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Content

Preface

The aim of the Nordic environmental screening is to obtain a snapshot of the occurrence of potentially hazardous substances, both in regions most likely to be polluted as well as in some very pristine environments. The focus is on less known, anthropogenic substances and their derivatives, which are either used in high volumes or are likely to be persistent and hazardous to humans and other organisms. In this study the occurrence of selected plasticisers in environmental samples from the Nordic countries has been investigated. This was done to gain experience in environmental screening of this kind of substances and provide a better knowledge of plasticisers in the environment. If the substances subjected to screening are found in significant amounts this may result in further investigations or monitoring on national level and measures to reduce contamination.

Some of the samples were also analyzed for selected sweeteners (aspartam, cyclamate, sucralose). These reults are only briefly presented.

The Nordic screening project is run by a steering group with representatives from the Danish Centre for Environment and Energy, Aarhus University, Denmark, the Finnish Environment Institute, the Icelandic Food and Biotech R&D, the Environment Agency of the Faroe Islands, the Climate and Pollution agency in Norway and the Swedish Environmental Protection Agency.

The project is financed and supported by the Nordic Council of Ministers through the Nordic Chemicals Group and the Aquatic Ecosystems Group as well as the participating institutions. The chemical analyses have been carried out jointly by IVL Swedish Environmental Research Institute (plasticisers) and NILU Norwegian Institute for Air Research (sweeteners).

The respective participating Nordic countries organised sample selection, collection and transport of samples based on a sample protocol and manuals provided by the analytical laboratories.

Summary

The overall aim of this screening study was to investigate the occurrence of selected plasticisers in environmentally related samples from the Nordic countries. Eight phthalates, four adipates and one azelate were analysed for. Measurements were carried out in effluents and sludges from waste water treatment plants, sediments and fish. Usually the participating countries (Denmark, Finland, Faroe Islands, Island, Norway and Sweden) contributed three samples from each sample type. A smaller number of bird eggs were also included. The relatively few samples and sample types are intended to give a "snap shot" of the situation.

For all sample types the phthalates DEHP, DINP and DIDP were most frequently detected and found in the higest concentrations. DBP and BBP were also found frequently but in lower concentrations. The remaining phthalates L79P, DOP and DUP were found occasionally at low concentrations. This can also be said for the adipates DEHA and BOA, while DINA and DBEEQ was not found at all. The azelate DEHZ was found only once in sludge.

In sludge DINP, DEHP and DIDP taken together made up 96–99.7% of the summed concentration of all measured plasticisers. The highest summed concentration was 260,000 µg/kg dw. As opposed to effluents where DEHP nearly always showed the highest concentration, all sludges were dominated by DINP.

A general observation regarding sediments is that sites in direct vicinity of waste water treatment plants showed increased concentrations of plasticisers.

Concentrations in fish muscle were generally low and quite close to LOQ.

The plasticisers included in the screening may be harmful to the environment. The fact that the concentrations found in effluents and sediments were close to or exceeded PNEC in several cases indicate the need for further studies to assess potential risks. Furthermore, some of the substances were also found in biota.

The effluents and some of the sludges were also analyzed for sweeteners. The results showed that there is a widespread occurrence of the sweeteners cyclamate and sucralose in effluent from WWTPs in all the Nordic countries and that these substances are not profoundly accumulated in sludge.

1. Frame of the study

The occurrence and the environmental risk of chemicals are prioritized issues in several international legislative acts (e.g. the EUs Water framework directive, Registration Evaluation Authorisation of Chemicals (REACH), Stockholm Convention on Persistent Organic Pollutants (POPs) and the Convention on long-range trans-boundary air pollution–LRTAP) and there is a focus on "emerging" chemicals in different research and monitoring programs.

In this screening study the concentrations of the chemicals were determined in a variety of media, collected at different locations and representing different source characteristics, e.g. point sources and dispersed use related to many activities and products. Background samples were also taken. In addition to finding out current levels of the selected chemicals in different matrices the aim was to highlight important transport pathways and to identify current emissions. The results of this screening project may be used to estimate the environmental risk posed by these chemicals to vulnerable Nordic ecosystems.

However, there are limitations and uncertainties in screening studies; measurements of a chemical are carried out in several media at several sites but only a few samples at each site which gives only a "snap shot" of the situation.

The plasticisers included in the screening study are listed in Table 1.1. Abbreviations and CAS number are given for each compound.

The selection of compounds was based on a literature study of plasticisers by Lambert et al. (2010).

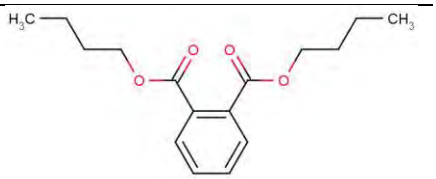
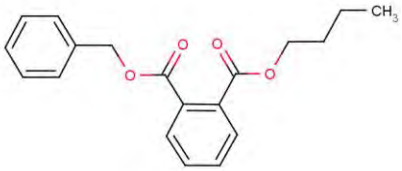
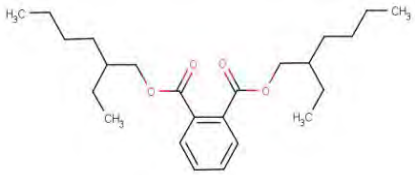
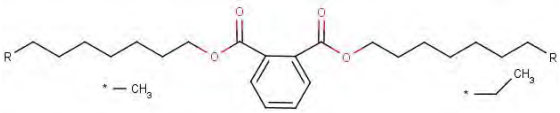
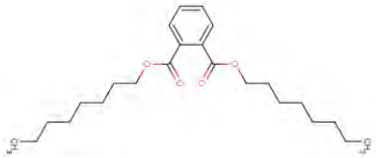
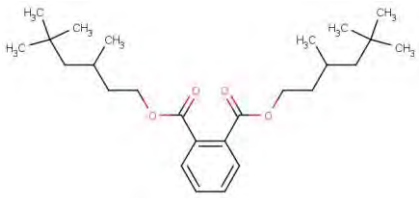
Table 1.1: Name, abbreviation and CAS-number for the plasticisers included in this study

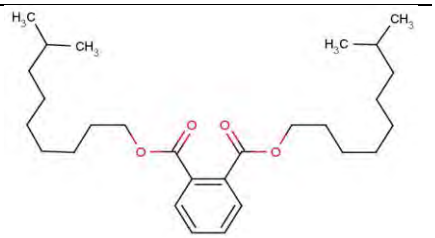
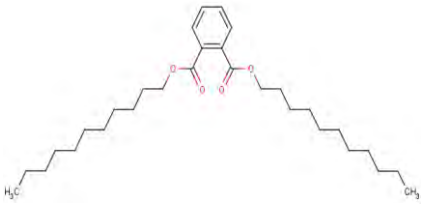
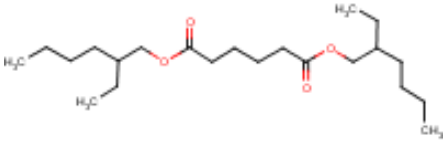
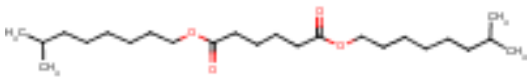
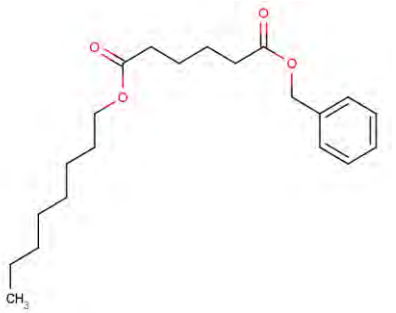
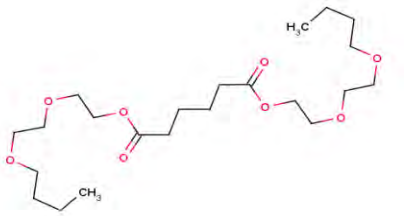
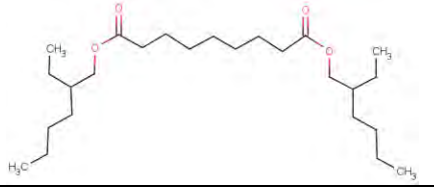
Compound name	Abbreviation	CAS #
Di butyl phthalate	DBP	84–74–2
Butyl benzyl phthalate	BBP	85–68–7
Di(2-ethylhexyl) phthalate (reference compound)	DEHP	117–81–7
Dialkyl(C7-C9) phthalate	L79P	68,515–41–3
Di-n-octyl phthalate	DOP	117–84–0
Diisononyl phthalate	DINP	28,553–12–0
Diisodecyl-phthalate	DIDP	26,761–40–0
Diundecyl phthalate	DUP	3,648–20–2
Di(2-ethylhexyl) adipate	DEHA	103–23–1
Diisononyl adipate	DINA	33,703–08–1
Benzyl octyl adipate	BOA	3,089–55–2
Di(2-(2-butoxyethoxy)ethyl) adipate	DBEEA	141–17–3
Di(2-etylhexyl) azelate	DEHZ	103–24–2

2. Background

Plasticisers are used to increase the plasticity of a material, especially plastics. The chemical structure of the plasticisers selected for this study are presented in Table 2.1.

Table 2.1: Structure of the substances selected for the study

Compound name CAS # Abbreviation	Structure
Dibutyl phthalate 84–74–2 DBP	
Butyl benzyl phthalate 85–68–7 BBP	
Di(2–ethylhexyl) phthalate 117–81–7 DEHP	
1,2-Benzenedicarboxylic acid, di-C7–9-branched and linear alkyl esters 68515–41–3 L79P	
Di-n-octyl phthalate 117–84–0 DOP	
Diisononyl phthalate 28553-12–0 DINP	

Compound name CAS # Abbreviation	Structure
Diisodecyl phthalate 26761–40–0 DIDP	
Diundecyl phthalate 3648–20–2 DUP	
Di(2-ethylhexyl) adipate 103–23–1 DEHA	
Diisononyl adipate 33703–08–1 DINA	
Benzyl octyl adipate 3089–55–2 BOA	
Di(2-(2-butoxyethoxy)ethyl) adipate 141–17–3 DBEEA	
Di(2-ethylhexyl) azelate 103–24–2 DEHZ	

All of the screened plasticisers are hydrophobic with log K_{ow} values larger than three and most of the substances have relatively low water solubility (Table 2.2, and more on water solubility below). They may thus be bioaccumulative, Lambert et al. (2010) did also conclude that all but DBEEA were bioaccumulative. Furthermore the vapour pressure of these substances are also relatively low, indicating that losses from water to air is expected to be low.

Table 2.2: Physical-chemical properties of the selected substances. Vapour pressure and water solubility was recalculated to uniform units. Est = estimated, calc=calculated

Abbre-viation	logKow	Water solubility	Vapour pressure Ref 1
DBP	4.57[Ref 1]	10 mg/l (20 °C)[Ref 1]	0.0097 +/- 0.0033 Pa (25 °C)
BBP	4.84[Ref 1]	2.69 mg/l (25 °C)[Ref 2]	0.00112 Pa (20 °C)
DEHP	7.5[Ref 1]	0.27 mg/l (25 °C)[Ref 2] 0.003 mg/l[Ref 5] 0.00249 mg/l (calc)[Ref 6]	0.0000189 Pa (25 °C)
L79P	6.9–8.6[Ref 7]	< 1 mg/l[Ref 1]	0.1 Pa (100 °C)
DOP	8.1[Ref 1]	0.022 mg/l (25 °C)[Ref 1] 0.0004–0.0005 mg/l[Ref 3,4] 0.00249 mg/l (calc)[Ref 6]	0.0000133 Pa (25 °C)
DINP	8.6 [Ref 6]	0.2 mg/l (20 °C)[Ref 2] 0.00011 mg/l[Ref 3] 0.000308 (calc)[Ref 6]	0.000072 Pa (25 °C)
DIDP	9.46[Ref 6]	0.00002 mg/l (20 °C)[Ref 1] 0.00017 mg/l[Ref 3] 0.0000381 (calc)[Ref 6]	0.000051 Pa (25 °C)
DUP	10.33[Ref 6]	0.83–1.39 mg/l (25 °C)[Ref 1] 0.00000441 mg/l (calc)[Ref 6]	0.015 Pa (25 °C)
DEHA	8.1 (Est)[Ref 1]	0.78 mg/l (22 °C)[Ref 1]	0.000113 Pa (20 °C)
DINA	9.56–10.4[Ref 1]	< 1 mg/l (20 °C)[Ref 1]	< 10 Pa (20 °C)
BOA	nd	nd	nd
DBEEA	3.24[Ref 1]	nd	0.0000131 Pa (25 °C)
DEHZ	9.6[Ref 1]	Insoluble in water[Ref 1]	0.000507 Pa (25 °C)

Ref 1: Lambert et al. (2010), in which the orginal references are listed
Ref 2: ChemIDplus Advanced
Ref 3: Letinksi et al. 1999
Ref 4: Ellington 1999
Ref 5: Stales et al. 1997
Ref 6: Cousins and Mackay 2000
Ref 7: CPSC 2010

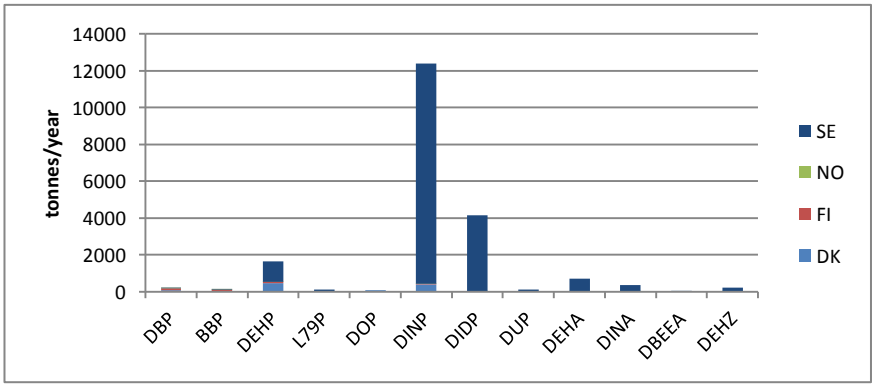
Precise water solubility measurements for compounds with water solubility >1 mg/l can easily be obtained with classic shake-flask experiments. For more hydrophobic plasticisers (e.g. DEHP, DOP, DINP, DIDP, DUP) this technique can generate unreliable results (Stales et al. 1997). The measurement problems are caused by emulsion or micelle for-

mation leading to overestimation of the true water solubility. Accurate direct measurements can be performed with "slow-stir" or "generator-column" techniques since it avoids the emulsion formation (De Bruijn et al. 1989, Ref 3,4,5 in Table 2.2). The water solubility may also be calculated by the "three-solubility" approach (Cousins and Mackay 2000, ref 6 in Table 2.2). Thus water solubilities cited from different sources can vary considerably. In this case lower values are probably more reliable.

2.1 Applications and use

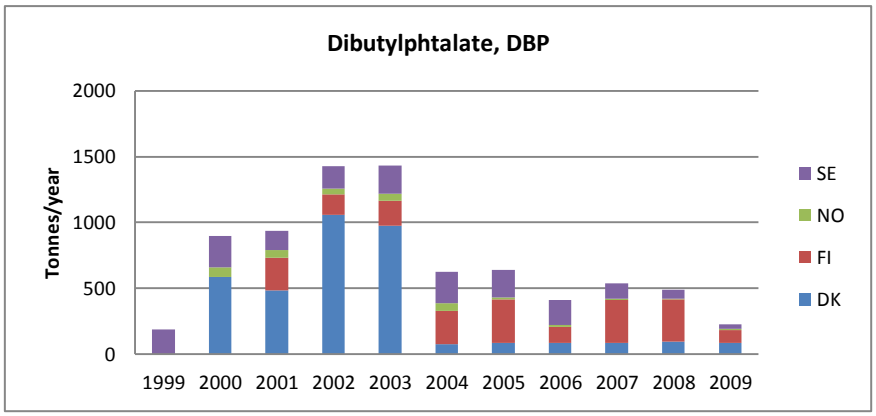
Phthalates (esters of phthalic acid) are the most commonly used plasticisers. The main use is as additive to polyvinyl chloride (PVC). Of the amount produced in Europe 93% is used for that purpose. Another area of use is in cosmetics (dibutyl phthalate, DBP). In the last ten years there has been a pronounced change in use from lower molecular weight (mainly di(2-ethylhexyl) phthalate, DEHP) to higher molecular weight (diisononyl phthalate DINP, diisodecyl phthalate, DIDP and others) phthalates. The high molecular weight phthalates now represent more than 80% of the amount beeing produced in Europe (ECPI, 2012). The change in use during the last decade is also evident from data on use of phthalates in the Nordic countries extracted from the SPIN database (SPIN, 2012). Data for DBP, butylbenzyl phthalate (BBP) and DEHP are illustrated in Figure 2.2, for DINP, DIDP, diundecyl phthalate (DUP) and dioctyl phthalate (DOP) in Figure 2.3. For dialkyl(C7–C9) phthalate (L79P) the only data according to SPIN are 753 tonnes for 2000, 454 tonnes for 2001 and 105 tonnes for 2009, all for Sweden. The latest avalible figures (2009), also for adipates and azelates, are illustrated in Table 2.3. The reporting to the SPIN database could vary between countries, and therefore uncertainties could be in the numbers, especially when comparisons are made between countries. It should also be remembered that SPIN lists ingredients in chemical preparations, not in finished consumer articles.

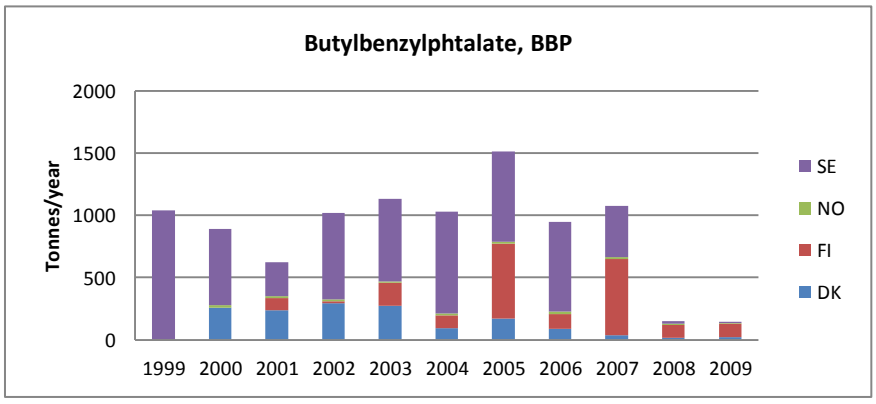
Figure 2.1 Use of plasticisers in the Nordic countries according to SPIN for the year 2009



Adipates (esters of adipic acid) are used as specialty plasticisers. In PVC applications, adipates offer enhanced low temperature properties compared to phthalates. It is not uncommon to use adipates in blends with high molecular weight phthalates to attain a desired combination of properties (ECPI, 2012). The use of di(2-ethylhexyl) adipate (DEHA) and diisononyl adipate (DINA) in the Nordic countries extracted from the SPIN database is illustrated in Figure 2.4. There were no data for benzyl octyl adipate (BOA) in the SPIN database.

