



COHIBA

CONTROL OF HAZARDOUS SUBSTANCES
IN THE BALTIC SEA REGION

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WP3 INNOVATIVE APPROACHES TO CHEMICAL CONTROLS OF HAZARDOUS SUBSTANCES

NATIONAL REPORT OF LITHUANIA

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Summary

Lithuanian effluents screening sites were located in less than 50 km distance from the Baltic Sea. The sites included two municipal (LT_MWWTP1 and LT_MWWTP2) and two industrial (LT_IWWTP1 and LT_IWWTP2) waste water treatment plants as well as storm water (LT_SW) and landfill leachate (LT_LW). The treated effluents from MWWTP1, MWWTP2 and IWWTP2 are discharged into the Curonian lagoon. The effluent from IWWTP1 is discharged into municipal WWTP and then, after treatment, is also discharged into the Curonian lagoon. All MWWTP and IWWTP effluents were sampled every second month, eight times starting with May 2009 for biotesting and six times starting with September 2009 for chemical analysis. Data of chemical analysis revealed that concentrations of priority 11 hazardous substances/classes in selected Lithuanian effluents sites did not exceed quantification or even detection levels. Toxicological screening of two WWTPs located near the coastal zone revealed rather high variability of effluents quality discharged into environment in terms of nitrogen containing compounds, in particular, ammonium. The contaminant exceeded Lithuanian MAC limits in several effluent samples. This was reflected by good correlation between ammonium concentration and integral toxicity index calculated from the test-battery data. The toxicity of majority of the effluent samples assessed by various tests did not reach 50% endpoint level. To increase relative sensitivity of the test-battery-based evaluation it was useful to include lower endpoint levels. Then measured positive toxic signals increased from approximately 10 to 20%. Biomarkers (vitellogenin induction, EROD activity assays) applied on two effluents (LT_MWWTP1 and LT_IWWTP1, sampling date 12-01-2010) revealed their potential toxicity, while chronic tests with *L. minor* growth inhibition and long-term *D. magna* reproduction did not show toxicity of these effluents.

1 Introduction

Control of Hazardous Substances in the Baltic Sea or COHIBA – project includes all Baltic Sea countries except Russia. The objective of COHIBA is to support the implementation of the HELCOM Baltic Sea Action Plan (BSAP) with regard to hazardous substances by developing joint actions to reach the goal of “Baltic Sea with life undisturbed by hazardous substances”. The 11 substances/substance groups identified in the BSAP as being of special concern to the Baltic Sea are also the focus of this project. The COHIBA project will last three years (2009-2012).

The COHIBA project aims to identify the most important sources of 11 hazardous substances of special concern. The project analyses flow patterns of these substances and quantifies their releases and inputs to the Baltic Sea. The project will also develop innovative and cost-effective evaluation practices of effluent ecotoxicity, based on the Whole Effluent Assessment (WEA) approach. The target is also to define toxicity-based discharge limits, a threshold toxicity, to effluents discharged into receiving waters in the Baltic Sea region. In addition, the aim is to share knowledge about best practices within the participating countries and to assist authorities and industries to control hazardous substances. The project will enhance the capability of the Baltic Sea countries to implement their international obligations under the forthcoming European Marine Strategy and the Water Framework Directive. It also contributes to national implementation programmes to reach the cessation targets for HELCOM/EU priority hazardous substances by 2020.

The COHIBA project consists of following work packages (WP:s):

- WP1. Project management and administration (lead by Finnish Environment Institute SYKE)
- WP2. Communication and Information (lead by HELCOM Secretariat)
- WP3. Innovative approaches to chemical controls of hazardous substances (lead by SYKE)
- WP4. Identification of sources and estimation of inputs/impacts on the Baltic Sea (lead by IVL Swedish Environmental Research Institute)
- WP5. Cost effective management options to reduce discharges, emissions and losses of hazardous substances (lead by Federal Environment Agency of Germany)
- WP6. Capacity building and knowledge transfer (lead by Baltic Environmental Forum).

The target of WP3 is to contribute to the identification of sources for the 11 hazardous substances in BSAP by performing screening in municipal and industrial waste waters, landfill effluents and storm waters, in all participating countries. WP3 also aims for developing joint evaluation of ecotoxicity of the effluents and for recommending PBT (persistent, bioaccumulating, toxic)-based discharge limit values based on the WEA approach for the Baltic Sea region. Currently most of the restrictions concerning discharges and emissions are based on the determination of chemical concentrations.

However, the majority of effluents comprise a mixture of chemicals. It is impossible to identify all these substances and their transformation products or to determine the effects of all individual substances or their synergistic interactions in the environment. Thus there is a need to regulate discharges on the basis of direct assessments of their biological effects, to complement the chemical analyses. Another important aim of WP3 is to harmonise the chemical and ecotoxicological assessment methods in the Baltic Sea region serving also EU WFD and REACH requirements. The results will be used as input to the integrated HELCOM assessment of hazardous substances and of the sources for the substances of concern to the Baltic Sea.

2 Monitoring programme

2.1 Case studies and monitoring points

The project COHIBA carried out case studies in each of the coastal countries, where municipal and industrial waste waters, landfill effluents and storm waters of selected sites were screened during one year period to identify their ecotoxicity and hence presence of hazardous substances in waste streams.

All Lithuanian cases were located in the western part of the country. In accordance with the project requirements, two municipal (MWWTP) and two industrial (IWWTP) waste water treatment plants located in less than 50 km distance from the Baltic Sea were chosen. Other selected sites were landfill and storm water discharger.

Monitoring sites are presented in Figure 1. The chosen municipal waste water treatment plants deal with mostly domestic waste waters, but partly also waste waters from the small-scale industry. The treated effluents from MWWTP1, MWWTP2 and IWWTP2 are discharged into the Curonian lagoon. The effluent from IWWTP1 is discharged into municipal WWTP and then, after treatment, is also discharged into the Curonian lagoon. All MWWTP and IWWTP effluents were sampled every second month, eight times starting with May 2009 for biotesting and six times starting with September 2009 for chemical analysis. Cases with some additional information are presented in Table 1.

