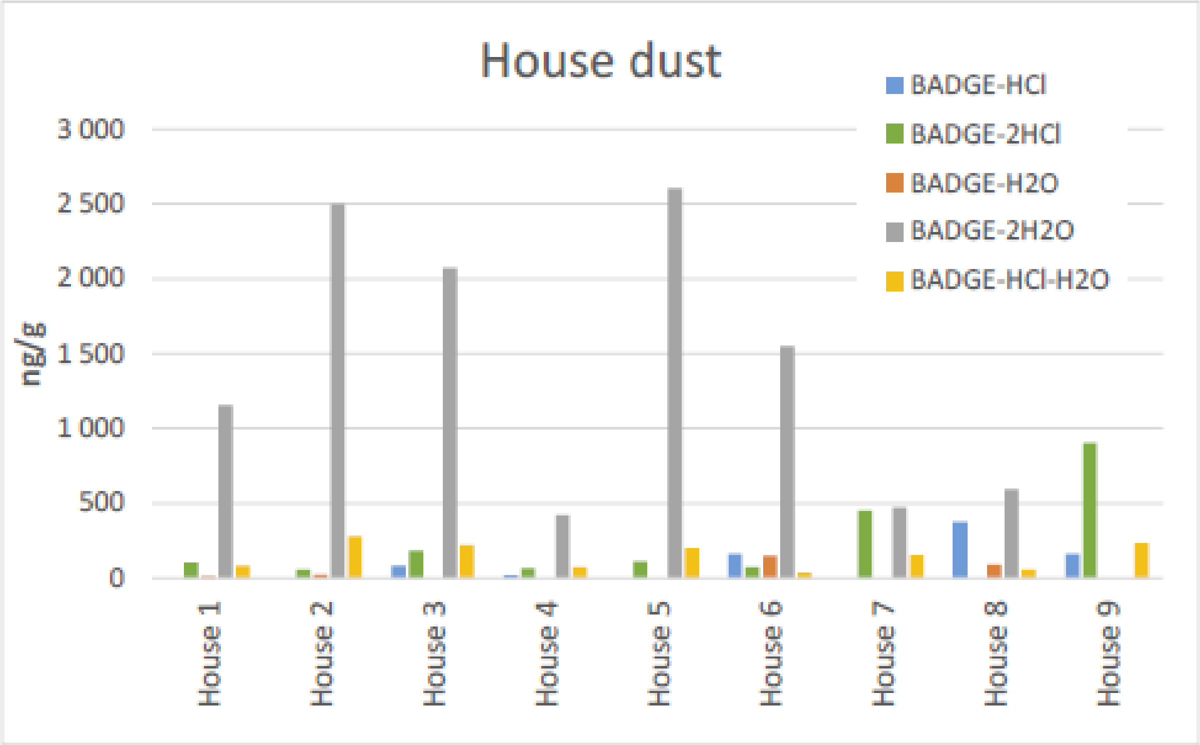
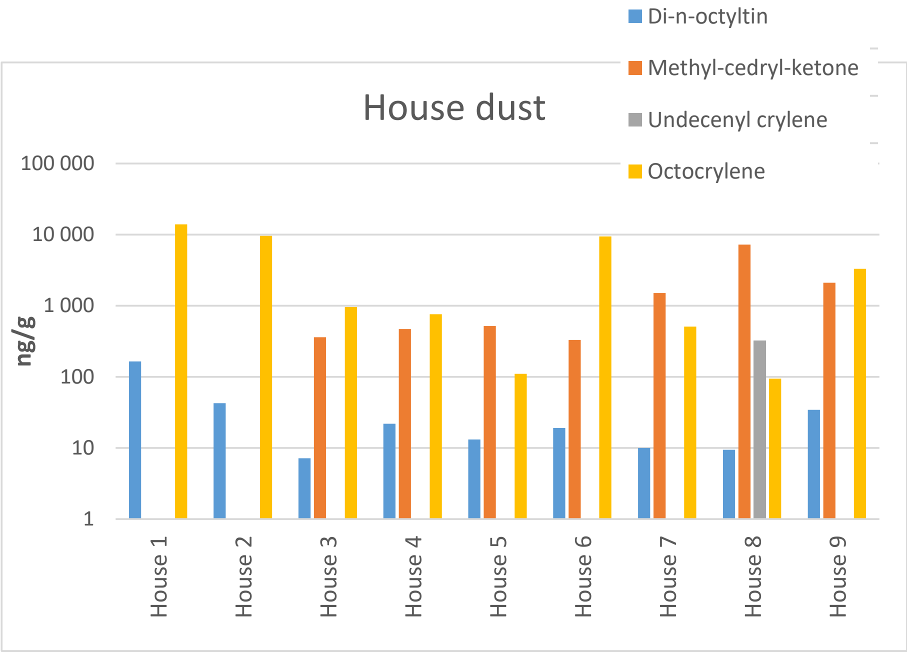


BADGE-relaterted compounds found in house dust only.