Figure 2.2 Use in the Nordic countries according to SPIN for DBP, BBP and DEHP for the years 1999–2009. Please note the different y-axis scales





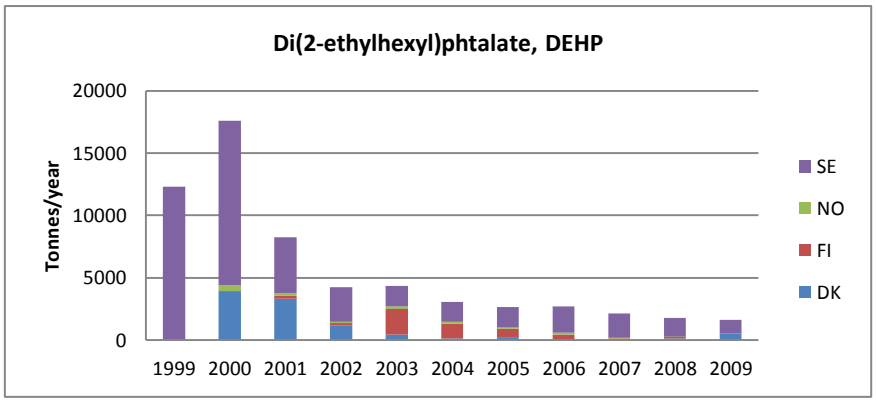
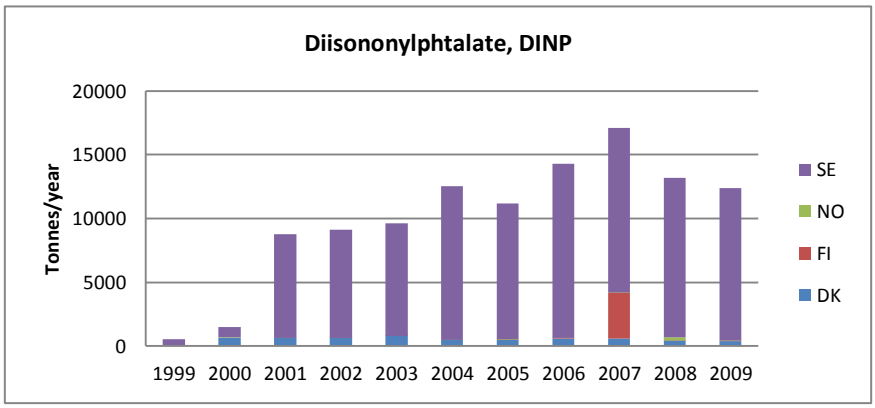
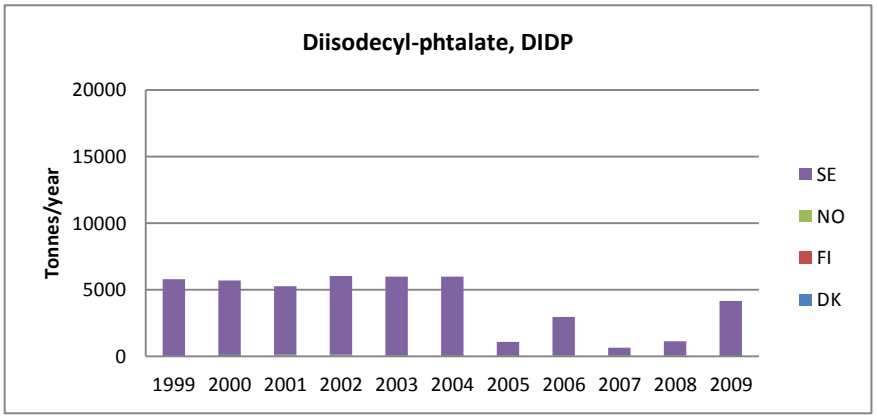
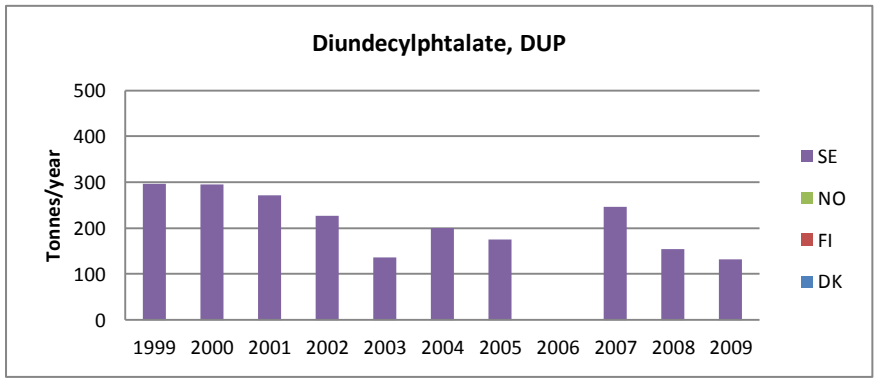
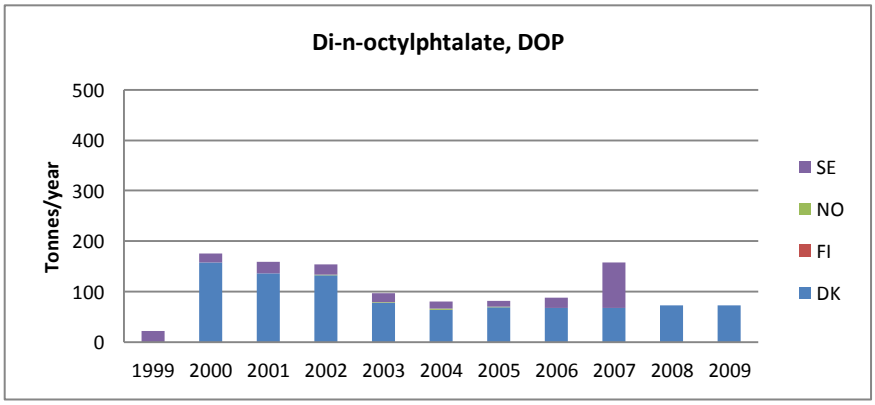


Figure 2.3 Use in the Nordic countries according to SPIN for DINP, DIDP, DUP and DUP for the years 1999–2009. Please note the different y-axis scales



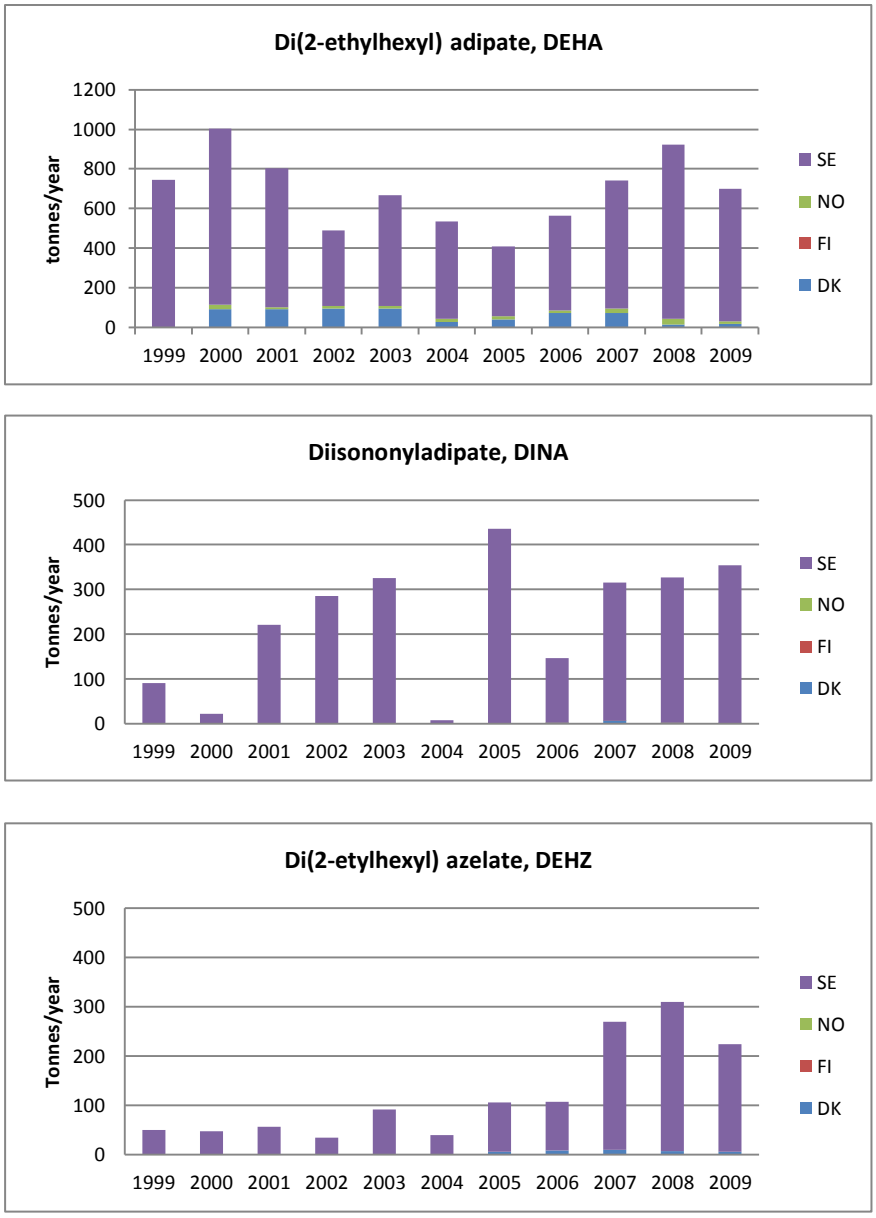






Azelates (esters of azelaic acid) impart low temperature performanc-es superior to adipates. Their use is generally limited to extremely demanding low temperature flexibility specifications (e.g. underground cable sheathing in arctic environments) (ECPI, 2012). The use of di(2-ethylhexyl) azalate (DEHZ) in the Nordic countries extracted from the SPIN database is illustrated in Figure 2.4.

Figure 2.4 Use in the Nordic countries according to SPIN for DOP, DEHA, DINA and DEHZ for the years 1999–2009. Please note the different y-axis scales



2.2 Ecotoxicology

In the substance descriptions in the report by Lambert et al. (2010) it is clear that the characteristics of the screened plasticisers are varying; some of the substances have been shown to be toxic to aquatic organisms and other substances have been shown not to be. Also the toxicity to mammals varies among the screened substances; effects seen for some substance in chronic toxicity tests were for example reprotoxic effects. Furthermore, suspicion of endocrine disruptive effects were mentioned for some substances, this will however not be further discussed here.

PNEC for the water and sediment compartments, as reported in EU risk assessments, are listed Table 2.3. For DEHP the $PNEC_{water}$ could not be specified as there were no reliable long-term studies below the water solubility of the compound (ECB 2008). However, DEHP has shown to induce effects in fish exposed via the food (ECB 2008). The $PNEC_{sediment}$ of DEHP was based on a study on frog. In the risk assessment it is also mentioned that effects on microbially mediated processes may occur at concentrations around 1 mg/kg dw (ECB 2008). In the EU risk assessments of DINP and DIDP $PNEC_{water}$ could not be specified as no toxic effects were seen in any of the performed long-term tests, nor were effects seen in studies of orally exposed fish (ECB 2003 a; ECB 2003 b). $PNEC_{sediment}$ could not be specified either, as no effects were seen in the tests conducted and as the equilibrium partitioning method was not applicable as $PNEC_{water}$ were lacking (ECB 2003 a).

Table 2.3: Predicted no effect levels (PNEC) as reported in the EU risk assessment reports. $PNEC_{sediment}$ marked with an asterix (*) were derived with the equilibrium partitioning method. For the majority of substances no EU risk assessment report was available (–)

Substance(reference)	$PNEC_{water}$	$PNEC_{sediment}$
DBP (ECB 2004)	10 µg/l	3.1* mg/kg dw
BBP (ECB 2007)	7.5 µg/l (freshwater) 0.75 µg/l (marine waters)	1.72* mg/kg ww (freshwater) 0.17* mg/kg ww (marine waters)
DEHP (ECB 2008)	na	>100 mg/kg dw[a]
L79P	-	-
DOP	-	-
DINP (ECB 2003a)	Na	na
DIDP (ECB 2003b)	Na	na
DUP	-	-
DEHA	-	-
DINA	-	-
BOA	-	-
DBEEA	-	-
DEHZ	-	-

a= effects on microbially mediated processes might occur at concentrations around 1 mg/kg dw (ECB 2008).

3. Methodology

3.1 Sampling sites and sample selection

The purpose of this screening was to sample several environmental matrices with a wide geographical distribution at locations where it's expected to find the compounds investigated.

Each country made their own selection of sample sites according to the strategy previously agreed upon in the steering group. Samples were chosen to represent areas directly influenced by human activities but also background areas. When possible, samples were chosen so as to facilitate comparisons between areas/regions. The goal was to cover the matrices effluent and sludge from waste water treatment plants (WWTPs), sediment and biota (fish and egg).

Sampling was done according to a sampling manual provided by the analysing laboratories (Appendix 1).

All samples collected are listed in Appendix 2, where also the sampling characteristics are given in detail. Below the different sampling sites from each country are presented and their locations are shown in maps, Figure 3. to Figure 3.

3.1.1 Denmark

WWTP effluent and sludge

Effluent and sludge were sampled at Esbjerg central WWTP and Ejby Mølle WWTP, Odense. Effluent was sampled at Råbylille strand WWTP, Vordingborg.

The WWTPs in Esbjerg and Odense had in 2010 loads on 115,000 and 275,000 pe respectively, while the load on Råbylille Strand was much smaller, 1,100 pe. Råbylille Strand only receives wastewater from households while the others receive from both household and industry.

Sediment

Sediment samples were collected at Vedbæk, Øresund, from Kolding Fjord and from Limfjorden.

Fish

Fish (Flounder) were sampled at Ho bugt (vicinity of Esbjerg), Hjelm bugt (vicinity of Vordingborg) and Agersö, Great Belt.

3.1.2 Faroe Islands

WWTP effluent and sludge
Effluent was sampled at Sersjantvikin WWTP, Torshavn and Klaksvik Hospital WWTP, Klaksvik. Sludge was sampled at Sersjantvikin WWTP, Torshavn and Main Hospital WWTP, Torshavn. (Influent was sampled at Main Hospital WWTP, Torshavn). The Sersjantvikin WWTP, Torshavn receives domestic wastewater only and from approx. 1,000 pe. The WWTP may be described as consisting of a primary purification step. The sampling site denoted Klaksvík Hospital WWTP in this report is best described as a sewage line. Klaksvík hospital is a small hospital with 36 hospital beds, and performes clinical chemical analyses and x-ray diagnostic analyses.

Sediment
Sediment was sampled in Torshavn harbour, near the marina and near the shipyard (station BA). Sediment was also sampled in Klaksvik harbour and in Kollafjord.

Fish
One fish sample (Arctic charr) was from Lake á Mýrunum, a background freshwater lake. Five samples (Cod, liver) came from Mýlingsgrunnur approximately 30 km NW on Faroe shelf.

Egg
Black guillemot eggs were sampled from two locations; island Skúvoy (6 eggs), and island Koltur (5 eggs). The eggs were analysed as one pooled sample from each sampling site. These locations are background areas, and will typically represent possible long-range transported contaminants.

3.1.3 Finland

WWTP effluent and sludge
Effluent and sludge samples (single, dewatered) were collected at three municipal WWTPs: Kakolanmäki, Turku (population equivalent 890,000), Viikki, Helsinki (pe 1,220,000), and Viinikanlahti, Tampere (pe 300,000). All these plants collect small or medium enterprise (SME) industrial wastewaters as well.

Sediments
Sediment was sampled at urban locations in the same cities as was used for WWTP sampling; Turku, Helsinki and Tampere.

Fish
Fish (Perch) were sampled at Pyhäjärvi in the vicinity of Tampere, at Kuhmoinen from the large lake Päijänne and in the Turku archipelago.

3.1.4 Iceland

WWTP effluent and sludge
Sludge was sampled at the WWTPs Hveragerði, Borg–Grímsnesi and Klettagarðar, Reykjavik. Effluent was not sampled.

Sediment
Two sediment samples were collected in central Reykjavik and one in the fresh water lake Þingvallavatn.

Fish
Two fish samples (Arctic charr and Brown trout) were from lake Þingvallavatn while one sample (Cod) was from the Island Seas, about 200 km NW off the coast.

3.1.5 Norway

WWTP effluent and sludge
Effluent and sludge were sampled at Rambekk WWTP, Gjøvik having lake Mjøsa as its receiving water. Rambekk WWTP treats waste water from residential buildings and industry in the municipality of Gjøvik. The treatment plant is a mechanical / chemical system with a capacity of ca. 45,000 pe (population equivalents). The plant treats 4.5–7 mill m^3 waste water each year. The plant treats sludge from Gjøvik, but also accept sludge from seven municipalities in the region. The plant produces 2,100 tonns of sludge granulat each year.

Sediment
Sediment was taken in Puddefjorden near central Bergen and in Kviturspollen approximately 30 km from Bergen towards the open sea.

Fish
Fish (Brown trout) were sampled at Vingrom, lake Mjøsa.

3.1.6 Sweden

WWTP effluent and sludge
Effluent and digested dewaterised sludge were sampled at Öhn WWTP, Umeå (129,000 pe), Ryaverken WWTP, Göteborg (640,000 pe), and Gässlösa WWTP, Borås (73,000 pe),

Sediment
Two sediment samples were collected from the vicinity of Göteborg (Björkö and Stockholmen) and one representing a more remote area in Kosterfjorden West of Strömstad. The samples from Björkö and Stockholmen were

collected with a grab sampler of Panar type. The sample from Kosterfjorden was received from the Geological Survey of Sweden (SGU).

Fish

Fish were collected at Hakefjorden at the mouth of Göta älv, Göteborg (Mackerel), at Kullen, Øresund (Herring) and at Holmöarna, Bothnian bay (Perch)

Egg

Eggs (Guillemot) were collected at Stora Karlsö. Samples (mixture of 5 eggs) were received from the Swedish Environmental Specimen Bank.

Figure 3.1 Sampling sites Denmark. Sample identification codes according to Appendix 2

Figure 3.2 Sampling sites Finland. Sample identification codes according to Appendix 2

Figure 3.3 Sampling sites Faroe Islands. Sample identification codes according to Appendix 2

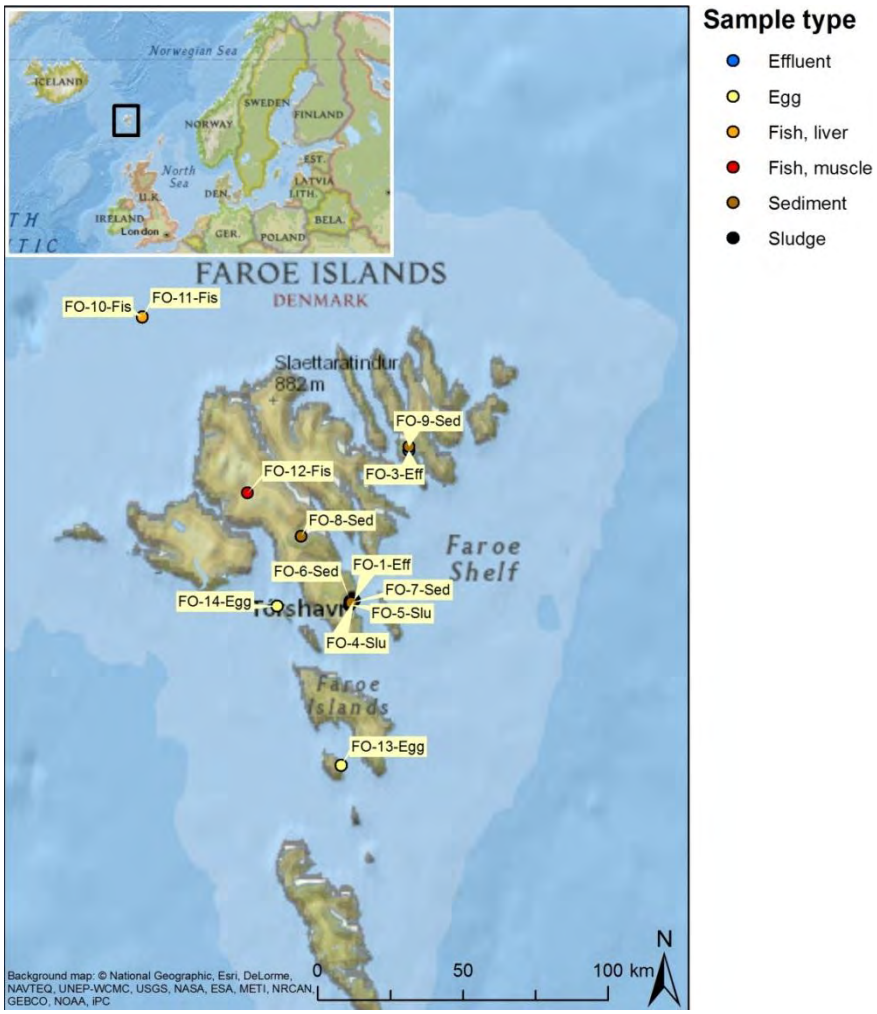


Figure 3.4 Sampling sites Iceland. Sample identification codes according to Appendix 2