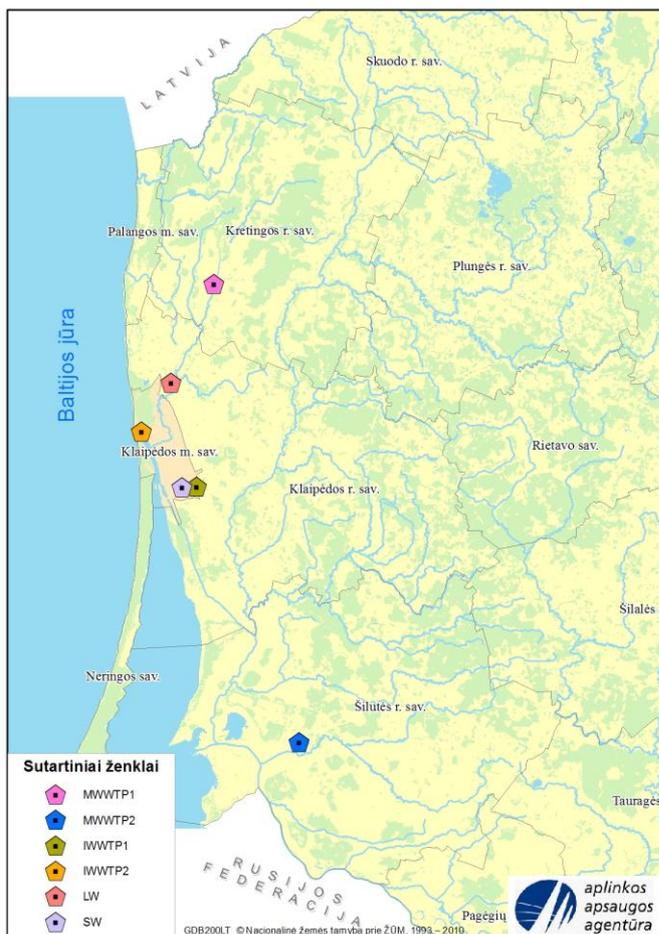


Figure 1. Monitoring sites in Lithuania.

Table 1. Characterization of sampling sites in Lithuania and some parameters of WWTP.

WWTP	Established	Type of treatment	Flow rate*, m ³ /d	Number of inhabitants	Domestic: Industrial	Discharged
MWWTP1	1978	Primary (mechanical), biological	2735	21 452	96:4	Tenzė (tributary of river Akmena-Danė) (approx. 17 km from the Curonian lagoon)
MWWTP2	1988	Primary (mechanical), biological	3342	20 945	90:10	Šyša (tributary of river Nemunas) (approx. 12 km from the Curonian lagoon)
IWWTP1	2003	Primary (mechanical), biological	373		82:18	MWWTP (approx. 9 km from the Curonian lagoon)
IWWTP2	1959	Primary (mechanical), biological	1016		10:90	Approx. 1 km from the Curonian lagoon
SW		None		184 657		Smiltelė stream (approx. 2,5 km from the Curonian lagoon)
LW	1986-2007	None		–		Drainage channel (approx. 9 km from the Curonian lagoon)

MWWTP – municipal waste water treatment plant, IWWTP – industrial waste water treatment plant, SW – storm water discharger, LW – landfill leachate.

* Average flow rate during year 2009.

The dump was used from 1986 to 2007. Landfill slopes were reinforced with a special network of filtration material and plastic film and then planted with grass. The surface of stack was covered with protective layer consisting of mixed soil, geosynthetic clay as well as drainage layer of sand and gravel. Afterwards, the surface was planted with grass. Landfill was equipped with effluent treatment plant. In 2010 the combustion of released biogas was started to produce electricity.

Storm water samples were taken in discharger No. 11, Klaipėda city (coordinates 21°11'45.91" E, 55°39'35.26" N). Storm water drainage basin covers an area approximately 527 ha (Figure 2). The basin area is marked with stripes.

Storm water samples were collected as grab samples. During the sampling in the storm water discharger flow rates were approximately 0,07 l/s (0,252 m³/h) in November 2009 and, respectively, 0,2 l/s (0,72 m³/h) in June 2010.

Storm water, landfill leachate and municipal sewage sludge were sampled twice, once during the cold and once during the warm season. Landfill leachate and storm water was analysed both for chemical parameters and toxicity, municipal sewage sludge – only for chemical parameters.



Figure 2. Storm water discharger No. 11 in Klaipėda city (the scheme provided by UAB “Klaipėdos vanduo”).

All samples were collected as grab samples, except sampling at IWWTP1 and MWWTP1 in January 2010. During that sampling campaign 24 h composite time adjusted samples were taken both for chemical and toxicity measurements.

Samples were analysed considering selected target substances as well as toxicity. The first two analysis rounds were performed concerning toxicity solely, the next four rounds both for toxicity and selected substances while the last two samplings – for chemical analyses.

Sampling schedule and monitoring points are shown in Table 2.

Table 2. Sampling schedule and monitoring points.

Monitoring point, matrix	Sampling date
MWWTP1, waste water	25-05-2009 (only for biotests)
MWWTP2, waste water	09-07-2009 (only for biotests)
IWWTP1, waste water	07-09-2009 (chemicals and biotests)
IWWTP2, waste water	09-11-2009 (chemicals and biotests)
	12-01-2010 (chemicals and biotests)
	12-04-2010 (chemicals and biotests)
	07-06-2010 (only for chemicals)
	16-08-2010 (only for chemicals)
MWWTP, sludge	12-01-2010 (only for chemicals)
	07-06-2010 (only for chemicals)

Monitoring point, matrix	Sampling date
MWWTP1, waste water	25-05-2009 (only for biotests)
MWWTP2, waste water	09-07-2009 (only for biotests)
IWWTP1, waste water	07-09-2009 (chemicals and biotests)
IWWTP2, waste water	09-11-2009 (chemicals and biotests)
	12-01-2010 (chemicals and biotests)
	12-04-2010 (chemicals and biotests)
	07-06-2010 (only for chemicals)
	16-08-2010 (only for chemicals)
MWWTP, sludge	12-01-2010 (only for chemicals)
	07-06-2010 (only for chemicals)
SW, storm water	09-11-2009 (chemicals and biotests)
	07-06-2010 (chemicals and biotests)
LW, landfill leachate	09-11-2009 (chemicals and biotests)
	07-06-2010 (chemicals and biotests)

2.2 Waste water sampling

Sampling was carried out by the specialists of Marine Research Department of Environmental Protection Agency (the former Center of Marine Research). Sampling was performed in accordance with national and/or international ISO EN standards.