Some BADGE compounds were found in all house dust samples.

The measured concentrations were in the same order of magnitude as BPA.



Di-n-octyltin probably from water-borne wall paint was found in all house dust samples.

The fragrant methyl cedryl ketone was in relatively high concentrations in all WWTP influent, effluent, and sludge samples ble funnet i relativt høye konsentrasjoner.
The calculated daily emissions from VEAS and HIAS were 70 and 15 g, respectively.
This compound were also found in some sediment samples from Lake Mjøsa and in nearly all house dust and indoor air samples.

The UV filter octocrylene were found in alle emission samples and in sediments and surface water taken close to these emission sources.
This compound were also found in dust samples.

Conclusions

The yellow marked compounds are either found frequently in two or more sample types or in all samples of one type and thus qualify for additional in-depth studies.

Table 9: Detection frequency for all studied compounds

Compound	Emission					Recipient			
	WWTP Effluent	Leachate water	Sludge	House dust	Indoor air	Surface water a.	Sediment	Rat	Brown trout
L3	na	na	4	4	4	na	na	0	0
L4	na	4	4	4	4	na	na	0	0
L5	na	4	4	4	4	na	na	0	3
Di-isopropylbenzenes	na	0	3	1	4	0	na	0	1
4-Isopropyl-1,1'-biphenyl	na	0	3	4	0	0	na	0	0
Dibromoaldrin	0	0	0	0	na	na	0	0	0
Dec 601	0	0	0	0	na	na	0	0	0
Dec 602	0	4	0	0	na	na	0	0	4
Dec 603	0	4	0	1	na	na	0	0	0
Dec 604	0	0	3	3	na	na	0	0	0
DP syn	0	4	4	3	na	na	0	4	0
DP anti	1	4	4	2	na	na	0	3	0
PCB-153	0	4	4	4	na	na	3	4	4
HPP	0	0	3	0	0	0	0	0	0
TTBP	1	0	0	0	0	0	0	0	0
BBM	1	0	2	0	0	0	0	0	0
DTEB	0	0	0	0	0	0	0	0	0
BPS	4	4	3	4	na	0	0	0	0
BPF	1	3	3	4	na	0	0	0	0
BPA	2	4	4	4	na	3	0	0	0
BPAF	0	0	0	1	na	0	0	0	0
BPAP	0	0	0	0	na	0	0	0	0
BPM	0	0	2	0	na	0	0	0	0

Table 9: Detection frequency for all studied compounds

Compound	Emission					Recipient			
	WWTP Effluent	Leachate water	Sludge	House dust	Indoor air	Surface water a.	Sediment	Rat	Brown trout
BADGE	na	na	na	na	na	na	na	na	na
BADGE-HCL	0	0	1	3	na	0	1	0	0
BADGE-2HCL	0	0	0	3	na	0	0	0	0
BADGE-H20	0	0	1	2	na	0	0	0	0
BADGE-2H20	0	1	0	3	na	0	1	0	0
BADGE-HCL-H2O	0	0	0	4	na	0	3	0	0
BFDGE	0	0	0	1	na	0	0	0	0
BFDGE-2HCL	0	0	0	1	na	0	0	0	0
BFDGE-2H20	0	0	0	0	na	0	0	0	0
Propargite	0	0	0	0	0	0	0	0	1
NBBSulfone	0	4	0	0	0	0	0	0	0
OTP	0	0	3	0	0	0	0	0	0
BPA-BDPP	0	0	0	2	0	0	0	0	0
TPPT	0	0	0	0	na	0	0	0	na
NFacrylat	0	0	0	0	na	0	0	0	na
Di-n-octyltin	0	0	0	4	na	0	0	0	0
BCPS	0	0	0	0	0	0	0	0	0
Methyl-cedryl-ketone	4	0	4	3	3	0	2	0	0
Undecenyl crylene	0	0	0	1	na	0	0	0	0
Octocrylene	4	4	4	4	na	4	4	na	0

Conclusions cont.

- For most of the studied compounds the information on environmental and health effects are very limited. A PNEC based classification of the environmental risk was possible for BPA, BPS, methyl cedryl ketone and octacrylene. For BPA some of the measured surface water concentrations were close to the PNEC and the environmental risk must therefore be classified as low to moderate.

- The sample type with most positive findings were house dust.
- Most of the studied compounds are part of products used in indoor environment or are easily transported into houses.
- House dust is a matrix which are comparable easy analysable matrix, where the compounds of interest are not «diluted» or hidden by a lot of interfering biological ballast.
- In contrast to many other sample types house dust is closely related to the original product.
- In addition, the composition of the dust is reflecting the composition of relatively new products. That means that new compounds of environmental concern will show much earlier in dust compared to leachate, sediments, or biota.

- We strongly suggest to apply indoor samples and especially dust as an early warning tool or watchdog for new compounds on the marked and to verify reported tonnages.

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