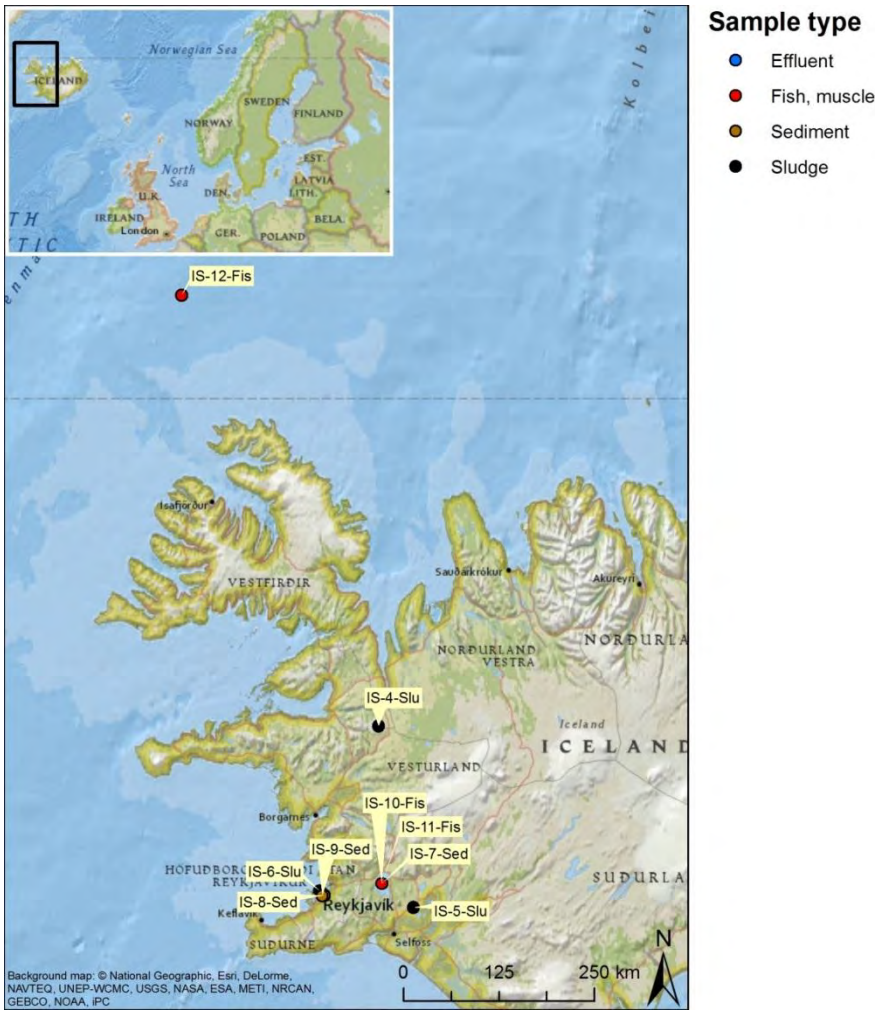


Figure 3.5 Sampling sites Norway. Sample identification codes according to Appendix 2

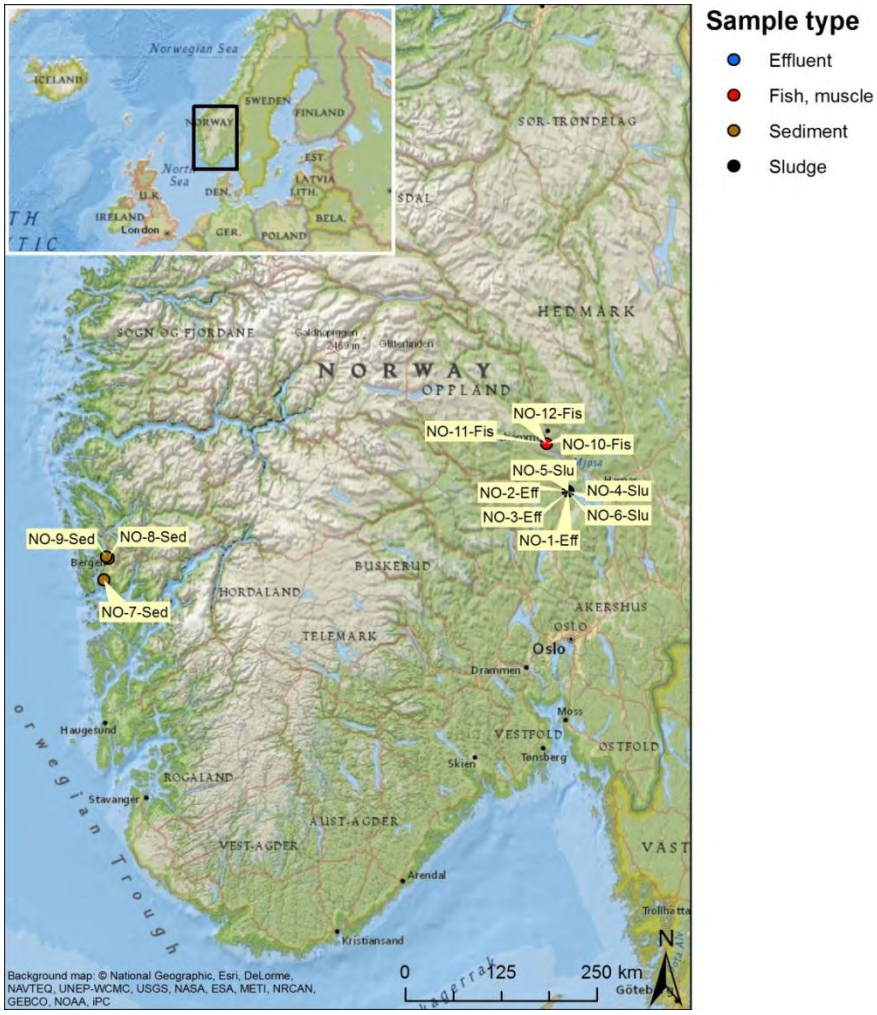


Figure 3.6 Sampling sites Sweden. Sample identification codes according to Appendix 2

3.2 Sampling methods

3.2.1 Effluent

Effluents were sampled as grab samples. For analysis of plasticisers sampling was done in glass bottles with PTFE seals and the samples were stored cool. For analysis of sweeteners 1 L PE-bottles with PP seals were used and samples were stored frozen (-18 °C) until analysis.

3.2.2 Sludge

The sewage treatment plants sludge samples were collected by trained personnel or the staff at the different plants. For analysis of plasticisers the sludge was transferred into previously preheated (400 °C) glass jars with the lid protected with Al-foil. For analysis of sweeteners 100 mL PP jars with PP lid were used. All samples were stored frozen (-18 °C) until analysis.

3.2.3 Sediment

Surface sediment (0–2 cm) samples were collected by means of a sampler. The sediment was transferred into glass jars as described for sludge and stored in a freezer (-18 °C) until analysed.

3.2.4 Biota

Fish were collected by means of suitable fishing-gear. The fish were stored in a freezer (-18 °C) until dissected. Fish samples were deliverd as whole fish or dissected in the respective countries. Fish muscle or liver was dissected for analysis by means of solvent cleaned scalpels.

A homogenised mixture of 5–10 eggs were stored in pre-cleaned glass jars at (–18 °C).

3.3 Analysis methods, plasticisers

3.3.1 Analytical standards

DINP and DIDP were donated by ECPI (European Council for Plasticisers & Intermediates, a sector group of CEFIC, March 26, 2000). The recovery (surrogate) standard deuterium labeled DEHP (d_4-DEHP) was purchased from CIL (Cambridge Isotope Laboratories,Andover, MA, USA), dipropylphthalate (DPP) from TCI (Tokyo Kasei, Organic Chemicals, Tokyo, Japan) and deuterium labeled DEHA (d_8-DEHA) from Chiron (Chiron As Trondheim Norway). Di(2-(2-butoxyethoxy)ethyl) adipate

(DBEEA) was purchased from Sigma-Aldrich, di(2-ethylhexyl) adipate (DEHA) and DEHZ from TCI, DBP, BBP, DEHP and DOP from Ultra Scientific and DUP, diisononyl adipate (DINA) and benzyl oktyl adipate (BOA) from Chiron. The phthalate mixture di-C7–9-branched and linear alkylester with the trade name Bisoflex L79P (L79P) was produced by Emery Oleochemicals GmbH.

DPP was used as recovery standard for the lower molecular weight phthalates (DBP, BBP), d_4-DEHP for the heavier molecular weight phthalates (DEHP, L79P, DOP, DINP, DIDP, DUP) and d_8-DEHA for the adipates and DEHZ.

3.3.2 Materials

Phthalates are recognised as ubiquitous pollutants in indoor as well as outdoor environments (Furtmann 1995). Therefore, it is not surprising that phthalates are generally detected even in high purity chemicals, ultra-pure water, organic solvents used for extraction and laboratory equipment (Tienpont, David et al. 2005). The contamination from laboratory equipment and environment limits the analysis of plasticisers, especially phthalates. Special routines are therefore essential in order to guarantee the integrity of the samples. The routines used at the laboratory in order to minimise the risk for contamination of the samples are briefly described below.

All glass equipment were wrapped in aluminium foil and heated to 400 °C for 8 hours. The aluminium foil prevents re-contamination of the equipment until used. All equipment made of Teflon and metal was washed with solvent before use. During the whole analytical procedure samples, extracts, solvents and chemicals were carefully protected from air precipitation and dust that has been proved to be a source of phthalates (Tienpont, David et al. 2005). This was accomplished by covering test tubes, jars and other equipment with clean aluminium foil (Parkman and Remberger 1994). Solvents used for extraction were delivered from Rathburn Chemical Ltd. (Peeblesshire, Scotland) and were checked before used. Ultra-pure water was produced by Milli-Q equipment. Batches of water from this equipment were stored in glass containers and were checked prior to use. The checked batches of solvents and water were exclusively applied for this project. Chemicals and equipment such as Na_2SO_4, were thermally treated at 400 °C before use.

SPE (solid phase extraction) columns containing ethylenediamine-N-propyl modified silica (PSA) were prepared in glass columns immediately before use. Pre-cleaned GF/C-filters were used as frits. The columns were pre-cleaned carefully and activated prior to use by passing hexane, MTBE, methanol and ultra-pure water through the columns.

SPE columns containing C18 used to concentrate water samples were carefully cleaned before used with MTBE, acetone, methanol and ultra-pure water. Low and consistent blank samples were difficult to achieve

even after such careful cleaning. The problem seems to be the frits placed on both sides of the SPE-phase. Soxhlet extraction of the frits before used decreased blank values.

3.3.3 Sample preparation

WWTP effluent

The effluent water samples (200–400 mL) were subjected to solid phase extraction (SPE; C18). The sample was spiked with recovery standards (d_8-DEHA, DPP and d_4-DEHP). To avoid clogging of the SPE-column filter aid (diatomaceous earth or Empore filter aid 400) was used. Following the sample the SPE-column was rinsed with ultra-pure water and dried under full vacuum suction on the vacuum manifold. The analytes were eluted with methanol, MTBE and finally hexane. The extracts were combined and methanol was washed away by shaking the extract with ultra-pure water. The extract was dried over sodium sulphate and cleaned up (see below).

Sludge and sediment

The pore water in the sediment samples was separated by centrifugation before extraction. Centrifugation of the sludge was not necessary because the analysed sludges were dewatered. Sludge samples were acidified with phosphorous acid prior to extraction. The sample was fortified with recovery standards (d_8-DEHA, DPP and d_4-DEHP), mixed carefully and subsequently extracted twice with acetone. The first extraction lasted for 16 h and the second 30 min including 5 min in ultrasonic bath. The acetone in the pooled extract was washed away by dilution with ultra-pure water and subsequent extraction with hexane:MTBE. The organic extract was safeguarded after phase separation. The water phase was extracted one more time with a mixture of hexane and MTBE. The combined extract was subjected to clean-up (see below).

Fish, Egg and liver

Samples (muscle, 5 g; liver 4 g, egg 2 g) were spiked with recovery standard (d_8-DEHA, DPP and d_4-DEHP) and homogenised in acetonitrile. Sodium sulphate was added to facilitate the phase separation. The sample was shaken vigorously for one minute. The extract was safeguarded after centrifugation and the samples were extracted once more with acetonitrile. The extracts were combined and the acetonitrile removed by shaking with water and heane:MTBE. The extracts were dried, concentrated and the solvent changed to hexane followed by clean-up.

Clean-up of extracts

The raw extracts contain high amounts of “matrix related compounds” that may interfere with the target analytes leading to inaccurate quantification. It may also lead to damage of the chromatographic system leading to poor separation and sensitivity.

PSA-columns were used for the clean-up of extracts from effluents, and biota. Phthalate contamination from these columns was a serious problem. This was solved by replacing the PP-frits with small GF/C-filters. The columns were carefully solvent cleaned before running the samples. The analytes were eluted with hexane: MTBE.

Clean-up of sludge and sediment-extracts were performed on a dual SPE-column containing graphitised carbon black (GCB) and PSA. This column has the advantage to retain the dark brown material and polar compounds that is co-extracted with the plasticisers. The sample, dissolved in hexane, was applied on the column and the analytes were eluted with toluen: MTBE.

The eluates were concentrated using a clean stream of N_2 gas, spiked with injection standard and stored in a freezer (-18 °C) prior to GC-MS/MS analysis.

3.3.4 Instrumental analysis

The extracts were analyzed on a 7890A gas chromatograph coupled to a 7000A Triple Quad MS (Agilent Technologies Inc., Santa Clara, CA, USA). The injection was made pulsed splitless at 250 °C. The fused silica capillary column (VF-5MS 30 m x 0.25 mm i.d. x 0.25 µm film thickness, Varian) was held at 45 °C for 1 min, ramped 15 °C/min to 300 °C, and held isothermal for 10 min. The transfer line was held at 280 °C. Helium was used as carrier gas. The detector was used in MRM mode with electron ionisation at energy 70 eV. The analytes were identified by their characteristic retention times and two characteristic precursor/product ion pairs (MRMs). The instrument was calibrated with a six point calibration curve. Quantification was based on comparison of peak abundance to the known response of the internal standard (biphenyl). The reported analyte concentrations were calculated according to the determined recovery of surrogate standard.

3.3.5 Quality control

When performing environmental screening all steps in the study such as selection of sampling site, sampling frequency, time of sampling, performing of sampling, transport and storage of samples, chemical analysis and data treatment are generating some degree of uncertainty. To quantitatively estimate the contribution of all steps is an extremely difficult task or not possible at all. However, we will discuss the relevance of the different contributors in a qualitative way.

One important question is whether a sample is representative for a given time period or a given region. Many of the selected compounds are intermittently emitted to the environment and a constant concentration of these compounds in the environment is not expected. In this screening, the samples were collected within a narrow time frame and at dif-

ferent geographical locations. The results obtained here are therefore only a snapshot of the reality at those places at the given time.

Factors with influence on sampling uncertainty are analyte loss due to adsorption to sample containers, wastewater flow and particle content, tidal water current, contamination and degradation during transport and storage. The uncertainty due to loss of analyte is minimised (especially for water samples) by the laboratory´s selection of sample containers.

The uncertainty of the chemical analysis is governed by loss during extraction and clean-up, interference from other compounds, trueness of analytical standards, instrumental parameters, and contamination.

The following quality criteria were used to ensure correct identification and quantification of the target compounds: (a) the retention times should match those of the standard compounds within ± 0.05 min., (b) the intensity ratios of the selected MRMs should be within ± 15 % of that observed for the standard compounds (c) the signal-to-noise ratios should be greater than 3:1.

Field blanks were collected at several sampling stations. An analytical method blank was included for each sample batch analysed to assess background interferences and possible contamination of the samples. Concentrations below field blank levels were treated as not detected. Possible background levels of analytes were subtracted from measured sample values.

Limit of quantification (LOQ) was defined as a signal 10 times the standard deviation of the blank values. Internal standard was added to the sample at the start of the working-up procedure. The internal standard has similar chemical and physical properties as the compounds to be analyzed. If available, isotopically labelled internal standards were used.

In this investigation four plasticisers consisting of isomeric mixtures were determined (L79P, DINP, DIDP, DINA). In this case the analytes were determined from the sum of a cluster of chromatographically unresolved peaks in a quite broad retention window (about 4 min). Substances with similar retention times which produce masses similar to the analytes can interfere with the analyte. This may lead to incompletely resolved signals and to additional signals in the chromatographic pattern. This can affect accuracy and precision of the analytical results.

The extraction efficiency for the different matrixes based on recovery standards are summarized in Table 3.1.

Table 3.1: Extraction efficiency (%) of recovery standards for different matrices

	Effluent	Sediment	Sludge	Biota
d_8-DEHA	90	86	77	55
DPP	99	78	75	51
d_4-DEHP	97	78	76	42

The performance of the analytical metods used was examined in spiking experiments. The authentic samples used were collected from lakes with known low antropogenic influence and are included in the national monitoring programme of contaminants (A. Bignert; The Environmental speciment bank at Museum of Natural History (NRM)). The added amount of analytes were close to specified LOQ. The spiked samples were treated according the the same analytical protocol as the samples.

The recovery that was obtained for the matrices effluent, sediment and biota of the detected analytes are presented in Table 3.2. The results are adjusted according to the respective recovery standards and the background subtracted (se 3.3.1).

Table 3.2: Extraction recovery (%) of the analytes for different matrices

	Effluent	Sediment	Biota
DBP	111	115	101
BBP	104	125	111
DEHP	120	107	62
L79P	99	77	44
DOP	78	96	39
DINP	76	98	42
DIDP	68	96	39
DUP	74	85	29
DEHA	90	90	nd

The results are adjusted according to the respective recovery standards

The uncertainty of the analytical method used was estimated by analysis of parallel samples of authentic matrices. From these results average concentration, standard deviation, and relative standard deviation, RSD (Table 3.3) were calculated. Due to low concentrations, RSD could not be evaluated in this way for all analytes. In these cases spiked samples were used (results indicated in italics). An approximate 95% confidence interval for the analytical results could be obtained by multiplying RSD from Table 3.3 with a "coverage factor" of 2 (ISO, 1993).

Table 3.3: RSD (%) evaluated from analysis of authentic parallel samples

	Effluent	Sludge	Sediment	Fish
DBP	7	7	*6*	*3*
BBP	5	3	27	*10*
DEHP	13	6	9	*7*
L79P	*10*	23	17	*17*
DOP	*12*	16	33	*26*
DINP	5	7	11	*9*
DIDP	7	13	13	*15*
DUP	*14*	6	*11*	*6*

Figures in italalics ar data obtained through spiking experiments.

LOQs were based on variation of blank samples (10 x SD) or (when data were not available) were estimated from the calibration curve (in italics), Table 3.4.

Table 3.4: Summary of LOQs for the matrices included in the investigation

	Effluent ng/l	Sludge µg/kg dw	Sediment µg/kg dw	Biota µg/kg ww
DBP	*50*	*8*	*8*	*4*
BBP	20	11	4	2
DEHP	200	80	20	4
L79P	*50*	8	8	*2*
DOP	*20*	10	5	1
DINP	80	100	30	*40*
DIDP	80	100	20	*40*
DUP	*20*	20	*10*	*6*
DEHA	25	10	1	*30*
BOA	*20*	*8*	*10*	*8*
DINA	*130*	*200*	*100*	*40*
DEHZ	*15*	*10*	*5*	*2*
DBEEA	*150*	*100*	*50*	*10*
Sample amount	1 l	1 g dw	4 g dw	10 g ww

The LOQs are based on variation of blank samples or (when data were not available) were estimated from the calibration curve (in italalics).

Those LOQs were used in the calculation of the results. In addition LOQs were also determined from variation of the results (10 x SD) for spiked samples (see above), Table 3.5.

Table 3.5: Summary of LOQ retrived in spiking experiments

	Effluent ng/l	Sediment ng/g dw	Biota ng/g ww
DBP	38	9	3
BBP	15	11	9
DEHP	30	42	6
L79P	22	5	4
DOP	3	8	4
DINP	108	60	44
DIDP	71	63	77
DUP	17	23	4
DEHA	8	8	nd
Sample amount	1 L	4 g dw	10 g ww

Sampling blanks (see Appendix 1 Sampling manual) for water were sent to Denmark, Norway and Finland. After being returned to the laboratory they were analysed together with the effluent samples. All plasticisers showed concentrations below LOQ.

Sampling blanks used for solid samples (sediments and biota) were sent to Faroe Islands, Island and Sweden. The results are summerised in the leftmost columns in Table 3.6. The concentrations were all below LOQ exept for a low concentration of DEHP in one of the blanks.