Waste water sampling was carried out in accordance with the following standards: LST EN ISO 5667-1:2007. Water quality. Sampling-Part 1: Guidance on the design of sampling programmes and sampling techniques (EN ISO 5667-1:2006); LST EN ISO 5667-3:2006. Water quality. Sampling-Part 3: Guidance on the preservation and handling of water samples (EN ISO 5667-3:2003); ISO 5667-10:1992. Water quality. Sampling-Part 10: Guidance on sampling of waste waters.

Before sampling all containers were washed and prepared according to the instructions sent by the laboratories.

After sampling the sample containers were labelled. During transportation the samples were stored in the freezing boxes. After arrival to the laboratory the samples were stored in the refrigerators at the temperature of +4°C.

Organotin compounds

1000 ml glass bottles were used for the sampling of waste water samples. Glass bottles (2 x 1000 ml) were rinsed with a taken sample, filled to the brim, covered with the aluminium foil and screwed. Phosphoric acid was added to the sample to adjust pH 3. Samples were kept in a cool (+4°C) and dark place.

Polybromodiphenyl ethers (PBDEs)

2500 ml glass bottles were used for samples the sampling of waste water samples. Glass bottles (4 x 2500 ml) were rinsed with a taken sample, filled to the brim, covered with the aluminium foil and screwed. Samples were kept in a cool (+4°C) and dark place.

Perfluorocompounds

1000 ml plastic containers were used for the sampling of waste water samples. The containers were rinsed with a taken sample, filled to the brim, covered with the aluminium foil and screwed. Samples were kept in a cool (+4°C) and dark place.

Hexabromocyclododecane isomers (HBDC)

2500 ml glass bottles were used for the sampling of waste water samples. Glass bottle was rinsed with a taken sample, filled to the brim, covered with the aluminium foil and screwed. Samples were kept in a cool (+4°C) and dark place.

Alkylphenols and their ethoxylates

1000 ml glass bottles were used for the sampling of waste water samples. Glass bottle was rinsed with a taken sample, filled to the brim, covered with the aluminium foil and screwed. Samples were kept in a cool (+4°C) and dark place.

In the presence of free chlorine, approximately 80 mg of sodium thiosulphate pentahydrate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5 \text{H}_2\text{O}$) should be added. Sample should be acidified with hydrochloric acid ($w(\text{HCl})=37\%$) to pH 2. If sodium thiosulphate pentahydrate was added to any of the samples that same amount should be added to blank container.

Cadmium

250 ml borosilicate glass bottles were used for the sampling of waste water samples. The bottle was rinsed with a taken sample, filled to the brim and screwed. Samples were acidified with nitric acid to pH 1-2. Samples were kept in a cool (+4°C) and dark place.

Mercury

250 ml borosilicate glass bottles were used for the sampling of waste water samples. The bottle was rinsed with a taken sample, filled to the brim and screwed. Samples were acidified with nitric acid (1:1) to pH 1-2. Samples were kept in a cool (+4°C) and dark place.

Endosulfan

1000 ml glass bottles were used for the sampling of waste water samples. Glass bottle was rinsed with a taken sample, filled to the brim, covered with the aluminium foil and screwed. Samples were kept in a cool (+4°C) and dark place.

Chlorinated paraffins

1000 ml glass bottles were used for the sampling of waste water samples. Glass bottles (2 x 1000 ml) were rinsed with a taken sample, filled to the brim, covered with the aluminium foil and screwed. Samples were acidified with phosphoric acid to pH 3. Samples were kept in a cool (+4°C) and dark place.

Dioxins

2500 ml glass bottles were used for the sampling of waste water samples. Glass bottle was rinsed with a taken sample, filled to the brim, covered with the aluminium foil and screwed. Samples were kept in a cool (+4°C) and dark place.

Biotests

1000 ml plastic containers were used for the sampling of waste water samples for acute and chronic test. Plastic containers (2 x 1000 ml) were rinsed with a taken sample, filled to the brim and screwed. Samples were kept in a cool (+4°C) and dark place.

500 ml plastic containers were used for the sampling of waste water samples for long-term biotests. Plastic containers (15 x 500 ml) were rinsed with a taken sample, filled to the brim and screwed. Samples were kept in a cool (+4°C) and dark place.

2.3 Storm water sampling

Storm water sampling was carried out in accordance with the following standards: LST EN ISO 5667-1:2007. Water quality. Sampling-Part 1: Guidance on the design of sampling programmes and sampling techniques (EN ISO 5667-1:2006); LST EN ISO 5667-3:2006. Water quality. Sampling-Part 3: Guidance on the preservation and handling of water samples (EN ISO 5667-3:2003) and Manual for storm water sampling [1].

Before sampling all containers were cleaned and prepared according to the instructions sent by the laboratories.

Storm water samples taken as grab samples. Sampling was started after 30 minutes or 1 hour when the rain started.

After sampling the samples containers were labelled. During transportation to the laboratories samples were stored in the freezing boxes. In the laboratory samples were stored in the refrigerators at the temperature of +4°C.

2.4 Sludge sampling

Sludge sampling was carried out in accordance with the following standards: LST EN ISO 5667-13:2000. Water quality. Sampling - Part 13: Guidance on sampling of sludges from sewage and water treatment works (EN ISO 5667-13:1997); LST EN ISO 5667-3:2006. Water quality. Sampling - Part 3: Guidance on the preservation and handling of water samples (EN ISO 5667-3:2003).

Before sampling all containers were cleaned and prepared according to the instructions sent by laboratories.

After the sampling the containers were labelled. During transportation the samples were stored in the freezing boxes. After arrival to the laboratory the samples were stored in the refrigerators at the temperature of + 4°C.

Organo tin compounds

250 ml glass jars was used for the sampling of sludge sample. The jars were filled approximately 2/3 of the volume, covered with the aluminium foil and screwed.

Polybromodiphenyl ethers (PBDEs)

500 ml glass jar was used for the sampling of sludge sample. The jar was filled approximately 2/3 of the volume, covered with the aluminium foil and screwed.

Perfluorocompounds

500 ml plastic jar was used for the sampling of sludge sample. The jar was filled approximately 2/3 of the volume, covered with the aluminium foil and screwed.