Table 3.6: Sampling blanks for solids. Results calculated as for biota samples

	FO-15-BLS µg/kg ww	IS-15-BLS µg/kg ww	SE-15-BLS µg/kg ww	IS-16-BLS Jar µg/kg ww	IS-16-BLS Lid µg/kg ww
DBP	<LOQ	<LOQ	<LOQ	<LOQ	7.9
BBP	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
DEHP	<LOQ	15	<LOQ	<LOQ	14
L79P	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
DOP	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ
DINP	<LOQ	<LOQ	<LOQ	620	8500
DIDP	<LOQ	<LOQ	<LOQ	7800	21000
DUP	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ

For LOQ values see Table 3.4.

For a few samples (FO-13-Egg, FO-14-Egg and IS-11-Fis) a different type of sampling jar than that sent out from the laboratory was used. This glass jar had a metall lid with a gasket made from a polymer material. An empty jar of this type (labeled IS-16-BLS) was analysed as a blank. Results for the jar and the lid are presented separately in Table 3.6, rightmost columns. DINP and DIDP was found predominantly on the lid, probably originating from the gasket. The jars were also fitted with a clean aluminium foil under the lid. This should protect the sample but problems with contamination cannot be fully ruled out *e.g.* due to damaged aluminium foil.

Originally two fish liver samples were delivered in the probably contaminating type of sample jars. The livers contained high concentrations of DINP and DIDP (data not shown). When additional fish from the same area were delivered as whole fish and the livers (FO-15-Fis–FO-19-Fis) analysed, DINP and DIDP concentrations were <LOQ.

3.4 Analysis methods, sweeteners

3.4.1 Sample preparation

Effluent water (50 mL) was acidified to pH 3 with HCl and internal standards (d6-sucralose, d3-aspartame and d11-cyclamate, all from Toronto Research Chemicals Inc.) were added.

Sludge (3 g) was shaken in 100 mL diluted HCl for 30 min and internal standards were added. The pH should be 3 in the suspension and was adjusted if necessary. The suspension was centrifuged at 1,000 rpm for 5 min and the liquid phase collected.

The aqueous samples, prepared in this way were solid phase extracted differently depending on the analyte.

Sucralose

The sample was solid phase extracted on a column (Oasis HLB plus, Waters Corp.) installed with a pre-filter (nylon), both preconditioned with methanol. The column was washed with 0.01M HCl, deionized water (MilliQ-plus), 1 v/v% methanol in 0.01 M HCl, 20 v/v% methanol in de-

ionized water and ammoniumhydroxide. The column was dried by suction and sucralose was eluted with 3 x 3 mL acetone : methanol (5:1, v/v). To remove additional matrix compounds the extract was passed through a mixed-mode ion exchange SPE-cartridge (Isolute-MM, Biotage), preconditioned with methanol. Any sucralose remaining in the SPE-resin was washed out with additional acetone:methanol, which was pooled with the extract. The extract volume was reduced to 1 mL by evaporation under nitrogen (Zymark TurboVap II Concentration Workstation, Caliper Life Sciences). Further clean up was performed by passing the extract through another mixed-mode ion exchange SPE cartridge (Oasis MAX, Waters Corp.) with subsequent extract volume reduction.

The method was based on NILU's already established method (Brorström-Lundén et al, 2008). The performance and quality of this method was tested in an international interlaboratory comparison (Kaj, 2009).

Cyclamate

The sample was solid phase extracted on a column (Oasis HLB plus, Waters Corp.) installed with a pre-filter (nylon), both preconditioned with methanol. The column was washed subsequently with 0.01M HCl, and 1 v/v% methanol in 0.01 M HCl. The column was dried by suction and cyclamate was eluted with 3 x 2 mL deionized water:methanol (5:1, v/v). The extract volume was reduced by evaporation under nitrogen (Zymark TurboVap II Concentration Workstation, Caliper Life Sciences). Several tests were done to optimize the SPE-procedure and to try to find a suitable method for further clean up. The method was based on a paper by Scheurer (Scheurer, M et al. 2009). A more polar elution solvent seemed to be more efficient than methanol, that was suggested in the paper. The SDB-1 columns (Bakerbond SDB-1, 200 mg/6 mL from J.T Baker) refered to in the paper were tested and were shown to have the same properties as the HLB column used for sucralose.

Aspartame

The sample preparation method described by Scheurer (Scheurer, M et al. 2009) could not be adopted directly for the current samples, as the matrix effects were significant. Several tests were done to optimize the SPE-procedure and to try to find a suitable method for further clean up. Effluent water samples were analysed with the method described below, without giving a reasonable LOQ. The matrix effects in sludge are assumed to be even greater. There were no significant amounts in any of the effluent water samples. As the amounts of sweeteners in general are assumed to be lower in sludge, it is not expected to find significant amounts and the LOQ was therefore only estimated.

The sample was solid phase extracted on a column (Oasis HLB plus from Waters Corp.) installed with a pre-filter (nylon), both preconditioned with methanol. The column was washed subsequently with 0.01M HCl and 1 v/v% methanol in 0.01 M HCl. The column was dried by suction and aspar-

tame was eluted with 3 x 3 mL of acetone: methanol (5:1, v/v). The extract volume was reduced by evaporation under nitrogen (Zymark TurboVap II Concentration Workstation, Caliper Life Sciences).

3.4.2 Instrumental analysis

The extracts were analyzed on an Acquity ultra performance liquid chromatograph coupled to a Time-of-flight Mass Spectrometer (Waters LCT Premier XE). Compound separation was performed with a reversed phase column (Acquity UPLC HSS T3, 2.1 mm ID x 100 mm length, particle size 1.8 µm, Waters). The injection volume was 5 µL. Deionized water was used as solvent A and methanol as solvent B. The flow rate was 0.4 mL/min.-The binary gradient started with 95% A. Solvent B was introduced at a linear rate up to 90 % B at 5 min and kept isocratic until 5.5 min. At 6 min solvent B was set to 5% and the column was equilibrated up to a total runtime of 7 min.

The analytical detector was equipped with a Z-spray electrospray ion source, which was optimized to the following values: Negative W mode, sample cone 30 V, capillary voltage 2,800 V, source temperature 100 °C, cone gas flow 4 L/h, desolvation temperature 350 °C and gas flow 767 L/h. The scan mass area was 50–500 m/z with 0.3 s frequency. Data processing and instrument control were performed by the Masslynx software, and the quantification was performed with signal extraction of a peak width of 100 ppm (sucralose/cyclamate) and 50 mDa (aspartame).

Table 3.7: Quantifier and qualifier ions used for mass spectrometric detection

Component	Mw	Quantifier M-1	Qualifier 2° M-1
d11-cyclamate	190	189	
Cyclamate	179	178	
d6-sucralose	402	401	403
Sucralose	398	397	395
d3-aspartame	297	296	
Aspartame	294	293	

The instrumental method was based on NILU's already established method for sucralose (Brorström-Lundén, E. et al. 2008) with adaption to an UPLC column.

3.4.3 Quality control

For each analyte laboratory blanks followed the sample preparation. Recovery for the added internal standard was determined. Since there were no measurable signal for either method blank nor field blank samples, a slightly different approach for determination of the Limits of quantification (LOQ) was applied. LOQ, defined as 10 times the signal to noise ratio (S/N), was determined manually by examining the S/N-level in the sample or in the quantification standard, with the recovery and

sample extraction volumes taken into account. The accuracy of the method was determined by spiking authentic samples and processing them according to the complete analytical process, while the repeatability was investigated by analyzing duplicate samples. Based on these data the measuring uncertainty is estimated to be lower than 40% for all three analytes. This uncertainty is in accordance with results of an international interlaboratory comparison on sucralose (Kaj, 2009).

Table 3.8: Results of quality assurance for effluent water

	Average recovery	Average lab blank
Sucralose	31 %	< 0.01 µg/L
Cyclamate	43 %	< 0.01 µg/L
Aspartame	25 %	< 0.01 µg/L

Table 3.9: Results of quality assurance for sludge

	Average recovery
Sucralose	46 %
Cyclamate	56 %
Aspartame	25 % *

* Estimated from effluent water

After the study was finalised, two papers were published showing the difficulties in Aspartame analysis and the rapid degradation of Aspartame under environmental conditions (Berset and Ochsenbein 2012; Scheurer et al. 2012).

4. Results and discussion, plasticisers

4.1 Measured concentrations

All measured concentrations of individual substances are tabulated in Appendix 3 and 4. An overview of the detection frequencies, *i.e.* the fraction of samples where a substance was found in a concentration above the limit of quantification (LOQ) for the different sample matrices, is given inTable 4.1.

Table 4.1: Percentage of samples with a concentration above LOQ for the individual substances in the different sample matrices

	Effluent	Sludge	Sediment	Fish	Egg
# of samples	14	15	18	21	4
DBP	79%	100%	84%	48%	50%
BBP	100%	100%	63%	0%	0%
DEHP	93%	100%	84%	81%	0%
L79P	7%	31%	16%	0%	0%
DOP	64%	88%	26%	0%	0%
DINP	86%	100%	95%	19%	50%
DIDP	64%	100%	84%	10%	0%
DUP	0%	31%	11%	0%	0%
DEHA	71%	94%	68%	0%	0%
BOA	14%	25%	0%	0%	0%
DINA	0%	0%	0%	0%	0%
DBEEA	0%	0%	0%	0%	0%
DEHZ	0%	6%	0%	0%	0%

Generally, the phthalates and the adipate DEHA were most frequently found. The adipates DINA and DBEEA were not found at all. The azelate DEHZ was found only once in sludge.

In the following, the concentrations found are presented in more detail.

4.1.1 WWTP effluent and sludge

Effluent

Seven of the plasticisers were frequently detected in effluents, two only occasionally. The dominating plasticisers were in declining order of their median concentration DEHP, DINP, DIDP, DBP, BBP, DEHA and DOP. The median concentration of DINP was approximately one third of the value for DEHP. The concentration of DIDP was significantly lower and at the same level as DBP and BBP (Table 4.2, Figure 4.1). The concentrations often ranged 2–3 orders of magnitude.

Table 4.2: Effluents (n=14). Percentage of samples with a concentration above LOQ (DF), minimum, median and maximum concentration (ng/l). Substances with DF=0 are not shown

	DF	Conc, min	Conc, median	Conc, max
DBP	79%	<50	130	510
BBP	100%	20	120	610
DEHP	93%	<200	1,600	15,000
L79P	7%	<50	<50	270
DOP	64%	<20	22	59
DINP	86%	<80	470	27,000
DIDP	64%	<100	180	4,000
DEHA	71%	<25	47	1,300
BOA	14%	<20	<20	270

Figure 4.1 Effluents. Median concentration (ng/l)

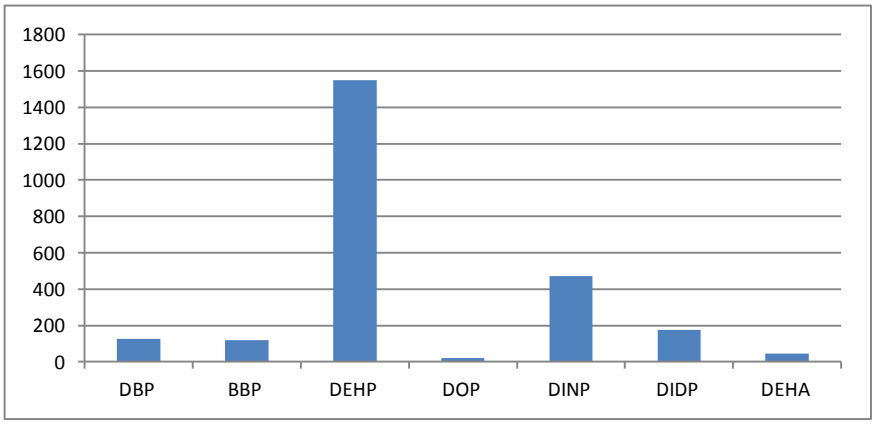
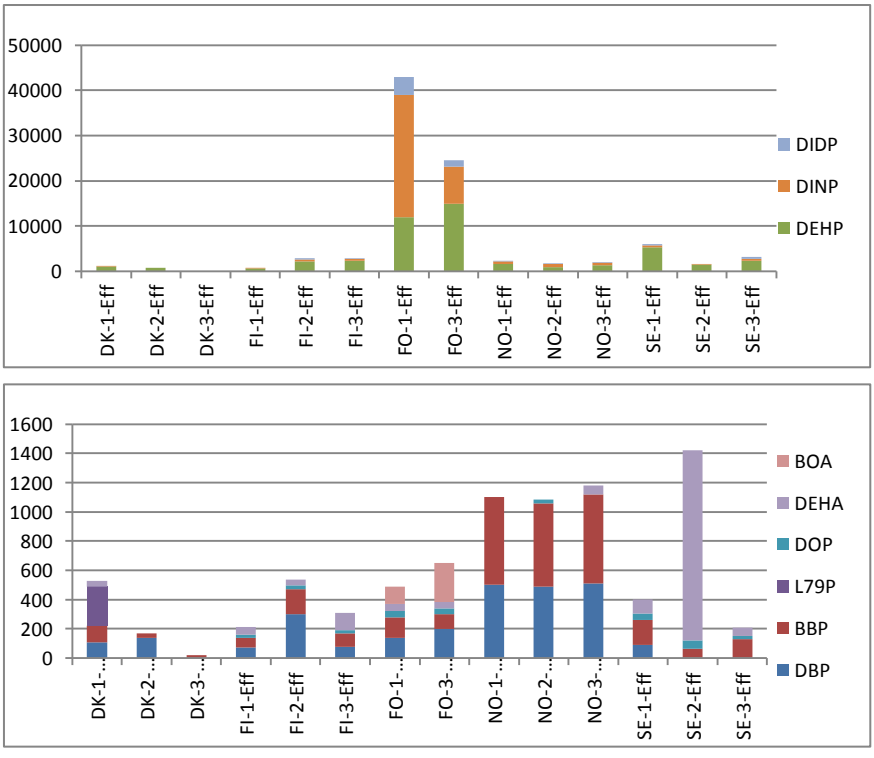


Figure 4.2 Concentration (ng/l) of plasticisers in individual effluents. Above: DEHP, DINP, DIDP, below: remaining substances, note the different y-axis scales.



The concentrations of plasticisers in the individual effluents are presented in Figure 4.2. The effluents from Faroe Islands clearly showed the highest summed concentrations.

DEHP was the dominating plasticiser in all effluents with two exceptions: FO-1-Eff (Torshavn) where DINP dominated and DK-3-Eff (Vordingborg) where only a low concentration of BBP was detected. DINP was in most cases the second compound of importance. DINP, DBP and BBP competed for third place. Again, with DK-3-Eff as an exception, DEHP, DINP and DIDP taken together made up 54–99 % of the total amount.

The relative concentrations of DIDP to DEHP in effluents from different countries were quite similar, the ratio DINP to DEHP somewhat more varying influnced by one of the effluents from Faroe Islands (FO-1-Eff) (Table 4.3). It should be observed that all Norwegian effluents are from the same WWTP. There is no data on effluents from Iceland since no samples were delivered.

Table 4.3: Relative concentrations of selected phthalates in effluents

Country	DINP / DEHP	DIDP / DEHP	DIDP / DINP
DK	0.15	–	-
FI	0.15–0.20	0.05–0.10	0.31–0.49
FO	0.54–2.3	0.09–0.33	0.15–0.17
NO	0.38–0.64	0.11–0.21	0.27–0.32
SE	0.09–0.23	0.04–0.16	0.41–0.70

The domination of DEHP is somewhat puzzling since the relative amounts of DEHP, DINP and DIDP in the effluents do not mirror the use of plasticisers according to the SPIN database (Figur 2.1). The use of DEHP has successively been replaced by DINP. One possible explanation to the discrepancy between the current consumption and what is detected in WWTP effluents may be that the total amount of DEHP accumulated in the technosphere under decades still is the dominating pool of phthalates and therefore also occurs in high concentrations in WWTP effluents. For example, phthalate-containing materials in buildings constitute a large reservoir of DEHP, and in-use release from this reservoir may be a significant environmental source (Batterman et al. 2009). Another contributing factor may be that DEHP have a higher water solubility than DINP and DIDP (see Table 2.2).

When DIDP was found the concentration was always lower than that for DINP and the ratio DIDP / DINP was fairly constant (Table 4.3).

The Norwegian effluents showed the highest concentrations of DBP and BBP.

Two of the adipates were detected in the effluents, DEHA and BOA. Of these, DEHA was most frequently found (71%) and it also has the highest usage of the adipates according to the SPIN database (Figure 2.4). DEHA was measured in concentrations <25–1,300 ng/l, with the highest concentration in an effluent from Sweden, SE-2-Eff (Ryaverken WWTP, Göteborg). In SE-2-Eff, DEHA occured in similar levels as DEHP.

In a previous Swedish screening (Remberger et al. (2005), DEHA was not detected in any effluent water. This discrepancy may be a result of the lower LOQ in the present study (25 ng/l) compared to 200 ng/l in the previous.

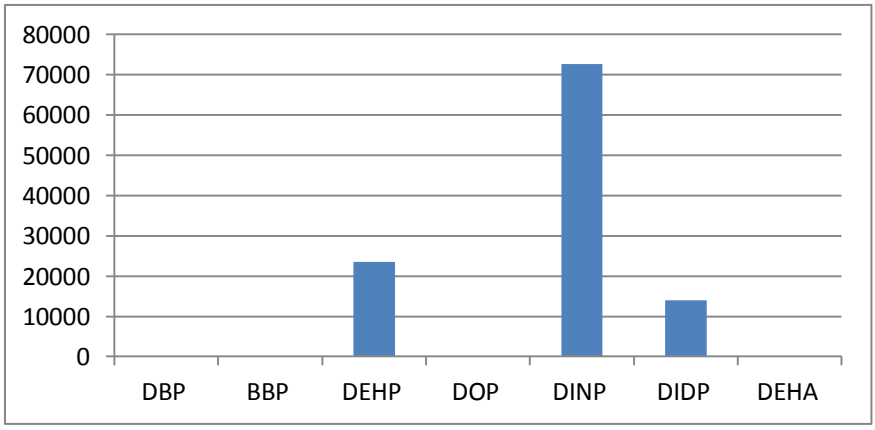
Sludge

DINP, DEHP and DIDP were the dominating plasticisers in sludge and were found in all samples. DBP, BBP, DOP and DEHA were always or nearly always found but at much lower concentrations. L79P, DUP, BOA and DEHZ was occasionally found (Table 4.4, Figure 4.3).

Table 4.4: Sludge (n=15). Percentage of samples with a concentration above LOQ (DF), minimum, median and maximum concentration (µg/kg dw). Substances with DF=0 are not shown

	DF	Conc, min	Conc, median	Conc, max
DBP	100%	27	85	610
BBP	100%	81	200	1,100
DEHP	100%	12,000	24,000	67,000
L79P	31%	<8	<8	390
DOP	88%	<10	68	220
DINP	100%	17,000	73,000	160,000
DIDP	100%	1,800	14,000	42,000
DUP	31%	<20	<20	1,400
DEHA	94%	<10	29	970
BOA	25%	<8	<8	1,100
DEHZ	6%	<10	<10	32

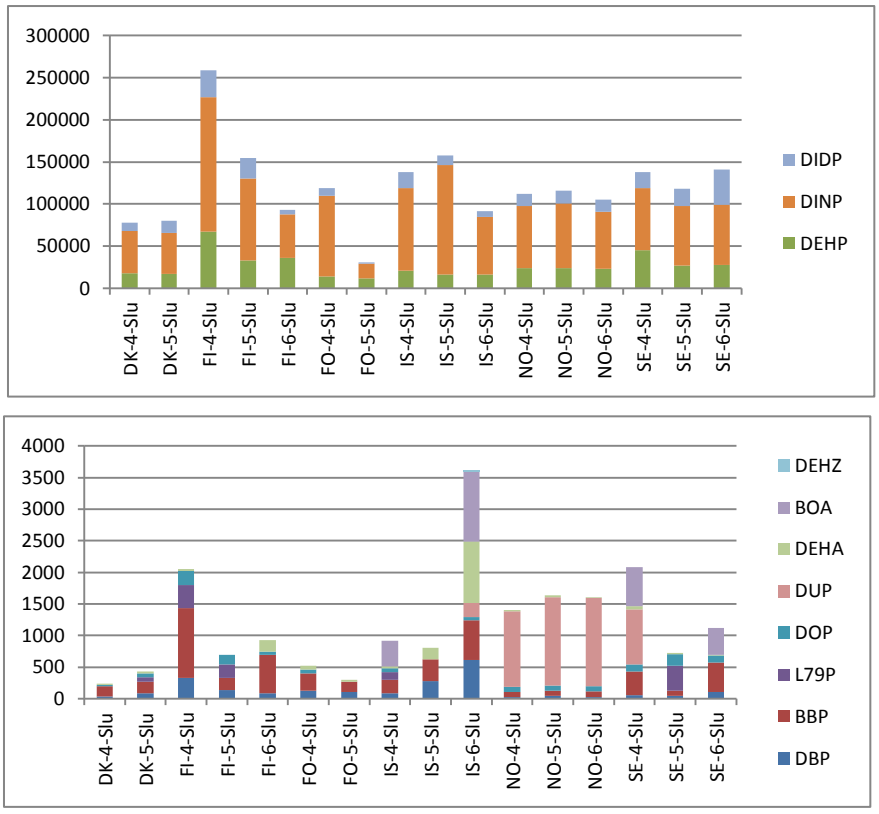
Figure 4.3 Sludge. Median concentrations (µg/kg dw).