Hexabromocyclododecane isomers (HBDC)

500 ml glass jar was used for the sampling of sludge sample. The jar was filled approximately 2/3 of the volume, covered with the aluminium foil and screwed.

Alkylphenols and their ethoxylates

500 ml glass jar was used for the sampling of sludge sample. The jar was filled approximately 2/3 of the volume, covered with the aluminium foil and screwed.

Cadmium

500 ml plastic jar was used for the sampling of sludge sample. The jar was filled approximately 2/3 of the volume and screwed.

Mercury

500 ml plastic jar was used for the sampling of sludge sample. The jar was filled approximately 2/3 of the volume and screwed.

Endosulfan

250 ml glass jar was used for the sampling of sludge sample. The jar was filled approximately 2/3 of the volume, covered with the aluminium foil and screwed.

Chlorinated paraffins

500 ml glass jar was used for the sampling of sludge sample. The jar was filled approximately 2/3 of the volume, covered with the aluminium foil and screwed.

Dioxins

500 ml glass jar was used for the sampling of sludge sample. The jar was filled approximately 2/3 of the volume, covered with the aluminium foil and screwed.

2.5 Landfill sampling

Landfill sampling was carried out in accordance with the following standards: LST EN ISO 5667-1:2007. Water quality. Sampling-Part 1: Guidance on the design of sampling programmes and sampling techniques (EN ISO 5667-1:2006); LST EN ISO 5667-3:2006. Water quality. Sampling-Part 3: Guidance on the preservation and handling of water samples (EN ISO 5667-3:2003); ISO 5667-10:1992. Water quality. Sampling-Part 10: Guidance on sampling of waste waters.

Before sampling all containers were washed and prepared according to the instructions sent by the laboratories.

After sampling the sample containers were labelled. During transportation the samples were stored in the freezing boxes. After arrival to the laboratory the samples were stored in the refrigerators at the temperature of +4°C.

3 Chemical parameters and methods

3.1 Basic parameters

The results of basic chemical parameters from municipal and industrial waste water treatments plants, landfill leachate and storm water are included into Appendixes B-G.

The methods used for determination of basic parameters under the COHIBA project are indicated in Table 3.

Table 3. The methods used for determination of basic chemical parameters.

Parameter	Method	Standard/procedure
Biochemical oxygen demand (BOD ₇)**	Dilution and seeding method with allylthiourea addition	ISO 5815-1:2003 ^A
Chemical oxygen demand (COD _{Cr})**	Titration method	LST ISO 6060:2003
Suspended solids**	Method by filtration through glass fiber filters	LST EN 872:2005 ^A
Total phosphorus (Tot-P)**	Spectrometric method	LST EN ISO 6878:2004 ^A
Phosphate phosphorous (PO ₄ -P)**	Spectrometric method	LST EN ISO 6878:2004 ^A
Total nitrogen (Tot-N)**	Spectrometric method	LST EN 26777:1999 LST ISO 7890-3:1998 LST EN 25663:2000
Ammonium nitrogen (NH ₄ -N)**	Spectrometric method	LST ISO 7150-1:1998
Alkalinity**	Titration method	LST EN ISO 9963-2:1999
pH**	Potentiometric method	LST ISO 10523:2009
Conductivity**	Electrometric method	LST EN 27888:1999
Iron (Fe)*	Atomic emission spectrometry with inductively coupled plasma (ICP-OES)	LST EN ISO 11885:2009
Sulphate (SO ₄ ²⁻)**	Turbidimetry	Standard operating procedure AK-SVP-16.
Total organic carbon (TOC)*	Infrared spectrometry	LST ISO 8245:2003 ^A

ERD EPA* and MRD EPA** laboratories have implemented a quality management system according to LST EN ISO/IEC 17025. A – accredited method.

3.2 Metals

Cadmium

The total concentration of cadmium in the sample after pretreatment was determined using method of atomic absorption spectrometry with graphite furnace (GFAAS) in accordance with standard method LST EN ISO 15586:2004. Water quality. Determination of trace elements using atomic absorption spectrometry with graphite furnace (EN ISO 15586:2003).

For analysis of cadmium 200 ml wastewater sample was preserved by addition of 1 ml concentrated nitric acid to pH<2. Particles were allowed to sediment before analysis. If necessary, 25 ml of the preserved sample was digested with 4 ml nitric acid in a closed system using microwave oven.

The analytical method for determination of concentration of cadmium was based on electrothermal atomic absorption spectrometry with graphite furnace (GFAAS) in accordance with standard method LST EN ISO 5961:2000. Water quality. Determination of cadmium by atomic absorption spectrometry (ISO 5961:1994).

Sludge sample was dried, grinded, homogenized and sieved if necessary. After that appropriate amount of the sample (approximately 0,1-0,3 g) was weighed and digested with 6 ml of nitric acid in a closed system using microwave oven. The extract was transferred to a 50 ml volumetric flask and diluted to volume with ultra pure water. The blank sample was determined in all sample series.

Limit of detection for cadmium is 0,040 µg/l, limit of quantification – 0,050 µg/l. Limit of detection for sludge samples is 0,004 mg/kg, whereas limit of quantification is 0,005 mg/kg.

Mercury

The total concentration of mercury in the sample after pretreatment was determined using atomic fluorescence spectrometry (AFS) in accordance with standard method LST EN ISO 17852:2008. Water quality. Determination of mercury. Method using atomic fluorescence spectrometry (ISO 17852:2006).

For analysis of mercury 200 ml wastewater sample was preserved by addition of 0,6 ml nitric acid (1:1) to pH<2. Particles were allowed to sediment before analysis. If necessary, 25 ml of the preserved sample was digested with 4 ml nitric acid in a closed system using microwave oven. Afterwards, 7,5 ml HCl (120 g/kg) and 0,1 N KBrO₃/KBr solution were added to 40 ml of sample and diluted with ultra pure water till 50 ml volume. The blank sample was determined in all sample series.

The concentration of mercury in the sample was determined using atomic fluorescence spectrometry (AFS) in accordance with standard operating procedure SVP 1-2-4:2009. Soil quality. Determination mercury. Method using using atomic fluorescence spectrometry.

Sludge sample was dried, grinded, homogenized and sieved if necessary. After that appropriate amount of the sample (approximately 0,1-0,3 g) was weighed and digested with 6 ml of nitric acid in a closed system using microwave oven. The extract was transferred to a 50 ml volumetric flask and diluted to volume with ultra pure water. The blank sample was determined in all sample series.