The concentrations of the individual plasticisers ranged about one order of magnitude which is quite small compared to the case for effluents.

The concentrations in sludge in idividual samples are presented in Figure 4.4.

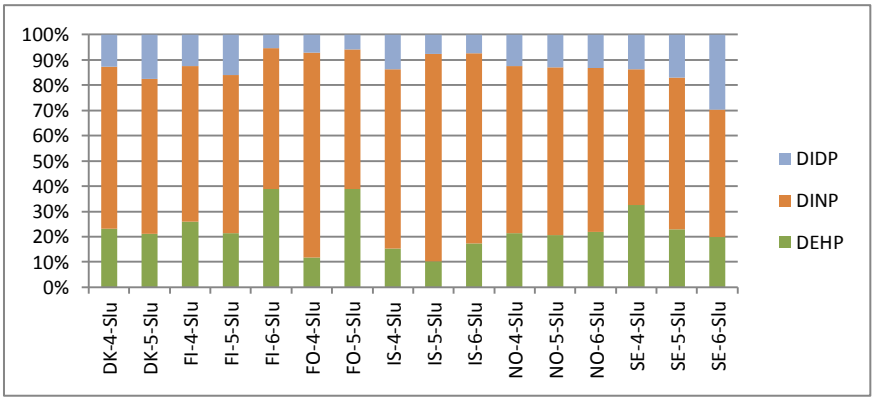
Figure 4.4 Concentration (µg/kg dw) of plasticisers in individual sludge samples. Above: DEHP, DINP, DIDP, below: remaining substances, note the different y-axis scales



DINP, DEHP and DIDP taken together made up 96–99.7% of the summed concentration of all measured plasticisers.

Relative concentrations of DEHP, DINP and DIDP are illustrated in Figure 4.5

Figure 4.5 Relative concentration of DEHP, DINP and DIDP in individual sludge samples



There were no great differences in the composition of the plasticisers in the sludge among the Nordic countries. The highest summed concentration,

260,000 µg/kg dw, was found in a FI-4-Slu (Turku). This concentration can also be stated as 0.026 % dw.

The concentrations of DINP, DEHP and DIDP were in the same range as detected in a Swedish survey 2006: DINP 37,000–65,000 µg/kg dw, DEHP 36,000–80,000 µg/kg dw, DIDP 15,000–51,000 µg/kg dw (Palm-Cousins et al. 2007).

The composition of phthalates in sludge (Figure 4.3) mirrors quite well the consumption of phthalates in the Nordic countries (Figure 2.1).

The median concentrations in effluent water and sludge were used to calculate the concentration ratios DINP / DEHP and DIDP / DEHP. The result (Table 4.5) of this calculation clearly shows that the heavier phthalates, DINP and DIDP, are enriched relative to DEHP in the solid phase (sludge). This is expected given their high K_{ow} and low water solubility.

Table 4.5: Relative concentrations (median for all samples) of DINP and DIDP to DEHP in effluent and sludge

Matrix	DINP / DEHP	DIDP / DEHP
Effluent	0.2	0.1
Sludge	3	0.6

The two adipates DEHA and BOA were, as was the case for the effluents, also detected in the sludge. The detection frequencies were 94% and 25%, respectively. BOA was only detected in sludge collected from Iceland and Sweden. The highest concentrations of both BOA (1 100 µg/kg dw) and DEHA (970 µg/kg dw) occurred in a sample from Island IS-6-Slu (Borg-Grimness).

4.1.2 Sediment

Six out of 13 plasticisers were frequently detected and three less frequently. The quantitatively dominating phatlates were in declining order DINP, DEHP and DIDP (Table 4.6, Figure 4.). Their concentrations ranged 1–2 orders of magnitude. The adipate, DEHA, was also frequently detected but in lower concentrations. Four plasticisers were not detected in any of the sediments: BOA, DEHZ, DINA, and DBEEA.

Table 4.6: Sediments (n=18). Percentage of samples with a concentration above LOQ (DF), minimum, median and maximum concentration (µg/kg dw). Substances with DF=0 are not shown

	Det freq	Conc, min	Conc, median	Conc, max
DBP	84%	<8	45	410
BBP	63%	<4	16	3,000
DEHP	84%	<80	1,000	9,500
L79P	16%	<8	<8	29
DOP	26%	<5	<5	12
DINP	95%	<30	1,900	17,000
DIDP	84%	<20	510	36,000
DUP	11%	<10	<10	58
DEHA	68%	<1	3	27

Figure 4.6 Median concentrations (µg/kg dw) of selected plasticisers in all sediment samples

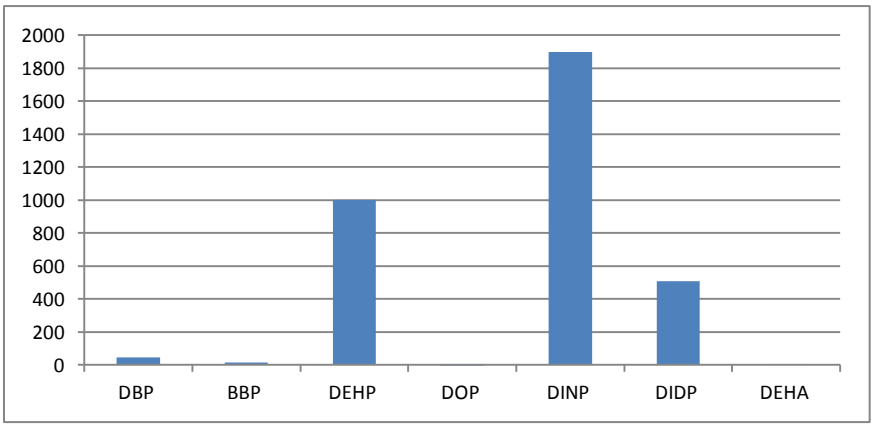
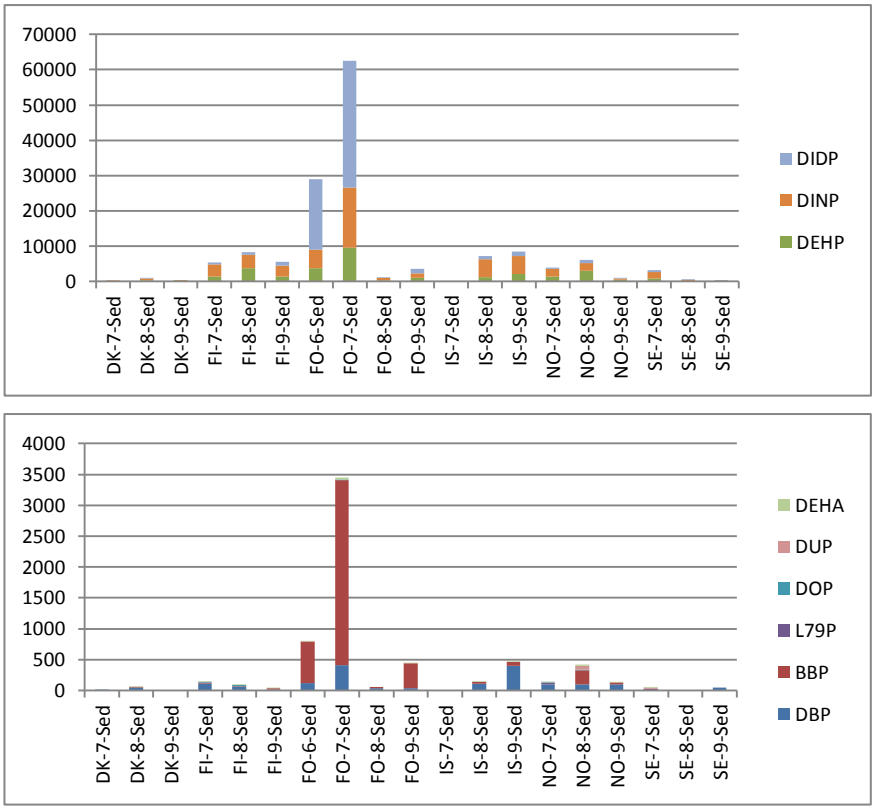
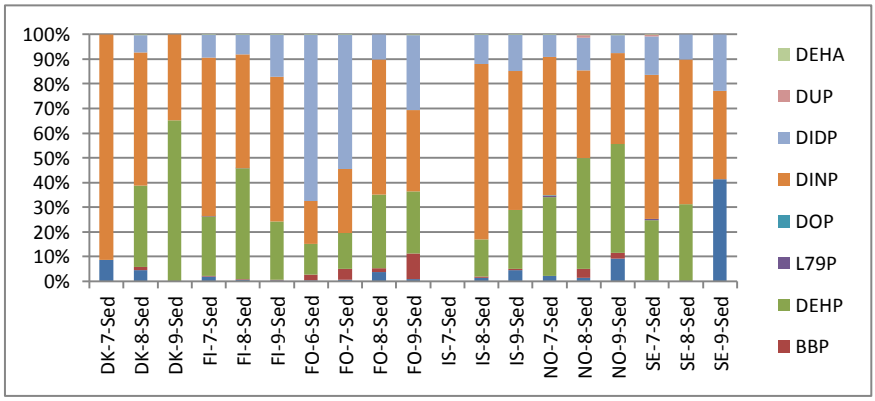


Figure 4.7 Concentration (µg/kg dw) of plasticisers in individual sediment samples. Above: DEHP, DINP, DIDP, below: remaining substances, note the different y-axis scales



The concentration of plasticisers in individual sediments are presented in Figure 4.7. Two samples from Faroe Islands (Torshavn harbour) showed the highest total concentrations dominated by DIDP. In general the sediments were dominated by DINP or DEHP (Figure 4.8).

Figure 4.8 Sediment. Relative concentrations of individual plasticisers. For the sample IS-7-Sed all concentrations were < LOQ



The Danish sediments are from locations not directly influenced by WWTPs while the opposite is true for the sediments from Finland. The Finnish sediments all show higher concentrations than the Danish.

The sediments showing the highest concentrations, FO-6-sed and FO-7-sed, are directly influenced by a WWTP.

NO-8-Sed and NO-9-Sed are from the harbor in Bergen (depth 26 and 79 m respectively), NO-7-Sed are 30 km away from Bergen and in a vicinity of a marina (depth 13 m). The sediment from the greatest depth showed the lowest concentrations which may reflect a higher degree of degradation in the older (deeper) sediments.

The sediments SE-7-Sed and SE-8-Sed are on increasing distance from the mouth of Göta River, the sediment SE-9-Sed is from a background location on the Swedish West Coast. This series of sediments showed clearly decreasing plasticiser concentrations when moving from urban to background areas.

4.1.3 Biota

Fish muscle

Four out of 13 plasticisers, all phthalates, were detected in fish muscle. The concentrations were close to LOQ making the quantification uncertain (Table 4.7).

Table 4.7: Fish muscle (n=16). Percentage of samples with a concentration above LOQ (DF), minimum, median and maximum concentration (µg/kg ww). Substances with DF=0 are not shown

	Det freq	Conc, min	Conc, median	Conc, max
DBP	50%	<4	0.1	30
DEHP	75%	<4	7.7	53
DINP	25%	<40	<40	290
DIDP	13%	<40	<40	410

Concentrations in individual fish muscle samples are illustrated in Figure 4.9 and Figure 4.10.

Figure 4.9 Concentration (µg/kg ww) of plasticisers in individual samples of fish muscle. IS-11-FIS may be contaminated, see text

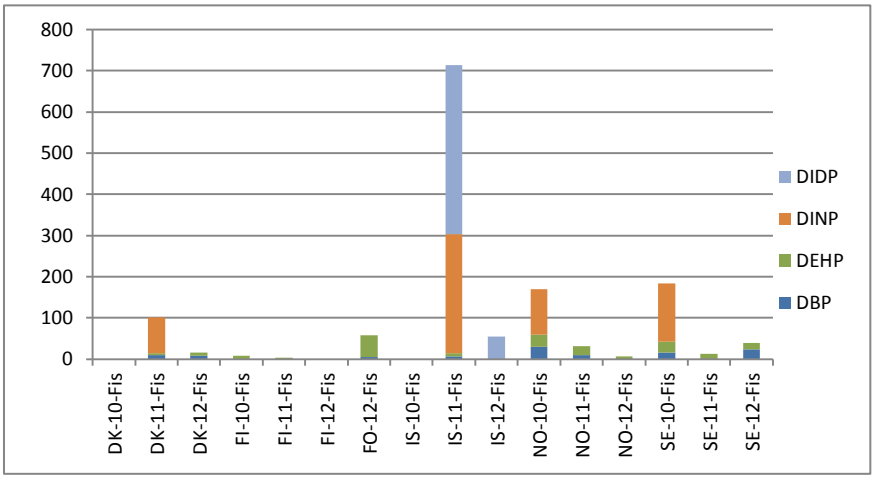
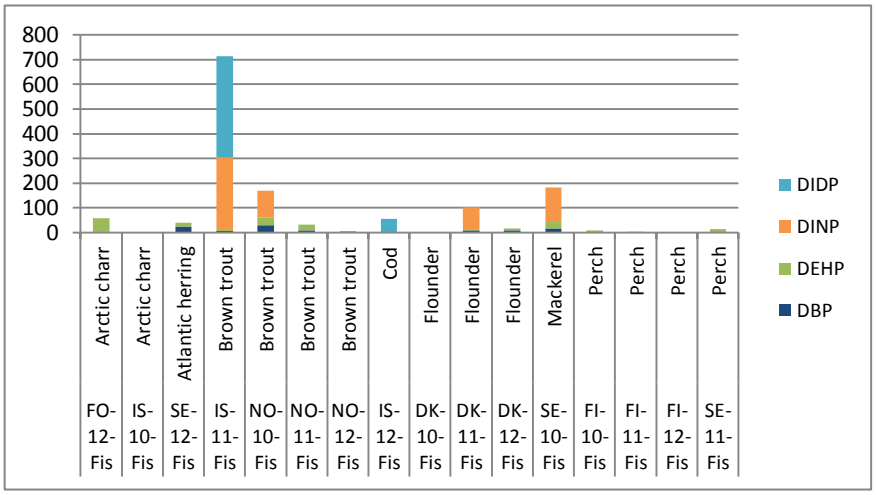


Figure 4.10 Concentration (µg/kg ww) of plasticisers in individual samples of fish muscle sorted according to species. IS-11-FIS may be contaminated, see text



The samples FO-12-Fis, FI-11-Fis, IS-12-Fis and SE-11-Fis represents remote areas without direct influence from discharges.

One sample, IS-11-Fis (Brown trout), showed somewhat elevated concentrations of DINP and DIDP compared to IS-10-Fis (Arctic charr) which was caught in the same lake (Þingvallavatn). Sample IS-11-Fis was delivered in a glass jar with a lid containing a gasket that proved to contain DINP and DIDP (see text following Table 3.6), sample IS-10-Fis (Arctic char), was delivered as whole fish and prepared at the analytical laboratory and stored in heat cleaned glass jars closed with aluminium foil lined lids without gasket (prepared in the analytical laboratory) until analysis. One possible explanation for the difference in results may be contamination from the gasket.

The results for DEHP agree with Swedish measurements 2004. In that investigation DEHP (but no other plasticiser) was analysed in fish muscle, mostly from perch *(Percha fluviatilis)*, collected from 15 reference lakes i

Sweden. Most of the samples contained <8 µg/kg ww of DEHP but in a few samples slightly higher concentrations were found (Sternbeck et al., 2004).

The low concentrations in fish muscle may be the result of the low water solubility of the plasticisers and the metabolism in fish (Albro et al., 1989; Barron et al., 1995; Silva et al., 2004).

Fish liver

Two out of 13 plasticisers, both phthalates (DBP and DEHP), were detected in fish liver. Five samples were analysed, DEHP was detected in all five, DBP i two (Table 4.8).

Table 4.8: Fish liver (n=5). Percentage of samples with a concentration above LOQ (DF), minimum, median and maximum concentration (µg/kg ww). Substances with DF=0 are not shown

	Det freq	Conc, min	Conc, median	Conc, max
DBP	40%	<4	<4	130
DEHP	100%	17	26	92

The samples were all liver from cod caught 30 km off the Faroe Island coast (background area). Individual results are given in Table 4.9.

Table 4.9: Fish liver. Concentration (µg/kg ww) of plasticisers in individual samples of fish liver

	Specie	DBP	DEHP
FO-15-Fis	Cod, Gadus morhua	130	92
FO-16-Fis	Cod, Gadus morhua	4.3	26
FO-17-Fis	Cod, Gadus morhua	<4	40
FO-18-Fis	Cod, Gadus morhua	<4	17
FO-19-Fis	Cod, Gadus morhua	<4	26

The concentrations of DEHP and DBP in liver were somewhat higher than in fish muscle (Table 4.7).

Bird eggs

Two out of 13 plasticisers, both phthalates (DBP and DINP) were detected in bird eggs. Four samples were analysed, DBP was detected in two, DINP in two (Table 4.10). The concentrations were close to LOQ making the quantification uncertain.

Table 4.10: Bird eggs (n=4). Percentage of samples with a concentration above LOQ (DF), minimum, median and maximum concentration (µg/kg ww). Substances with DF=0 are not shown

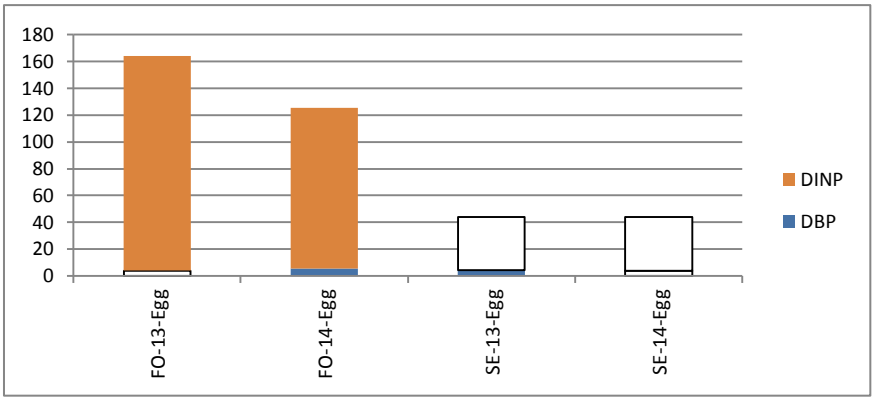
	Det freq	Conc, min	Conc, median	Conc, max
DBP	50%	4.2	4.8	5.3
DINP	50%	<40	40	160

The investigation included four guillemot egg mixtures (mixture of five egg), two from Sweden (*Uria aalge*) and two from Faroe Islands (*Cephus grylle*). Individual results are given in Table 4.11 and Figure 4.11.

Table 4.11: Bird eggs. Concentration (µg/kg ww) of plasticisers in individual samples of bird eggs

	Specie	DBP	DINP
FO-13-Egg	Black guillemot *Cepphus grylle*	<4	160
FO-14-Egg	Black guillemot *Cepphus grylle*	5.3	120
SE-13-Egg	Common Guillemot, *Uria aalge*	4.2	<40
SE-14-Egg	Common Guillemot, *Uria aalge*	<4	<40

Figure 4.11 Concentration (µg/kg ww) of plasticisers in individual samples of bird eggs. Unfilled bars indicate that the substance was not found; the heights of the bars indicate LOQ



In the two samples from Stora Karlsö, Sweden, the only plasticiser found was DBP in one of the samples at a close to LOQ concentration. The eggs from Faroe Islands were collected at a remote area. The concentration of DBP was in the same range as in the Swedish eggs but also DINP was found. The samples from Faroe Islands were stored in the type of jars having a gasket potentially containing DINP and DIDP (see text following Table 3.6). Therefore, contamination from the sample container could not be ruled out.