Limit of detection limit for mercury is 0,010 µg/l, limit of quantification – 0,020 µg/l. Limit of detection for sludge sample is 0,015 mg/kg, whereas limit of quantification is 0,05 mg/kg.

3.3 Organotin¹

Organotin compounds in water were determined using simultaneous ethylation and extraction followed by analysis using GC-MS-MS (SS-EN ISO 17353:2005 mod.).

Organotin compounds in sediment were determined using acidic extraction of freeze dried sediment followed by ethylation and analysis using GC-MS-MS. (ISO 23161:2009 mod).

3.4 Phenolic substances²

For analysis of phenolic compounds in wastewater samples, the surrogate standard (¹²C-heptylphenol) as well as the quantification standards (¹³C₆-ring 4-NP, ¹³C₆-ring 4-NP1EO, ¹³C₆-ring NP2EO, ¹³C₆-ring OP, ¹³C₆-ring OP1EO, ¹³C₆-ring OP2EO and *d*₁₆-BPa) were added to acidified (pH <3) samples before extraction. 100 ml of water sample was extracted and purified with conditioned solid phase extraction disks using vacuum. The compounds were eluted from the cartridges with acetone. Elute was evaporated near to dryness (30°C) with stream of nitrogen. Methanol/water was used to re-dissolve the sample to the final volume and the injection standard (¹²C-pentylphenol) was added.

Surrogate standard (¹²C-heptylphenol) was added to the sludge sample (2-3 g dw) prior to shaking with acetone-pentane (2 h). Acetone was removed by shaking with water. Separated pentane layer was evaporated just to the dryness and the sample was re-dissolved to methanol. Water (pH 2-3) was added so that methanol volumr is less than 40%. The quantification standards (¹³C₆-ring 4-NP, ¹³C₆-ring 4-NP1EO, ¹³C₆-ring NP2EO, ¹³C₆-ring OP, ¹³C₆-ring OP1EO, ¹³C₆-ring OP2EO and *d*₁₆-BPa) were added. The conditioned solid phase extraction cartridge (styrene-divinylbenzene

¹Swedish Environmental Research Institute (IVL).

²Finnish Environment Institute (SYKE).

polymer, SDB) was used for sample purification. The acetone extract was evaporated to the dryness (30°C) with nitrogen, re-dissolved with methanol/water and the injection standard (¹²C-pentylphenol) was added.

Instrumental analysis was performed using liquid chromatography (LC) ion trap mass spectrometry (IT-MS) with electrospray ionization. The blank and control samples were determined in the all sample series.

Limit of detection for bisphenol A and 4-nonylphenol (mix.) is 0,1 µg/l, whereas limit of quantification for these substances is 0,35 µg/l. Limit of detection for 4-nonylphenol monoethoxylate (mix.) and octylphenol is 0,05 µg/l, limit of quantification for these substances is 0,17 µg/l. Limit of detection for 4-nonylphenol diethoxylate (mix.), octylphenol monoethoxylate and octylphenol diethoxylate is 0,02 µg/l, limit of quantification for these substances is 0,07 µg/l. Limits of quantification for sludge samples are <5 µg/kg.

3.5 Pesticides

The concentration of analytes was determined using gas chromatography with electron capture detector (GC-ECD) according to standard method LST EN ISO 6468:2000. Water quality. Determination of certain organochlorine insecticides, polychlorinated biphenyls and chlorobenzene. Gas chromatographic method after liquid-liquid extraction (ISO 6468:1996).

For analysis of isomers of endosulfan (α -endosulfan, β -endosulfan and endosulfan sulphate) 1000 ml of waste water sample was extracted. Liquid-liquid extraction with hexane was performed twice to attain good recoveries (over 75%). Extraction was performed using a shaking machine and the extract was concentrated using a rotary evaporator. The extract was cleaned with an alumina/silver nitrate column. The final result was corrected by recovery factor. The blank sample was determined in all sample series.

The concentration of analyte is determined using gas chromatography with electron capture detector (GC-ECD) in accordance with standard method ISO 10382:2002. Soil quality. Determination of organochlorine pesticides and polychlorinated biphenyls. Gas-chromatographic method with electron capture detection. The final result was corrected by recovery factor. The blank sample was determined in all sample series.

Sludge sample was dried, grinded, homogenized and sieved if necessary. After that 10 g of the sample was weighed and extracted with petroleum ether/acetone (2:1) mixture. The extraction was performed using Soxhlet system. The extract was cleaned with an alumina column. Elemental sulphur was removed from the concentrated extract by the treatment with tetrabutylammonium sulfite solution.

Limit of detection for α -endosulfan, β -endosulfan is 0,002 µg/l, for endosulfan sulphate – 0,003 µg/l and limit of quantification is 0,004 µg/l (α -endosulfan, β -endosulfan) and 0,005 µg/l (endosulfan sulphate). Limit of detection for sludge samples is 0,0003 mg/kg, whereas limit of quantification is 0,001 mg/kg.

3.6 Dioxins, furans and PCBs³

Analytes were liquid-liquid extracted from water samples with toluene, using about 200 ml of toluene per liter of water. Sludge samples were dried and Soxhlet extracted with 30% ethanol-

³National Institute for Health and Welfare (THL).

toluene mixture. After extraction the solvent was exchanged to hexane, and sulfur-containing compounds were precipitated with activated copper.

The extract in hexane was transferred to silica gel column (20 mm x 250 mm) containing, from top to bottom, AgNO₃-silica, Na₂SO₄, neutral silica, 15% H₂SO₄-silica, and 44% H₂SO₄-silica. PCDD/Fs and PCBs were eluted from silica gel column with 200 ml of hexane that was concentrated to about 1 ml. Residue of extract was transferred to 2 cm column of aluminium oxide in a Pasteur pipette with the aid of a small additional amount of hexane. The first 2 ml of hexane eluted out of the column were discarded. Then analytes were eluted from the column of aluminium oxide with 5 ml of 20% dichloromethane-hexane to 5 mm x 50 mm carbon column. Mono- and di-*ortho*-PCBs were eluted out from the carbon column in forward direction with the same 5 ml of 20% dichloromethane-hexane and concentrated to 500 µl in hexane for GC-MS analysis. PCDD/Fs and non-*ortho*-PCB were eluted from the carbon column in reverse direction with 15 ml of toluene and subsequently concentrated to 15 µl in nonane for separate GC-MS analysis.

The quantification was performed using gas chromatography - high resolution mass spectrometry (GC-HRMS) with operating in selective ion recording mode. Gas chromatograph Agilent 6890 was connected either high resolution mass spectrometers Waters Autospec Ultima or Waters VG-70 250 SE (resolution 10000). Column used in the GC was DB-Dioxin column (J&W Scientific, 60m, ID 0,25 mm, 0,15 µm).

Internal ¹³C PCDD/PCDF standards (altogether 16 standards) were used to quantitate the amount of PCDDs/PCDFs. ¹²C PCB 30 and ¹³C-labelled PCB congeners (PCB 80, 101, 105, 118, 123, 138, 153, 156, 157, 170, 180, 194 and 209), and ¹³C-labelled non-*ortho*- (co-PCB) congeners (PCB 77, 81, 126 and 169) were used as internal standards for PCBs and co-planar PCBs.

Recoveries of internal standards ranged mainly between 60-120%. However, for some non-*ortho*-PCB also lower recoveries were registered. The blank sample was determined in all sample series.

Limits of quantification for individual PCDD/Fs and non-*ortho*-PCBs congeners in water (sludge) samples were at the range of 0,1 – 2,0 pg/l (pg/g dw), and for individual mono- and di-*ortho*-PCBs – 10 – 100 pg/l (pg/g dw).

3.7 Chloroparaffins (SCCP⁴ and MCCP⁵)

Preservation of water samples for the determination of SCCP and MCCP was carried out in the following way: a set amount of orthophosphoric acid was added to water samples to acidify them to pH 3. Then, the samples were cooled to the temperature of about 4°C and kept at this temperature until analysed.

Short chain chlorinated paraffins SCCP (C₁₀-C₁₃) were determined chromatographically with a Hewlett Packard gas chromatograph HP 6890 Series equipped with µ-ECD detector. The column was Agilent DB-1, 30m x 0,32mm ID, 0,1 µm film thickness, the carrier gas was nitrogen at a constant flow rate of 1ml/min. An aliquot (1 l) of unfiltered effluent was passed through BAKERBOND C-18 cartridge. Before use the cartridges were washed consecutively with 6 ml of methanol and 6 ml of distilled water. The effluent sample was passed through the cartridge at a flow rate of about 5 ml/min. After that, all cartridges were air dried using vacuum for 30 min. SCCPs were eluted using 10 ml of hexane. The solvent in the eluate was evaporated to approximately 0,1 ml under the nitrogen stream. Due to a strong matrix all samples were cleaned-up in the next SPE

⁴Institute for Ecology of Industrial Areas (IETU).

⁵Institute of Non-Ferrous Metals.

procedure with cartridges filled with 1 g of BAKERBOND Amino (NH₂) and 1 g of BAKERBOND Cyan (CN) phases. The cartridges were washed with 10 ml of hexane before use. The concentrated eluate from the previous cleaning stage was quantitatively transferred onto the top of the cartridge. SCCPs were eluted from the bed by passing 10 mL of hexane through the cartridge. The solvent excess was evaporated to approximately 0,3 ml under the nitrogen stream and the final volume was set at 0,5 ml using hexane.

Samples were determined chromatographically under the following conditions:

Make-up gas: nitrogen at 60 ml/min,

Split ratio: 2:1,

Injector temperature: 320°C,

Detector temperature: 330°C,

The oven temperature program: 110°C (held for 2 min), to 320°C at 10°C/min, then 320°C (held for 3 min),

Injected volume: 1 ml.

Pretreatment of samples for determination of SCCP in sludge was carried out in the following way: the fresh sludge was air dried in a dark room and after grinding it was passed through a 0,25 mm sieve. An aliquot of air dried sludge (2 g) was mixed thoroughly with 2 g of diatomaceous earth, placed in extraction cell and mounted in the tray of Dionex ASE 350 extractor (Accelerated Solvent Extractor).

Extraction parameters are given below:

Pressure: 10 Mpa,

Oven temperature: 100°C,

Oven heat-up time: 5 min,

Static time: 10 min,

Solvent: hexane/acetone (1:1 v/v),

Nitrogen purge: 1 MPa for 60 s.

The obtained extract was evaporated to the volume of 1 ml and then quantitatively transferred onto the top of the cartridge filled with 1 g of BAKERBOND Amino (NH₂) and 1 g of BAKERBOND Cyan (CN) phases. Before use the cartridges were washed with 10 ml of hexane. SCCP were eluted using 10 ml of hexane. The solvent excess was evaporated to approximately 0,3 ml under the nitrogen stream and the final volume was set at 0,5 ml using hexane. The chromatographic parameters were the same as in the effluent analyses.

Medium chain chlorinated paraffins MCCP (C₁₄-C₁₇) were determined chromatographically using a gas chromatograph with ECD detector. 1 l water samples were filtrated. Discovery DSC-18 (C-18) cartridges were used. Before use the cartridges were washed with 2 ml of methanol and 2 ml of distilled water. 1 l water samples were passed through the cartridges at a flow rate of about 5ml/min. After that, cartridges were vacuum-dried for 15 min. Chlorinated paraffins were eluted using 6 ml of hexane. The solvent in the eluant was evaporated just to dryness and reconstituted in 1 ml of hexane. The samples were determined chromatographically with Perkin Elmer Clarus 500 Gas Chromatograph equipped with ECD detector, column: Elite – MS (DB-5MS), 30m x 0,25mm, 0,25 µm film thickness. The carrier gas was helium used at a constant flow rate of 1ml/min.

Samples were determined under the following conditions:

Injector temperature 250°C,

Detector temperature: 310°C,

The oven temperature program: 110°C (held for 1 min) to 200°C at 25°C/min, then to 300°C at 8°C (held for 20 min).

Limit of detection is 0,2 µg/ml, limit of quantification – 0,6 µg/ml.