4.2 Initial ecotoxicological assessment

As an initial ecotoxicological assessment the measured concentrations in effluents and sediment were compared with PNEC values. This was made for the substances frequently found in these matrices. In addition to the PNECs presented in Table 2. tentative PNECs were derived for DUP and DEHA based on the ecotoxicological data presented by Lambert et al. (2010) and the assessment factor methodology for the $PNEC_{water}$ and the equilibrium partition methodology (EqP) for the $PNEC_{sediment}$ (ECHA 2008; ECHA 2010), see Table 4.12. For DOP and L79P there were not sufficient data available for PNEC derivation.

Table 4.12: Tentative predicted no effect concentrations (PNEC) estimated for DUP and DEHA, based on data presented in Lambert et al. (2010). No consideration was taken to potential differences between freshwater and marine water species. No in depth review of the data was possible within the scope of the present study, which means that the PNECs presented here are only rough estimations

Substance	PNEC	Comment
DUP	$PNEC_{freshwater}$ ≈ 2 µg/l	PNEC based on the acute NOEC (96 h, *Cyprinodon variegatus*) 0.22 mg/l as it was lower than the chronic NOECs, and AF 100 as data from two chronic studies was available but not covering the potentially most sensitive species.
	$PNEC_{sediment}$ ≈ 3000 mg/kg dw	PNEC freshwater sediment derived with the EqP method and a K_{oc} of 14,110,000 l/kg as predicted with the EPI Suite 4.10 software. An AF of 10 may be added to consider ingestion as an exposure route.
DEHA	$PNEC_{freshwater}$ ≈ 0.5 µg/l	PNEC based on MATC* (21 d, *Daphnia magna*) = 0.024–0.052 mg/l and AF 50 as data from two chronic studies were available.
	$PNEC_{sediment}$ ≈ 0.7 mg/kg dw	PNEC derived with the EqP method and a K_{oc} of 15,488 l/kg listed as an experimental value in the EPI Suite 4.10 software. An AF of 10 may be added to consider ingestion as an exposure route.

* MATC= maximum acceptable concentration, i.e. an AF may already have been applied

DBP and BBP and DEHA were all found frequently in effluent waters. The maximum concentration of DEHA found in the effluent waters did exceed the PNEC. WWTP effluents will however be diluted after discharge. Also DEHP, DINP and DIDP were frequently detected but these substances are not expected to cause negative effects due to exposure via water (see Chapter 0). The DEHP environmental quality standard (AA-EQS) of 1.3 µg/l (EU Directive 2008/105/EC) was however exceeded in a number of samples, in one sample also after the dilution factor of 10 was applied. The EQS was set to prevent secondary poisoning of predators (DEHP Substance data sheet 2005), which makes this an indication of a risk for the aquatic ecosystem and not only the pelagic community. For DOP no assessment was possible due to lack of data but as the maximum concentration measured in the effluents (59 ng/l) was not exceeding the concentrations measured for the other substances, there is thus no strong indication of risk but a substance specific PNEC is needed for a proper assessment.

All substances included in the screening, with the exception of BOA, DINA, DEHZ and DBEEA, were more or less frequently found in sediments. Comparisons of the maximum levels found with the PNEC for freshwater show that for DBP, DEHP, DUP, DEHA and BBP[1] the measured concentration did not exceed the PNEC. Adding a safety factor of 10

[1] $PNEC_{sediment}$ on wet weight basis converted to a dry weight concentration with ECHA standard values ((RHOsolid 2500 kg/m³*Fsolid susp 0.1 m³/m³)/RHO susp 1150 kg/m³) (ECHA 2010)

to the PNEC to also consider ingestion (for PNEC derived with EqP) showed that DBP and BBP occurred in concentrations above the PNEC. Adding further safety factors to consider the marine environment (ECHA 2008) showed that maximum concentrations of DBP, BBP and DEHA all exceeded the PNEC. It should also be noted that DEHP concentrations (maximum 9.5 mg/kg dw, median 1.0 mg/kg dw) were in levels which may affect microbial processes (see Chapter 0). DINP and DIDP were also frequently found in sediments but PNECs are not available and no negative effects are expected (see Chapter 0). For L79P and DOP no assessment is possible due to lack of data.

DBP, DEHP, DINP and DIDP were found in one or more biota sample. DBP and DEHP have been found to be toxic, DINP and DIDP to be potentially toxic, over prolonged exposure (Lambert et al. 2010). The fact that these substances were found in biota may thus be of concern but it is out of scope in the present study to assess whether the measured concentrations are in levels that can cause negative effects.

5. Conclusions, plasticisers

The majority of plasticisers included in the screening were frequently found in effluents from WWTPs in all the Nordic countries. The phthalates DEHP, DINP and DIDP were quantitatively dominating. The individual concentrations were generally in the range up to 6,000 ng/l, but in effluents from Faroe Islands concentrations up to 27,000 ng/l were measured.

The phthalates DBP and BBP and the adipate DEHA, were also frequently detected in effluents but in lower concentrations . This indicates that municipal sewage systems can be an important pathway for these substances to the environment

Almost all (11 of 13) plasticisers analysed for were found in WWTP sludge. The compounds which occurred in the highest concentrations were DINP, DEHP and DIDP. The individual compounds were found approximately in proportion to their current use and earlier use patterns.

Although the number of samples were too small to draw extensive conclusions on differences between the countries it is striking that the highest concentrations in effluents and sediments were found in samples from Faroe Islands, where also low concentrations in sludge could indicate an inefficient treatment plant process.

Many of the substances were found in sediments. Higher concentrations were generally found in samples from potentially affected areas compared to background areas. The relative concentration of substances in sediments showed a similar pattern to what was found for sludge.

The phthalates DBP, DEHP, DINP and DIDP were detected in one or more biological sample in concentrations close to LOQ. Adipates were not detected at all.

The low concentrations observed in fish may be a consequence of the low water solubility (low exposure) of the most common plasticisers (Table 2.2). Moreover, the ester bonds in the plasticisers are readily hydrolysed (Albro et al., 1989; Barron et al., 1995; Silva et al. 2004) by different enzymes (esterase and lipase) (Shintani 2000; Suzuki et al. 2001, Kato et al. 2003) which means that compounds containing ester bonds have short half-life in organisms.

Some of the substances included in the screening may be harmful to the environment. The fact that the concentrations found in effluents and sediments were close to or exceeded PNEC in several cases indicate the need for further studies to assess potential risks. Furthermore, some of the substances were also found in biota.

As already mentioned, all stages of the determination process must be carefully checked for possible contamination. This is underlined by the discussion on possible contamination of liver and egg samples in this work. To avoid such a pitfall, blank controls of the sampling equipment must be performed before starting collection of samples for analysis, and this equipment only should be used for all sampling and sample storage. The sampling and preparation environment may be controlled by "sampling blanks" as was used in this study. Also, the analytical method was carefully checked for possible contamination. This implies control of equipment, solvents and procedure (analytical blank samples).

6. Results, sweeteners

The measured concentrations of sweeteners in individual samples are tabulated in Appendix 6.

Effluents

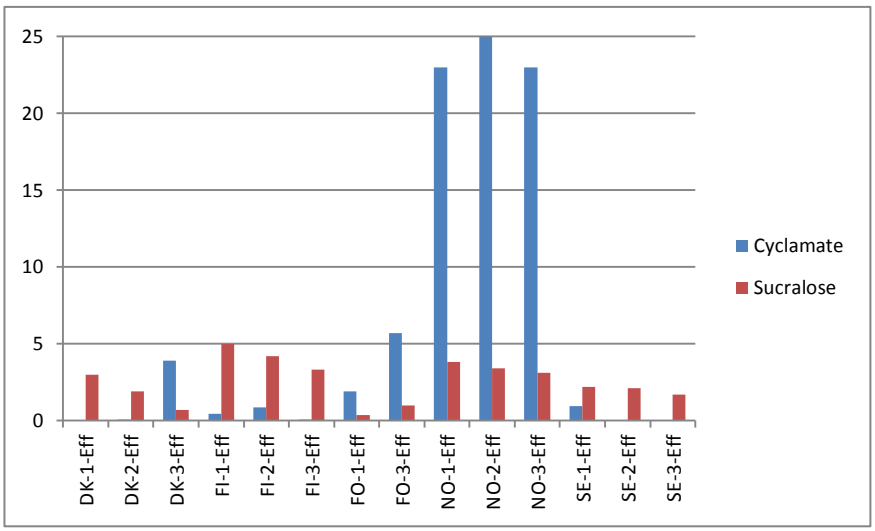
An overview of the results for effluents, where detection frequencies, minimum, median and maximum concentration are shown, is given in Table 6.1.

Table 6.1: Effluents (n=14). Percentage of samples with a concentration above LOQ (DF), minimum, median and maximum concentration (µg/l)

	DF	Conc, min	Conc, median	Conc, max
Aspartam	0%		<0.08	
Cyclamate	86%	<0.01	0.91	25
Sucralose	100%	0.37	2.6	5

Sucralose was most frequently detected followed by cyclamate, while the concentrations of aspartam were below the LOQ in all samples. The concentrations of cyclamate and sucralose in the individual effluents are shown inFigure 6.1.

Figure 6.1 Concentration (µg/l) of cyclamate and sucralose in individual effluents



The highest effluent concentrations of cyclamate (23–25 µg/l) were measured in the Norwegian samples. They were however collected at the same WWTP and during the same day which explains the small variation in concentration among the those samples. The cyclamate concen-

tration in the remaining samples were scattered in the range <0.01–5.7 µg/l. Sucralose spanned almost the same concentration range (0.37–5 µg/l) Ther was no apparent correlation between cyclamate and sucralose concentrations. The highest sucralose concentrations were measured in Finnish samples, the lowest in samples from Faroe Islands and Denmark.

Effluents from the three WWTPs representing Sweden in this investigation were also analysed for sucralose in 2007 (Brorström-Lundén 2008). The results were approximately equal or (in one case) somewhat lower in 2011 than in 2007 (Table 6.2).

Table 6.2: Sucralose in effluent waters (µg/l) sampled in 2007 (Brorström-Lundén 2008) and 2011 (current investigation)

		2007	2011
SE-1-Eff	Öhn WWTP, Umeå	4.0	2.2
SE-2-Eff	Ryaverken WWTP, Göteborg	2.8	2.1
SE-3-Eff	Gässlösa WWTP, Borås	1.8	1.7

Sludge

An overview of the results for sludge, where detection frequencies, minimum, median and maximum concentration are shown, is given in Table 6.3.

Table 6.3: Sludge (n=12). Percentage of samples with a concentration above LOQ (DF), minimum, median and maximum concentration (µg/kg ww)

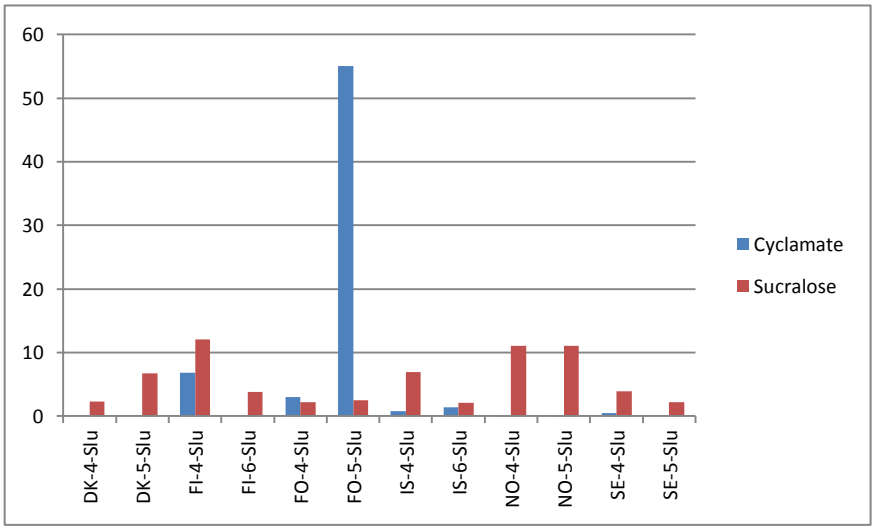
	DF	Conc, min	Conc, median	Conc, max
Aspartam	0%		<1	
Cyclamate	62%	<0.2	0.33	55
Sucralose	92%	2.1	3.9	12

As was the case for effluents, sucralose was most frequently detected followed by cyclamate, while the concentrations of aspartam were below the LOQ in all samples.

The range of results for sludge (on a wet weight basis) do not differ much from the results for effluents (Table 6.1). This indicates that these substances are not preferentially adsorbed to sludge.

The concentrations of cyclamate and sucralose in individual sludge samples are shown in Figure 6.2.

Figure 6.2 Concentration (µg/kg ww) of cyklamate and sucralose in individual sludge samples



The sample FO-5-Slu showed the highest result for cyclamate. This was not a typical sludge; it was inhomogeneous and contained paper like residues which made it difficult to get representative subsamples which could have influenced the result.

The ratio concentration in sludge (on a wet weght basis) to concentration in effluent from the same WWTP ranged 0.01–16 (median 1.3) for cyclamate and 0.8–5.9 (median 2.4) for sucralose. This supports the conclusion that although there is variations in individual results the substances do not have a high affinity to sludge.

A compilation of previous measurements of sweeteners in the aquatic environment can be found in a recent review by Lange et al. (2012).

7. Acknowledgements

7.1 Denmark

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9. Sammanfattning

Det övergripande syftet med föreliggande screeningstudie var att undersöka förekomsten av ett antal mjukgörare i miljörelaterade prover från de nordiska länderna. Proven analyserades på åtta ftalater, fyra adipater och en azelat. Provtyperna var utgående vatten och slam från kommunala reningsverk, sediment och fisk. Vanligtvis bidrog de deltagande länderna (Danmark, Finland, Färöarna, Island, Norge och Sverige) med tre prov av varje provtyp. Ett mindre antal fågelägg inkluderades också. Det relativt låga antalet prov och provtyper är avsedda att ge en ögonblicksbild av situationen.

För all provtyper detekterades ftalaterna DEHP, DINP och DIDP mest frekvent och i högst koncentrationer. DBP och BBP hittades också frekvent men i lägre koncentrationer. Detta gäller också för adipaterna DEHA och BOA, medan DINA och DBEEQ inte hittades alls. Azelaten DEHZ hittades enbart i ett slamprov.

I slam utgjorde DINP, DEHP och DIDP tillsammans 96–99.7% av den totalt uppmätta halten av mjukgörare. Den högsta sammanlagda koncentrationen var 260,000 µg/kg TS. I avloppsvattnen hade DEHP nästan alltid den högsta koncentrationen, men alla slamprov dominerades av DINP.

Sediment från platser i närheten av reningsverk visade högre halter av mjukgörare än sediment från bakgrundsområden.

Koncentrationen i fiskmuskel var generellt låg, under eller nära kvantifieringsgränsen.

Mjukgörarna som ingick i screeningen kan vara skadliga för miljön. Att koncentrationer i effluenter och sediment var nära eller över PNEC-värden indikerar ett behov av ytterligare studier för att utvärdera risken för negativa effekter. Vidare detekterades några av de screenade substanserna även i biota.

Avloppsvattnen och vissa av slamproven analyserades också på sötningsmedel. Resultaten visade att cyclamat och sucralos är vanligt förekommande i avloppsvatten från reningsverk i de nordiska länderna och att dessa substanser inte i någon stor utsträckning ackumuleras i slam.

10. Appendices

10.1 Analysis of selected plasticizers and additional sweeteners in a Nordic cooperation on screening

10.1.1 Sampling manual

Each country are asked to contribute

- 3 WWTP effluent waters
- 3 WWTP sludges
- 3 sediments
- 3 fish samples
- 2 egg samples (only FO, IS, SE)

All samples will be analyzed for plasticizers. The effluent waters and 2 of the sludges will be analyzed for sweeteners.

Equipment provided

- 2.5 L glass bottles (effluent water samples).
- 120 ml glass jars (sludge & sediment for plasticizers)
- 250 ml PE jars (sludge for sweeteners)
- Plastic bags for jars.
- Muffled Al-foil (packed in Al-foil).
- Labels.
- Plastic gloves.
- Sampling protocol.

10.1.2 Planning the sampling

Start by opening the provided "Sampling protocol" in excel format. Fill in data for each planned sample, one sample on each row. Use the suggested indication of matrix type or change among the possible choices on the list. This will give each sample a *Sample identification* in the format *CO-no-Mat* where *CO* indicates country, *no* is a consecutive number and *Mat* indicates matrix. The samples should be labelled with their respective Sample identification.

Example: DK-3-Slu, the third Danish sample which is a sludge sample.

Use a printout of the sample list to record sample date and other relevant observations. The sheet Notes can also be used.

Please give coordinates as LAT and LONG in the WGS84 reference system expressed as degrees and decimal minutes. See example in the

sampling protocol. We strongly suggest to cross-check the registered coordinates with an independent geographic tool like Google Earth or similar. Select all six Excel cells containing the coordinates, copy the content with Ctrl-C and paste it into the "fly to" window in the upper-left corner of Google Earth. By clicking onto the magnifier symbol Google Earth should show you the correct sample position.

10.1.3 Precautions to be taken in advance of sampling to avoid contamination

Cosmetic formulations contain various chemicals that potentially can contaminate the samples. Do not use products such as antiperspirant, eye shadow, hair spray, or skin lotions on the day of sampling. Only specially cleaned sampling containers provided by the laboratory should be used.

To check for contamination sampling blanks are used. The sampling blank to be used for water contains MilliQ-water, the sampling blank to be used for sludge and sediment sampling contains granulated diatomaceous earth containing 10 % water. The sampling blanks should not be emptied or filled. They shall only be opened and closed at the time of sampling.

The number of sampling blanks is limited. Each country are assigned one water blank or one solid blank. The sites used for blank sampling should be selected at random before the start of sampling. Blank samples should be labelled BLW and BLS respectively.

10.1.4 A. Sampling of water

To minimize the contamination risk grab samples are preferred over composite samples.

- Arrange the sampling bottles to be used on a clean spot on the sampling site. Put on the supplied gloves.
- Immediately before sampling open the lid of the sampling container. Make sure that the white PTFE seal is not lost.
- Fill the sample container (2.5 L) and close the lid on the sample bottle.
- Mark bottles with Sample identification.
- Make notes on the sample protocol.
- Store the samples in a refrigerator (5–10 °C). (Both plasticizers and sweeteners will be analysed on the same sample).

B. Sampling of sludge

- Put on the supplied gloves.
- Open the lid of the jar.
- Fill the jar with sludge and close the lid. If the Al-foil protecting the lid of the jar is ruptured replace it with new Al-foil.
- Mark the sample with Sample identification.
- If sweeteners are going to be analysed fill also a plastic jar.
- Put each sample in a plastic bag (2 L).
- Make notes on the sample protocol.
- Store the samples in a freezer (-18 °C) if the sample has low water content (30% or less). Sludges with high water content should not be frozen (risk of breaking the jar).

C. Sampling of sediment

- Put on the supplied gloves.
- Open the lid of the jar.
- Fill the jar with sediment and close the lid. If the Al-foil protecting the lid of the jar is ruptured replace it with new Al-foil.
- Mark the sample with Sample identification.
- Put each sample in a plastic bag (2 L).
- Make notes on the sample protocol.
- Store the samples in a freezer (-18 °C).

D. Sampling of fish

The number of fish per sample should be 5–10 or more depending on weight.

The quantity should be sufficient to prepare a composite sample of at least 50 g fish muscle.

- Put on the supplied gloves.
- Wrap each fish individually in Al-foil provided.
- Put the Al-foil packages making up one sample in one common plastic bag, or, if needed, mark additional bags accordingly.
- Mark the samples with Sample identification.
- Make notes on the sample protocol.
- Store the samples in a freezer (-18 °C).