Sediment extraction to determine MCCP in sludge was carried out in the following way: 10 g sediment sample was extracted in the ultrasonic bath in 75 ml of dichloromethane for 1 hour. After the extraction, the extract was filtrated and evaporated just to dryness on the vacuum evaporator. Before the chromatographic analysis, the extract was reconstituted in 1 ml of hexane and sulphur was removed by adding copper. The recovery of this method is estimated at 93,4%. Detection limit of this method is 10 µg/kg, quantification limit - 30 µg/kg. Combined standard uncertainty for this method is estimated at 11,2%.

3.8 Brominated flame retardants⁶

For analysis of polybrominated biphenyls (PBDEs) surrogate standard (¹³C₁₂-BDE 77) was added to the samples before water samples (approx. 3200 ml) were extracted as whole samples. The liquid-liquid extraction with DCM was performed two times to attain good recoveries (over 70%).

Freeze-dried sludge samples (1 g) were extracted with DCM (dichloromethane) at ASE instrument (Accelerated Solvent Extraction). Surrogate standard (¹³C₁₂-BDE 77) was added to the samples before extraction.

The extracts were cleaned with multilayer silica column and basic alumina column (according to standard ISO 22032). The samples were concentrated and solvent was changed to nonane before instrumental analysis. Low pressure gas chromatography tandem mass spectrometry (LP-GC-TQ MS) was used for instrumental analysis of PBDE compounds. Prior to injection, ¹³C₁₂-BDEs 28, 47, 99, 153, 183, 209) were added as quantification standards. The final results are recovery corrected. The blank samples were determined in the all sample series.

For analysis of isomers of hexabromocyclododecanes (HBCD) surrogate standards (¹³C₁₂- α-, β- and γ-HBCD) were added to the samples before water samples (approx. 1600 ml) were extracted as whole samples. The liquid-liquid extraction with DCM was performed twice to assure good recoveries (over 70%).

Freeze-dried sludge sample (1 g) was extracted with DCM at ASE instrument (Accelerated Solvent Extraction). Surrogate standards (¹³C₁₂- α-, β- and γ-HBCD) were added to the samples before extraction.

The extracts were cleaned with acidified silica and basic alumina columns. The samples were concentrated and solvent was changed to methanol before instrumental analysis. Ultra performance liquid chromatography (UPLC) tandem mass spectrometry (TQ MS) was used for instrumental analysis of three HBCD diastereomers (α-, β- and γ-HBCD). Prior to injection, *d*₁₈ α-, β- and γ-HBCD were added as quantification standards. The final results are recovery corrected. The blank samples were determined in the all sample series.

Limit of detection for PBDEs is 0,03 ng/l, whereas limit of quantification for these substances is 0,15 ng/l.

3.9 Perfluorinated compounds⁶

For analysis of perfluorocompounds (PFC) surrogate standards (¹³C₂-PFHxA, ¹³C₄-PFOS and ¹³C₂-PFDA) were added to the samples before centrifugation. The solid phase extraction (copolymer cartridges) was used for sample purification and concentration. A portion of the extract was

⁶Finnish Environment Institute (SYKE).

analysed without concentration. Prior to injection, $^{13}\text{C}_4\text{-PFOA}$ was added as a quantification standard.

Freeze-dried sludge sample (1 g) was placed in a PP tube and surrogate standards ($^{13}\text{C}_2\text{-PFHxA}$, $^{13}\text{C}_4\text{-PFOS}$ and $^{13}\text{C}_2\text{-PFDA}$) were added. Analytes were extracted in wrist-action shaker with 200 mM NaOH (in MeOH), 2 M HCl (in MeOH) and MeOH. The extracts were concentrated, and then purified with activated carbon and glacial acetic acid. Purified extract was diluted with Milli-Q water and $^{13}\text{C}_4\text{-PFOA}$ was added as a quantification standard.

Instrumental analysis was performed with ultra performance liquid chromatography (UPLC) tandem mass spectrometry (TQ MS). The LC effluents were water and methanol buffered with ammonium acetate. Calibration was done using linear-only standards and the results were reported as a sum of both linear and branched isomers. The final results are recovery corrected. The blank samples were determined in the all sample series.

Limit of detection for perfluoro-n-hexanoic acid is 0,03 ng/l, limit of quantification – 0,5 ng/l. Limit of detection method for perfluorooctane sulfonate, perfluorooctanoic acid and perfluoro-n-decanoic acid is 0,04 ng/l, limit of quantification – 0,5 ng/l. Limit of detection for sludge samples is 0,06 $\mu\text{g}/\text{kg}$, whereas limit of quantification is 0,1 $\mu\text{g}/\text{kg}$.

4 Biotesting methods

4.1 Obligatory tests

4.1.1 Algaltox

Freshwater algal growth inhibition test with unicellular green algae (ISO 8692:2004. Water quality)

All samples after arrival to the laboratory are immediately filtered through membrane filter of 0,45µm, held in refrigerator (3-6)°C and tested according to standard method.

Freshwater algal growth inhibition test with unicellular green algae *Selenastrum capricornutum* (*Pseudokirchneriella subcapitata*), immobilized in algal beads.

Equipment for measuring algal cell density - VIS-Spectrophotometer;

Culture flasks- 250 ml conical flasks with air permeable stoppers or 25 ml cells.

Incubate the test vessels at 21-25° C, under continuous, white light, continuously shake in an incubator. Determine the OD in each cell after 24 h, 48 h and 72h incubation. Calculate the mean daily OD values for the 3 replicate and determine the 72h ErC50 by using appropriate software.

4.1.2 Daphtox

Determination of the inhibition of the mobility of *Daphnia magna* Straus (Cladocera, Crustacea). (Water quality, LST EN ISO 6341+AC:2000); (Acute toxicity test, EN ISO 6341:1996/AC:1998).

All samples after arrival to the laboratory are immediately filtered through membrane filter of 0,45 µm, held in refrigerator (3-6)°C and tested according to standard method.

24 to 48 h EC50 bioassays are performed in disposable multiwell test plates departing from neonates, uniform in size and in age, hatched from ephippia. Hatching of the ephippia must be initiated 3 days prior to the start of the toxicity test.

The embryonic development of *Daphnia magna* eggs takes about 3 days in optimal conditions under the illumination and temperature conditions indicated above (i.e. 6000 lux and 20-22° C) the first neonates may even appear before 72h incubation, but the largest hatching will occur between 72h and 80h of incubation. The neonates should not be older than 24h at the start of the toxicity test. The organism must be collected at the latest 90h after the start of the incubation. Dilution series (100%-75%-50%-25%-12,5%).