10.1.5 Storage and transport

Send all samples to IVL in Stockholm as soon as possible in such a way that the samples will reach the laboratory within one day (DHL or equivalent courier service). Send samples in the same containers used for providing sampling material. Make sure to insulate glass bottles properly to avoid breakage during transport. Include a printout of the Sampling protocol. Send the samples to the address below.

When sending the samples please send also an e-mail with the filled in Sample protocol attached to Lennart Kaj (lennar.kaj@ivl.se) and Mikael Remberger (mikael.remberger@ivl.se).

Address

IVL Swedish Environmental Research Institute
Lennart Kaj
Valhallavägen 81
SE-114 27 Stockholm
Sweden.
Tel +46 (0)8-598 563 00

10.2 Appendix 2 Sample list, plasticisers

MR#	Sample ident.	Location	Site	Species etc	Sampling date	LAT WGS84 Deg., decimal min.			LONG WGS84 Deg., decimal min		
9517	DK-1-Eff	Esbjerg	Esbjerg central WWTP		2011-11-02	55	29.223	N	8	25.491	E
9492	DK-2-Eff	Odense	Ejby Mølle WWTP		2011-10-24	55	23.576	N	10	25.139	E
9491	DK-3-Eff	Vordingborg	Råbylille strand WWTP		2011-10-11	54	58.196	N	12	23.697	E
9518	DK-4-Slu	Esbjerg	Esbjerg central WWTP		2011-11-02	55	29.223	N	8	25.491	E
9494	DK-5-Slu	Odense	Ejby Mølle WWTP		2011-10-24	55	23.573	N	10	24.499	E
9728	DK-7-Sed	Øresund	Vedbæk		2010-12-26	55	48	N	12	35.65	E
9729	DK-8-Sed	Kolding Fjord			2011-11-09	55	30.11	N	9	37.46	E
9730	DK-9-Sed	Limfjorden			2011-11-16	56	39.4	N	8	42.32	E
9731	DK-10-Fis	Ho bugt	Wadden Sea	Flounder, *Platichthys flesus,* muscle	2011-09-19	55	35.16	N	8	18.42	E
9732	DK-11-Fis	Hjelm bugt	Baltic Sea	Flounder, *Platichthys flesus,* muscle	2011-11-15	54	56.36	N	12	25.7	E
9733	DK-12-Fis	Agersø	Great Belt	Flounder, *Platichthys flesus,* muscle	2011-09-27	55	15.67	N	11	10.36	E
9544	FI-1-Eff	Turku	Kakolanmäki WWTP		2011-11-10	60	26.712	N	22	14.204	E
9545	FI-2-Eff	Helsinki	Viikki WWTP		2011-11-14	60	13.544	N	24	59.612	E
9546	FI-3-Eff	Tampere	Viinikanlahti WWTP		2011-11-14	61	29.319	N	23	46.043	E
9644	FI-4-Slu	Turku	Kakolanmäki WWTP		2011-11-10	60	26.712	N	22	14.204	E
9645	FI-5-Slu	Helsinki	Viikki WWTP		2011-11-14	60	13.544	N	24	59.612	E
9646	FI-6-Slu	Tampere	Viinikanlahti WWTP		2011-11-14	61	29.319	N	23	46.043	E
9647	FI-7-Sed	Turku	Turku harbour		2011-11-14	60	26.286	N	22	13.050	E
9648	FI-8-Sed	Tampere	Viinikanlahti		2011-11-01	61	29.374	N	23	45.808	E
9649	FI-9-Sed	Helsinki	Vanhankaupunginlahti		2011-11-11	60	11.550	N	24	59.575	E
9650	FI-10-Fis	Tampere	Pirkkalan Pyhäjärvi	Perch, *Perca fluviatilis,* muscle	2011-10-25	61	28.943	N	23	39.272	E
9651	FI-11-Fis	Kuhmoinen	Päijänne Tehinselkä	Perch, *Perca fluviatilis,* muscle	2011-10-28	61	31.670	N	25	21.900	E
9652	FI-12-Fis	Turku Archipelago	Airisto Seili	Perch, *Perca fluviatilis,* muscle	2010-09-23	60	13.783	N	21	57.053	E
9629	FO-1-Eff	Torshavn	Sersjantvikin WWTP	Effluent	2011-11-21	62	0.49	N	6	45.717	W
9630	FO-2-Inf	Torshavn	Main Hospital WWTP	Influent!	2011-11-21	62	0.098	N	6	46.538	W
9631	FO-3-Eff	Klaksvik	Klaksvik Hospital WWTP	Effluent	2011-11-21	62	13.5	N	6	35.47	W
9632	FO-4-Slu	Torshavn	Sersjantvikin WWTP	sludge	2011-11-21	62	0.49	N	6	45.717	W
9633	FO-5-Slu	Torshavn	Main Hospital WWTP	sludge	2011-11-21	62	0.098	N	6	46.538	W
9634	FO-6-Sed	Torshavn	Harbour, near shipyard, Stn BA	sediment, marine	2011-11-21	62	0.430	N	6	46.439	W
9635	FO-7-Sed	Torshavn	Harbour, near marina	sediment, marine	2011-11-21	62	0.31	N	6	46.220	W
9636	FO-8-Sed	Kollafjord	Station 6	sediment, marine	2011-11-11	62	6.064	N	6	55.519	W
9637	FO-9-Sed	Klaksvik	Harbour, á Stongum	sediment, marine	2011-11-19	62	13.835	N	6	35.476	W
9639	FO-10-Fis	NW Faroe shelf	Mýlingsgrunnur, background area	Cod, *Gadus morhua,* liver	2011-10-01	62	25	N	7	25	W
9640	FO-11-Fis	NW Faroe shelf	Mýlingsgrunnur, background	Cod, *Gadus morhua,* liver	2011-10-01	62	25	N	7	25	W
9641	FO-12-Fis	Lake á Mýrunum	Freshwater lake, background	Arctic charr, *Salvelinus alpinus,* muscle	2011 June/July	62	9.83	N	7	5.48	W
9642	FO-13-Egg	Skúvoy	Small island, background area	Black guillemot *Cepphus grylle,* eggs	2010 June	61	46.1	N	6	48.1	W
9643	FO-14-Egg	Koltur	Small island, background area	Black guillemot *Cepphus grylle*, eggs	2010-06-04	62	0.0	N	7	0.0	W
1384-1	FO-15-Fis	NW on Faroe shelf	Mýlingsgrunnur, background area	Cod, *Gadus morhua,* liver	2012-05-16	62	25	N	7	25	W
1384_2	FO-16-Fis	NW on Faroe shelf	Mýlingsgrunnur, background area	Cod, *Gadus morhua,* liver	2012-05-16	62	25	N	7	25	W
1384_3	FO-17-Fis	NW on Faroe shelf	Mýlingsgrunnur, background area	Cod, *Gadus morhua,* liver	2012-05-16	62	25	N	7	25	W
1384_4	FO-18-Fis	NW on Faroe shelf	Mýlingsgrunnur, background area	Cod, *Gadus morhua,* liver	2012-05-16	62	25	N	7	25	W
1384_5	FO-19-Fis	NW on Faroe shelf	Mýlingsgrunnur, background area	Cod, *Gadus morhua,* liver	2012-05-16	62	25	N	7	25	W
9533	IS-4-Slu	Hveragerði	WWTP		2011-10-24	64	59.295	N	21	10.638	W

MR#	Sample ident.	Location	Site	Species etc	Sampling date	LAT WGS84 Deg., decimal min.			LONG WGS84 Deg., decimal min		
9534	IS-5-Slu	Borg - Grímsnesi	WWTP		2011-10-24	64	04.29	N	20	46.09	W
9535	IS-6-Slu	Klettagarðar - Reyk.	WWTP		2011-11-02	64	9.324	N	21	52.405	W
9536	IS-7-Sed	Þingvallavatn			2011-10-11	64	11.52	N	21	8.6	W
9537	IS-8-Sed	Grafarvogur - Reyk.	Gullinbrú		2011-10-27	64	8.028	N	21	48.879	W
9538	IS-9-Sed	Naustavogur - Reyk.	Háubakkar		2011-10-28	64	7.814	N	21	50.698	W
9539	IS-10-Fis	Þingvallavatn		Arctic charr, *Salvelinus alpinus,* muscle	2011-10-11	64	11.52	N	21	8.6	W
9540	IS-11-Fis	Þingvallavatn		Brown trout, *Salmo trutta,* muscle	2009, summer	64	11.52	N	21	8.6	W
9541	IS-12-Fis	Iceland Seas	NNV - mið	Cod, *Gadus morhua,* muscle	2010-03-10	67	2.55	N	23	29.93	W
9495	NO-1-Eff	Gjøvik	Rambekk WWTP		2011-10-25	60	46.35	N	10	42.21	E
9496	NO-2-Eff	Gjøvik	Rambekk WWTP		2011-10-25	60	46.35	N	10	42.21	E
9497	NO-3-Eff	Gjøvik	Rambekk WWTP		2011-10-25	60	46.35	N	10	42.21	E
9499	NO-4-Slu	Gjøvik	Rambekk WWTP		2011-10-25	60	46.35	N	10	42.21	E
9500	NO-5-Slu	Gjøvik	Rambekk WWTP		2011-10-25	60	46.35	N	10	42.21	E
9501	NO-6-Slu	Gjøvik	Rambekk WWTP		2011-10-25	60	46.35	N	10	42.21	E
9604	NO-7-Sed	Bergen	Kviturspollen	depth: 13.1 m	2002-11-11	60	15.773	N	5	15.223	E
9605	NO-8-Sed	Bergen	Puddefjorden	depth: 26 m	2005-10-11	60	23.267	N	5	18.473	E
9606	NO-9-Sed	Bergen	Puddefjorden	depth: 79 m	2005-10-11	60	23.725	N	5	17.079	E
9657	NO-10-Fis	Vingrom	Mjøsa	Brown trout, *Salmo trutta,* muscle	2011-08-01	61	2.67	N	10	27.1	E
9658	NO-11-Fis	Vingrom	Mjøsa	Brown trout, *Salmo trutta,* muscle	2011-08-01	61	2.67	N	10	27.1	E
9659	NO-12-Fis	Vingrom	Mjøsa	Brown trout, *Salmo trutta,* muscle	2011-08-01	61	2.67	N	10	27.1	E
9628	SE-1-Eff	Umeå	Öhn WWTP		2011-11-21	63	48.25	N	20	17.5	E
9597	SE-2-Eff	Göteborg	Ryaverken WWTP		2011-11-10	57	41.83	N	11	53.5	E
9598	SE-3-Eff	Borås	Gässlösa WWTP		2011-11-16	57	42.3	N	12	55.5	E
9532	SE-4-Slu	Umeå	Öhn WWTP		2011-11-08	63	48.25	N	20	17.5	E
9599	SE-5-Slu	Göteborg	Ryaverken WWTP		2011-11-10	57	41.83	N	11	53.5	E
9600	SE-6-Slu	Borås	Gässlösa WWTP		2011-11-16	57	42.3	N	12	55.5	E
9601	SE-7-Sed	Göteborg	Stockholmen		2011-10-17	57	40.426	N	11	49.531	E
9602	SE-8-Sed	Göteborg	Björkö		2011-10-20	57	43.469	N	11	41.407	E
9593	SE-9-Sed	Strömstad	Kosterfjorden	depth: 95 m	2011-05-11	58	52.069	N	11	6.649	E
9614	SE-10-Fis	Göteborg	Hakefjorden	Mackerel, *Scomber scombrus,* muscle	2011-10-26	57	40	N	11	45	E
1108	SE-11-Fis	Holmöarna	Golf of Bothnia	Perch, *Perca fluviatilis*, muscle	2011-08-16	63	40.834	N	20	52.618	E
1109	SE-12-Fis	Kullen	Kattegat	Atlantic herring, *Clupea harengus*, muscle	2011-10-06	56	19.497	N	12	22.855	E
1112	SE-13-Egg	St.Karlsö	Baltic Sea	Common Guillemot, *Uria aalge*, egg	2011-05-06	57	16.975	N	17	58.462	E
1113	SE-14-Egg	St.Karlsö	Baltic Sea	Common Guillemot, *Uria aalge*, egg	2011-05-06	57	16.975	N	17	58.462	E

Sample ident. suffix: Eff=Effluent, Inf=influent, Slu=Sludge, Sed=Sediment, Fis=Fish, Egg=Eggs

10.3 Appendix 3 Individual results, plasticisers

MR#	Sample ident.	Location	Unit	DBP	BBP	DEHP	L79P	DOP	DINP	DIDP	DUP	DEHA	DINA	BOA	DBEEA	DEHZ
9517	DK-1-Eff	Esbjerg	ng/l	110	110	1,100	270	<20	160	<100	<20	39	<20	<130	<150	<15
9492	DK-2-Eff	Odense	ng/l	140	29	800	<50	<20	<80	<100	<20	<25	<20	<130	<150	<15
9491	DK-3-Eff	Vordingborg	ng/l	<50	20	<200	<50	<20	<80	<100	<20	<25	<20	<130	<150	<15
9518	DK-4-Slu	Esbjerg	µg/kg dw	35	160	18,000	<8	23	50,000	9,900	<20	19	<8	<200	<100	<10
9494	DK-5-Slu	Odense	µg/kg dw	80	190	17,000	68	59	49,000	14,000	<20	30	<8	<200	<100	<10
9728	DK-7-Sed	Øresund	µg/kg dw	8.6	<4	<80	<8	<5	92	<20	<10	<1	<10	<50	<50	<5
9729	DK-8-Sed	Kolding Fjord	µg/kg dw	41	12	300	<8	<5	490	63	<10	3	<10	<50	<50	<5
9730	DK-9-Sed	Limfjorden	µg/kg dw	<8	<4	110	<8	<5	59	<20	<10	<1	<10	<50	<50	<5
9731	DK-10-Fis	Ho bugt	µg/kg ww	<4	<2	<4	<2	<2	<40	<40	<6	<30	<8	<40	<5	<2
9732	DK-11-Fis	Hjelm bugt	µg/kg ww	9.3	<2	5.5	<2	<2	87	<40	<6	<30	<8	<40	<5	<2
9733	DK-12-Fis	Agersø	µg/kg ww	7.4	<2	9.2	<2	<2	<40	<40	<6	<30	<8	<40	<5	<2
9544	FI-1-Eff	Turku	ng/l	74	64	720	<50	22	120	<100	<20	54	<20	<130	<150	<15
9545	FI-2-Eff	Helsinki	ng/l	300	170	2200	<50	26	450	220	<20	43	<20	<130	<150	<15
9546	FI-3-Eff	Tampere	ng/l	79	90	2400	<50	22	350	110	<20	120	<20	<130	<150	<15
9644	FI-4-Slu	Turku	µg/kg dw	330	1,100	67,000	370	220	160,000	32,000	<20	27	<8	<200	<100	<10
9645	FI-5-Slu	Helsinki	µg/kg dw	140	190	33,000	210	150	97,000	25,000	<20	<10	<8	<200	<100	<10
9646	FI-6-Slu	Tampere	µg/kg dw	86	610	36,000	<8	53	52,000	4,900	<20	180	<8	<200	<100	<10
9647	FI-7-Sed	Turku	µg/kg dw	110	7.6	1300	15	8.2	3500	510	<10	1.8	<10	<50	<50	<5
9648	FI-8-Sed	Tampere	µg/kg dw	54	17	3700	<8	12	3800	660	<10	5	<10	<50	<50	<5
9649	FI-9-Sed	Helsinki	µg/kg dw	15	16	1300	<8	<5	3200	940	<10	2.6	<10	<50	<50	<5
9650	FI-10-Fis	Tampere	µg/kg ww	<4	<2	7.4	<2	<2	<40	<40	<6	<30	<8	<40	<5	<2
9651	FI-11-Fis	Kuhmoinen	µg/kg ww	<4	<2	4.1	<2	<2	<40	<40	<6	<30	<8	<40	<5	<2
9652	FI-12-Fis	Turku Archipelago	µg/kg ww	<4	<2	<4	<2	<2	<40	<40	<6	<30	<8	<40	<5	<2
9629	FO-1-Eff	Torshavn	ng/l	140	140	12,000	<50	41	27,000	4,000	<20	49	120	<130	<150	<15
9630	FO-2- Inf	Torshavn	ng/l	180	130	62,000	<50	<20	3100	1,500	<20	<25	<20	<130	<150	<15
9631	FO-3-Eff	Klaksvik	ng/l	200	99	15,000	<50	39	8100	1,400	<20	44	270	<130	<150	<15
9632	FO-4-Slu	Torshavn	µg/kg dw	130	270	14,000	<8	62	96,000	8,600	<20	64	<8	<200	<100	<10
9633	FO-5-Slu	Torshavn	µg/kg dw	110	160	12,000	<8	<10	17,000	1,800	<20	28	<8	<200	<100	<10
9634	FO-6-Sed	Torshavn	µg/kg dw	120	670	3,700	<8	<5	5200	20,000	<10	11	<10	<50	<50	<5
9635	FO-7-Sed	Torshavn	µg/kg dw	410	3,000	9,500	<8	11	17,000	36,000	<10	26	<10	<50	<50	<5
9636	FO-8-Sed	Kollafjord	µg/kg dw	40	17	320	<8	<5	590	110	<10	<1	<10	<50	<50	<5
9637	FO-9-Sed	Klaksvik	µg/kg dw	31	410	1,000	<8	<5	1300	1,200	<10	13	<10	<50	<50	<5
9641	FO-12-Fis	Lake á Mýrunum	µg/kg ww	4.2	<2	53	<2	<2	<40	<40	<6	<30	<8	<40	<5	<2
9642	FO-13-Egg	Skúvoy	µg/kg ww	<4	<2	<4	<2	<2	160	<40	<6	<30	<8	<40	<5	<2
9643	FO-14-Egg	Koltur	µg/kg ww	5.3	<2	<4	<2	<2	120	<40	<6	<30	<8	<40	<5	<2
1384:1	FO-15-Fis	NW on Faroe shelf	µg/kg ww	130	<2	92	<2	<2	<40	<40	<6	<30	<8	<10	<5	<2
1384:2	FO-16-Fis	NW on Faroe shelf	µg/kg ww	4.3	<2	26	<2	<2	<40	<40	<6	<30	<8	<10	<5	<2
1384:3	FO-17-Fis	NW on Faroe shelf	µg/kg ww	<4	<2	40	<2	<2	<40	<40	<6	<30	<8	<10	<5	<2
1384:4	FO-18-Fis	NW on Faroe shelf	µg/kg ww	<4	<2	17	<2	<2	<40	<40	<6	<30	<8	<10	<5	<2
1384:5	FO-19-Fis	NW on Faroe shelf	µg/kg ww	<4	<2	26	<2	<2	<40	<40	<6	<30	<8	<10	<5	<2
9533	IS-4-Slu	Hveragerði	µg/kg dw	84	210	21,000	130	60	98,000	19,000	<20	31	400	<200	<100	<10
9534	IS-5-Slu	Borg - Grímsnesi	µg/kg dw	280	340	16,000	<8	<10	130,000	12,000	<20	180	<8	<200	<100	<10
9535	IS-6-Slu	Klettagarðar - Reyk.	µg/kg dw	610	630	16,000	<8	48	69,000	6,700	230	970	1,100	<200	<100	32
9536	IS-7-Sed	Þingvallavatn	µg/kg dw	<8	<4	<80	<8	<5	<30	<20	<10	<1	<10	<50	<50	<5

MR#	Sample ident.	Location	Unit	DBP	BBP	DEHP	L79P	DOP	DINP	DIDP	DUP	DEHA	DINA	BOA	DBEEA	DEHZ
9537	IS-8-Sed	Grafarvogur - Reyk.	µg/kg dw	110	25	1,100	<8	<5	5,200	880	<10	1.8	<10	<50	<50	<5
9538	IS-9-Sed	Naustavogur - Reyk.	µg/kg dw	400	56	2,100	<8	<5	5,000	1,300	<10	4.5	<10	<50	<50	<5
9539	IS-10-Fis	Þingvallavatn	µg/kg ww	<4	<2	<4	<2	<2	<40	<40	<6	<30	<8	<40	<5	<2
9540	IS-11-Fis	Þingvallavatn	µg/kg ww	6.3	<2	7.9	<2	<2	290	410	<6	<30	<8	<40	<5	<2
9541	IS-12-Fis	Iceland Seas	µg/kg ww	<4	<2	<4	<2	<2	<40	55	<6	<30	<8	<40	<5	<2
9495	NO-1-Eff	Gjøvik	ng/l	500	600	1,600	<50	<20	610	180	<20	<25	<20	<130	<150	<15
9496	NO-2-Eff	Gjøvik	ng/l	490	570	970	<50	23	620	200	<20	<25	<20	<130	<150	<15
9497	NO-3-Eff	Gjøvik	ng/l	510	610	1,300	<50	<20	620	170	<20	61	<20	<130	<150	<15
9499	NO-4-Slu	Gjøvik	µg/kg dw	28	81	24,000	<8	74	74,000	14,000	1,200	21	<8	<200	<100	<10
9500	NO-5-Slu	Gjøvik	µg/kg dw	43	82	24,000	<8	78	77,000	15,000	1,400	36	<8	<200	<100	<10
9501	NO-6-Slu	Gjøvik	µg/kg dw	27	88	23,000	<8	81	68,000	14,000	1,400	13	<8	<200	<100	<10
9604	NO-7-Sed	Bergen	µg/kg dw	95	<4	1,300	29	5.6	2,300	370	<10	5.1	<10	<50	<50	<5
9605	NO-8-Sed	Bergen	µg/kg dw	98	230	2,900	<8	10	2,300	850	58	27	<10	<50	<50	<5
9606	NO-9-Sed	Bergen	µg/kg dw	99	25	480	<8	<5	400	77	<10	4	<10	<50	<50	<5
9657	NO-10-Fis	Vingrom	µg/kg ww	30	<2	30	<2	<2	110	<40	<6	<30	<8	<40	<5	<2
9658	NO-11-Fis	Vingrom	µg/kg ww	8.9	<2	22	<2	<2	<40	<40	<6	<30	<8	<40	<5	<2
9659	NO-12-Fis	Vingrom	µg/kg ww	<4	<2	7.3	<2	<2	<40	<40	<6	<30	<8	<40	<5	<2
9628	SE-1-Eff	Umeå	ng/l	90	170	5,300	<50	46	490	200	<20	97	<20	<130	<150	<15
9597	SE-2-Eff	Göteborg	ng/l	<50	62	1,500	<50	59	170	<100	<20	1,300	<20	<130	<150	<15
9598	SE-3-Eff	Borås	ng/l	<50	130	2,300	<50	21	530	370	<20	59	<20	<130	<150	<15
9532	SE-4-Slu	Umeå	µg/kg dw	51	380	45,000	<8	110	74,000	19,000	870	48	620	<200	<100	<10
9599	SE-5-Slu	Göteborg	µg/kg dw	42	84	27,000	390	190	71,000	20,000	<20	21	<8	<200	<100	<10
9600	SE-6-Slu	Borås	µg/kg dw	110	460	28,000	<8	110	71,000	42,000	<20	14	430	<200	<100	<10
9601	SE-7-Sed	Göteborg	µg/kg dw	8.3	<4	800	15	<5	1,900	510	21	3.8	<10	<50	<50	<5
9602	SE-8-Sed	Göteborg	µg/kg dw	<8	<4	170	<8	<5	320	55	<10	<1	<10	<50	<50	<5
9593	SE-9-Sed	Strömstad	µg/kg dw	45	<4	<80	<8	<5	39	25	<10	<1	<10	<50	<50	<5
9614	SE-10-Fis	Göteborg	µg/kg ww	16	<2	27	<2	<2	140	<40	<6	<30	<8	<40	<5	<2
1108	SE-11-Fis	Holmöarna	µg/kg ww	<4	<2	13	<2	<2	<40	<40	<6	<30	<8	<40	<5	<2
1109	SE-12-Fis	Kullen	µg/kg ww	23	<2	16	<2	<2	<40	<40	<6	<30	<8	<40	<5	<2
1112	SE-13-Egg	St. Karlsö	µg/kg ww	4.2	<2	<4	<2	<2	<40	<40	<6	<30	<8	<40	<5	<2
1113	SE-14-Egg	St. Karlsö	µg/kg ww	<4	<2	<4	<2	<2	<40	<40	<6	<30	<8	<40	<5	<2

10.4 Appendix 4 Individual results, optional plasticisers

MR#	Sample ident.	Location	Unit	DIBP	DPHP	DIDP
9517	DK-1-Eff	Esbjerg	ng/l	<130	<20	<130
9492	DK-2-Eff	Odense	ng/l	<130	<20	<130
9491	DK-3-Eff	Vordingborg	ng/l	<130	<20	<130
9518	DK-4-Slu	Esbjerg	µg/kg dw	<200	1,000	<200
9494	DK-5-Slu	Odense	µg/kg dw	<200	1,600	<200
9728	DK-7-Sed	Øresund	µg/kg dw	<50	<10	<50
9729	DK-8-Sed	Kolding Fjord	µg/kg dw	<50	<10	<50
9730	DK-9-Sed	Limfjorden	µg/kg dw	<50	<10	<50
9731	DK-10-Fis	Ho bugt	µg/kg ww	<40	<5	<40
9732	DK-11-Fis	Hjelm bugt	µg/kg ww	<40	<5	<40
9733	DK-12-Fis	Agersø	µg/kg ww	<40	<5	<40
9544	FI-1-Eff	Turku	ng/l	<130	<20	<130
9545	FI-2-Eff	Helsinki	ng/l	<130	<20	<130
9546	FI-3-Eff	Tampere	ng/l	<130	<20	<130
9644	FI-4-Slu	Turku	µg/kg dw	<200	2,200	<200
9645	FI-5-Slu	Helsinki	µg/kg dw	<200	3,500	<200
9646	FI-6-Slu	Tampere	µg/kg dw	<200	390	<200
9647	FI-7-Sed	Turku	µg/kg dw	<50	<10	<50
9648	FI-8-Sed	Tampere	µg/kg dw	<50	<10	<50
9649	FI-9-Sed	Helsinki	µg/kg dw	<50	<10	<50
9650	FI-10-Fis	Tampere	µg/kg ww	<40	<5	<40
9651	FI-11-Fis	Kuhmoinen	µg/kg ww	<40	<5	<40
9652	FI-12-Fis	Turku Archipelago	µg/kg ww	<40	<5	<40
9629	FO-1-Eff	Torshavn	ng/l	<130	210	<130
9630	FO-2- Inf	Torshavn	ng/l	<130	93	<130
9631	FO-3-Eff	Klaksvik	ng/l	<130	88	<130
9632	FO-4-Slu	Torshavn	µg/kg dw	<200	650	<200
9633	FO-5-Slu	Torshavn	µg/kg dw	<200	93	<200
9634	FO-6-Sed	Torshavn	µg/kg dw	<50	27	<50
9635	FO-7-Sed	Torshavn	µg/kg dw	<50	250	<50
9636	FO-8-Sed	Kollafjord	µg/kg dw	<50	<10	<50
9637	FO-9-Sed	Klaksvik	µg/kg dw	<50	108	<50
9641	FO-12-Fis	Lake á Mýrunum	µg/kg ww	<40	<5	<40
9642	FO-13-Egg	Skúvoy	µg/kg ww	<40	<5	<40
9643	FO-14-Egg	Koltur	µg/kg ww	<40	<5	<40
1384:1	FO-15-Fis	North-West on the Faroe shelf	µg/kg ww	<40	<5	<40
1384:2	FO-16-Fis	North-West on the Faroe shelf	µg/kg ww	<40	<5	<40
1384:3	FO-17-Fis	North-West on the Faroe shelf	µg/kg ww	<40	<5	<40
1384:4	FO-18-Fis	North-West on the Faroe shelf	µg/kg ww	<40	<5	<40
1384:5	FO-19-Fis	North-West on the Faroe shelf	µg/kg ww	<40	<5	<40
9533	IS-4-Slu	Hveragerði	µg/kg dw	<200	600	<200
9534	IS-5-Slu	Borg - Grímsnesi	µg/kg dw	<200	370	<200
9535	IS-6-Slu	Klettagarðar - Reyk.	µg/kg dw	<200	380	<200
9536	IS-7-Sed	Þingvallavatn	µg/kg dw	<50	<10	<50
9537	IS-8-Sed	Grafarvogur - Reyk.	µg/kg dw	<50	47	<50
9538	IS-9-Sed	Naustavogur - Reyk.	µg/kg dw	<50	140	<50
9539	IS-10-Fis	Þingvallavatn	µg/kg ww	<40	<5	<40
9540	IS-11-Fis	Þingvallavatn	µg/kg ww	<40	<5	<40
9541	IS-12-Fis	Iceland Seas	µg/kg ww	<40	<5	<40
9495	NO-1-Eff	Gjøvik	ng/l	<130	24	<130
9496	NO-2-Eff	Gjøvik	ng/l	<130	36	<130
9497	NO-3-Eff	Gjøvik	ng/l	<130	31	<130
9499	NO-4-Slu	Gjøvik	µg/kg dw	<200	1,400	<200
9500	NO-5-Slu	Gjøvik	µg/kg dw	<200	1,400	<200
9501	NO-6-Slu	Gjøvik	µg/kg dw	<200	1,400	<200
9604	NO-7-Sed	Bergen	µg/kg dw	<50	<10	<50
9605	NO-8-Sed	Bergen	µg/kg dw	<50	21	<50
9606	NO-9-Sed	Bergen	µg/kg dw	<50	<10	<50
9657	NO-10-Fis	Vingrom	µg/kg ww	<40	<5	<40
9658	NO-11-Fis	Vingrom	µg/kg ww	<40	<5	<40
9659	NO-12-Fis	Vingrom	µg/kg ww	<40	<5	<40

MR#	Sample ident.	Location	Unit	DIBP	DPHP	DIDP
9628	SE-1-Eff	Umeå	ng/l	<130	<20	<130
9597	SE-2-Eff	Göteborg	ng/l	<130	<20	<130
9598	SE-3-Eff	Borås	ng/l	<130	<20	<130
9532	SE-4-Slu	Umeå	µg/kg dw	<200	1,700	<200
9599	SE-5-Slu	Göteborg	µg/kg dw	<200	2,100	<200
9600	SE-6-Slu	Borås	µg/kg dw	<200	2,400	<200
9601	SE-7-Sed	Göteborg	µg/kg dw	<50	21	<50
9602	SE-8-Sed	Göteborg	µg/kg dw	<50	<10	<50
9593	SE-9-Sed	Strömstad	µg/kg dw	<50	<10	<50
9614	SE-10-Fis	Göteborg	µg/kg ww	<40	<5	<40
1108	SE-11-Fis	Holmöarna	µg/kg ww	<40	<5	<40
1109	SE-12-Fis	Kullen	µg/kg ww	<40	<5	<40
1112	SE-13-Egg	St. Karlsö	µg/kg ww	<40	<5	<40
1113	SE-14-Egg	St. Karlsö	µg/kg ww	<40	<5	<40

10.5 Appendix 5 Sample list, sweeteners

MR#	Sample ident.	Location	Site	Sampling date	LAT WGS84 Deg., decimal min.			LONG WGS84 Deg., decimal min		
9517	DK-1-Eff	Esbjerg	Esbjerg central WWTP	2.11.2011	55	29.223	N	8	25.491	E
9492	DK-2-Eff	Odense	Ejby Mølle WWTP	24.10.2011	55	23.576	N	10	25.139	E
9491	DK-3-Eff	Vordingborg	Råbylille strand WWTP	11.10.2011	54	58.196	N	12	23.697	E
9518	DK-4-Slu	Esbjerg	Esbjerg central WWTP	2.11.2011	55	29.223	N	8	25.491	E
9494	DK-5-Slu	Odense	Ejby Mølle WWTP	24.10.2011	55	23.573	N	10	24.499	E
9544	FI-1-Eff	Turku	Kakolanmäki WWTP	10.11.2011	60	26.712	N	22	14.204	E
9545	FI-2-Eff	Helsinki	Viikki WWTP	14.11.2011	60	13.544	N	24	59.612	E
9546	FI-3-Eff	Tampere	Viinikanlahti WWTP	14.11.2011	61	29.319	N	23	46.043	E
9644	FI-4-Slu	Turku	Kakolanmäki WWTP	10.11.2011	60	26.712	N	22	14.204	E
9646	FI-6-Slu	Tampere	Viinikanlahti WWTP	14.11.2011	61	29.319	N	23	46.043	E
9629	FO-1-Eff	Torshavn	Sersjantvikin WWTP	21.11.2011	62	0.49	N	6	45.717	W
9630	FO-2-Inf	Torshavn	Main Hospital WWTP,INFLUENT!	21.11.2011	62	0.098	N	6	46.538	W
9631	FO-3-Eff	Klaksvik	Klaksvik Hospital WWTP	21.11.2011	62	13.5	N	6	35.47	W
9632	FO-4-Slu	Torshavn	Sersjantvikin WWTP	21.11.2011	62	0.49	N	6	45.717	W
9633	FO-5-Slu	Torshavn	Main Hospital WWTP	21.11.2011	62	0.098	N	6	46.538	W
9533	IS-4-Slu	Hveragerði	WWTP	24.10.2011	64	59.295	N	21	10.638	W
9535	IS-6-Slu	Klettagarðar Reyk.	WWTP	2.11.2011	64	9.324	N	21	52.405	W
9495	NO-1-Eff	Gjøvik	Rambekk WWTP	25.10.2011	60	46.35	N	10	42.21	E
9496	NO-2-Eff	Gjøvik	Rambekk WWTP	25.10.2011	60	46.35	N	10	42.21	E
9497	NO-3-Eff	Gjøvik	Rambekk WWTP	25.10.2011	60	46.35	N	10	42.21	E
9499	NO-4-Slu	Gjøvik	Rambekk WWTP	25.10.2011	60	46.35	N	10	42.21	E
9500	NO-5-Slu	Gjøvik	Rambekk WWTP	25.10.2011	60	46.35	N	10	42.21	E
9628	SE-1-Eff	Umeå	Öhn WWTP	21.11.2011	63	48.25	N	20	17.5	E
9597	SE-2-Eff	Göteborg	Ryaverken WWTP	10.11.2011	57	41.83	N	11	53.5	E
9598	SE-3-Eff	Borås	Gässlösa WWTP	16.11.2011	57	42.3	N	12	55.5	E
9532	SE-4-Slu	Umeå	Öhn WWTP	8.11.2011	63	48.25	N	20	17.5	E
9599	SE-5-Slu	Göteborg	Ryaverken WWTP	10.11.2011	57	41.83	N	11	53.5	E

Sample ident. suffix: Eff=Effluent, Slu=Sludge

10.6 Appendix 6 Individual results, sweeteners

MR#	Sample ident.	Location	Site	Unit	Aspartam	Cyclamate	Sucralose	Dry weight
9517	DK-1-Eff	Esbjerg	Esbjerg central WWTP	µg/l	<0.06	0.02	3.0	
9492	DK-2-Eff	Odense	Ejby Mølle WWTP	µg/l	<0.05	0.07	1.9	
9491	DK-3-Eff	Vordingborg	Råbylille strand WWTP	µg/l	<0.08	3.9	0.69	
9518	DK-4-Slu	Esbjerg	Esbjerg central WWTP	µg/kg w w	<1	<0.2	2.3	22%
9494	DK-5-Slu	Odense	Ejby Mølle WWTP	µg/kg w w	<1	<0.2	6.7	31%
9544	FI-1-Eff	Turku	Kakolanmäki WWTP	µg/l	<0.13	0.43	5.0	
9545	FI-2-Eff	Helsinki	Viikki WWTP	µg/l	<0.07	0.87	4.2	
9546	FI-3-Eff	Tampere	Viinikanlahti WWTP	µg/l	<0.08	0.08	3.3	
9644	FI-4-Slu	Turku	Kakolanmäki WWTP	µg/kg w w	<1	6.8	12	23%
9646	FI-6-Slu	Tampere	Viinikanlahti WWTP	µg/kg w w	<1	<0.2	3.8	30%
9629	FO-1-Eff	Torshavn	Sersjantvikin WWTP	µg/l	<0.03	1.9	0.37	
9630	FO-2-Inf	Torshavn	Main Hospital WWTP, INFLUENT!	µg/l	<0.23	8.0	0.34	
9631	FO-3-Eff	Klaksvik	Klaksvik Hospital WWTP	µg/l	<0.05	5.7	1.0	
9632	FO-4-Slu	Torshavn	Sersjantvikin WWTP	µg/kg w w	<1	3.0	2.2	19%
9633	FO-5-Slu	Torshavn	Main Hospital WWTP	µg/kg w w	<1	55	2.5	7%
9533	IS-4-Slu	Hveragerði	WWTP	µg/kg w w	<1	0.80	6.9	11%
9535	IS-6-Slu	Klettagarðar - Reyk.	WWTP	µg/kg w w	<1	1.4	2.1	83%
9495	NO-1-Eff	Gjøvik	Rambekk WWTP	µg/l	<0.18	23	3.8	
9496	NO-2-Eff	Gjøvik	Rambekk WWTP	µg/l	<0.17	25	3.4	
9497	NO-3-Eff	Gjøvik	Rambekk WWTP	µg/l	<0.17	23	3.1	
9499	NO-4-Slu	Gjøvik	Rambekk WWTP	µg/kg w w	<1	0.20	11	25%
9500	NO-5-Slu	Gjøvik	Rambekk WWTP	µg/kg w w	<1	0.20	11	25%
9628	SE-1-Eff	Umeå	Öhn WWTP	µg/l	<0.08	0.94	2.2	
9597	SE-2-Eff	Göteborg	Ryaverken WWTP	µg/l	<0.08	<0.01	2.1	
9598	SE-3-Eff	Borås	Gässlösa WWTP	µg/l	<0.10	<0.01	1.7	
9532	SE-4-Slu	Umeå	Öhn WWTP	µg/kg w w	<1	0.46	3.9	34%
9599	SE-5-Slu	Göteborg	Ryaverken WWTP	µg/kg w w	<1	<0.2	2.2	30%

Sample ident. suffix: Eff=Effluent, Slu=Sludg

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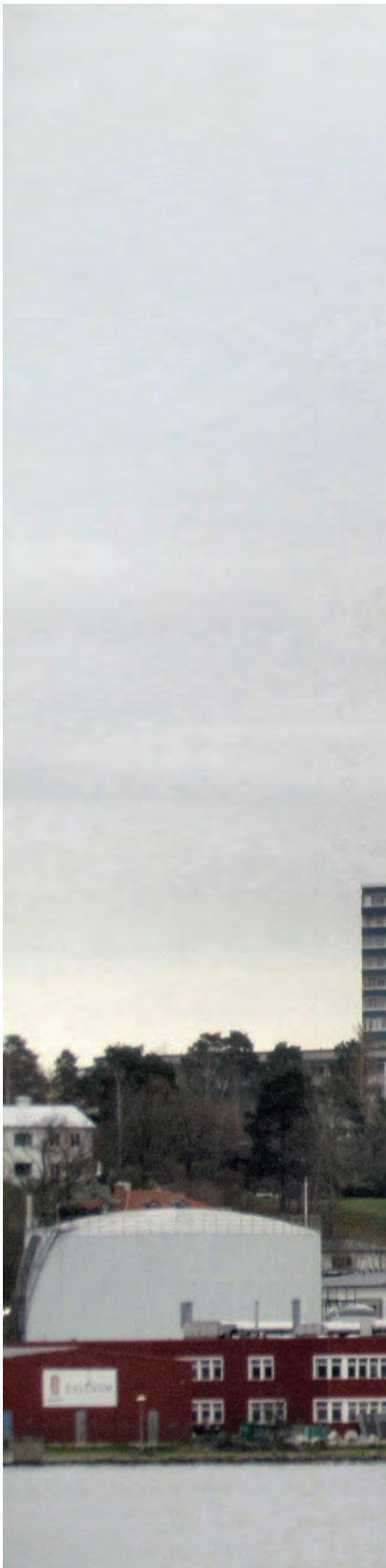
Selected Plasticisers and Additional Sweeteners in the Nordic Environment

The report Selected plasticisers and additional sweeteners in the Nordic Environment describes the findings of a Nordic environmental study. The study has been done as a screening, that is, it provides a snapshot of the occurrence of selected plasticisers and sweeteners, both in regions most likely to be polluted as well as in some very pristine environments. The plasticisers analysed were long chained phthalates and adipates, and the sweeteners analysed were aspartame, cyclamate and sucralose. The purpose of the screening was to elucidate levels and pathways of hitherto unrecognized pollutants. Thus the samples analysed were taken mainly from sewage lines, but also in recipients and biota, both in assumed hot-spot areas and in background areas.



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