For a statistically acceptable evaluation of the effects, each test concentration as well as the control has to be assayed in 4 replicates.

4.1.3 Microtox

Determination of the inhibitory effect of water samples on the light emission of *Vibrio fischeri* (Luminescent bacteria test), (Water quality, LST EN ISO 11348-3:2009); Part 3: Method using freeze-dried bacteria (EN ISO 11348-3:2008).

All samples after arrival to the laboratory are immediately filtered through membrane filter of 0,45 µm, held in refrigerator (3-6)°C and tested according to standard method.

The test criterion is the luminescence, measured after a contact of 30 min, taking into account a correction factor (f_{kt}), which is a measure of intensity changes of control samples during the exposure time. The inhibitory effect of the water sample can be determined EC50 values by means of a dilution series.

Equipment - Microtox Model 500 Analyzer with programs (MicrotoxOmni™ Software). This software allows users of the Microtox Model 500 analyzer to run tests, visualize the obtained data and associate calculated statistic.

If the sample pH lies between 6 and 8,5 there is generally no adjustment.

Prepare the test suspensions directly in the test tubes.

4.2 Optional tests

4.2.1 Thamnotox

Acute freshwater toxicity microbioassay with anostracan crustacean *Thamnocephalus platyurus* (THAMNOTOXKIT F)

according to Standard Operational Procedure (MicroBioTests Inc., Belgium)

Test principle and test criterion

The acute THAMNOTOXKIT F is a 24h assay based on mortality of the test organisms, with calculation of the 24hLC50.

All samples after arrival to the laboratory are immediately filtered through membrane filter of 0,45 µm, held in refrigerator (3-6)°C and tested according to standard method.

24 h LC50 bioassay is performed in a multiwell test plate using instar II-III larvae of the fairy shrimp *Thamnocephalus platyurus*, which are hatched from cysts. Cyst hatching should be initiated 24 hours prior to the start of the toxicity test (prehydration of the cysts-during 30 minutes period with shake it at regular intervals; and then hatching Petri dish incubate at 25° C for 20-22 h, under continuous illumination (light source of min. 3000-4000 lux).

A dilution series (100%-75%-50%-25%-12,5 %); Each toxicant dilution has to be transferred into all the wells of one column in the multiwell plate (1 ml to each well). Control-dilution water. Transfer of the fairy shrimp larvae to the multiwell plate is accomplished in two steps: a) transfer of the larvae from the Petri dish into the rinsing wells of the multiwell plate; b) transfer of the larvae from the rinsing wells to the actual test wells (3 replicates). Put the strip of Parafilm back on the test plate, cover the multiwell and incubate at 25°C in darkness, for 24 hours.

Calculate the percentages of mortality at each tested concentration. Indicate the concentrations or dilutions used in the dilution series on the Y-axis. Plot the percent mortality on the horizontal line at the height of each concentration. Connect the plotted mortality points with a straight line. Locate the two most adjacent points on the graph which are separated by the vertical 50% mortality line, and read the LC50 at the intercept of the two lines. Alternatively to graphical interpolation, appropriate software programme can be used.

4.2.2 Rotox

Acute freshwater toxicity microbioassay with rotifer *Brachionus calyciflorus* (ROTOXKIT F)

according to Standard Operational Procedure (MicroBioTests Inc., Belgium)

Test principle and test criterion

The acute ROTOXKIT F is a 24h assay based on mortality of the test organisms, with calculation of the 24hLC50.

1. Preparation of standard freshwater

- a) Fill a 1 liter volumetric flask with approximately 800 ml deionized (or distilled) water.
- b) Uncap the vial with concentrated salt solution labeled number 1 (NaHCO₃), and pour the content in the flask.
- c) Repeat step 2 for the other vials with concentrated salt solutions, i.e. two vials number 2 (CaSO₄), one vial number 3 (MgSO₄) and one vial number 4 (KCl), respecting this sequence.
- d) Add deionized water up to the 1000 ml mark and shake to homogenize the medium.

2. Pre-aeration of the Standard Freshwater

The Standard Freshwater must be aerated for at least 15 min prior to use it for the hatching of the cysts and for the preparation of the toxicant dilutions. Pre-aeration can be performed very easily by air bubbling through a tube connected to an aquarium pump or a flask with the medium is kept uncapped for approximately 1 hour.

3. Storage of the medium

Store Standard Freshwater in the refrigerator in darkness. Take care to bring the cooled medium (gradually) back to room temperature prior to use.

4. Hatching of the rotifer cysts

Rotifer cyst hatching should be initiated one day prior to the start of the toxicity test.

1. Add 2 ml Standard Freshwater to the hatching trough. One can also use a 5 cm petridish (with 10 ml Standard Freshwater) to perform hatching.
2. Empty the contents of one vial with cysts into the hatching trough of the test plate; make sure most of the cysts are removed from the vial. To secure complete transfer of the cysts, the vial should be rinsed with 0.5 ml Standard Freshwater.
3. Put a strip of Parafilm on the test plate, cover the multiwell and incubate the plate at 25°C for 16-18 hours, with continuous illumination (light source of 3000-4000 lux).

5. Preparation of toxicant dilution series

A dilution series 100% - 50% - 25% - 12.5% and 6.25% of the effluent sample is prepared by serial 1:2 dilution.

1. Add 5 ml dilution water to test tubes 2, 3, 4 and 5.
2. Add 10 ml effluent sample to test tube 1 and rinse the pipet.
3. Using the same pipet, transfer 5 ml of test tube 1 to test tube 2 and rinse the pipet; cap and shake test tube 2.
4. Repeat this procedure (step 3) for the next dilutions: 5 ml from test tube 2. to 3; 5 ml from test tube 3. to 4; 5 ml from test tube 4. to 5.
5. Proceed to the section 6: Filling of the Test Plate.

6. Filling of the Test Plate

Controls

1. Add 0.7 ml Standard Freshwater to the rinsing troughs of the top row.
2. Add 0.3 ml Standard Freshwater to each of the six test wells of the top row.

Toxicant

3. Shake each (capped) test tube thoroughly before the transfer of the toxicant.
4. Transfer 0.7 ml of test tube 5 to the rinsing trough of row 1.
5. Transfer 0.3 ml of test tube 5 to each of the 6 test wells in row 1.
6. Repeat this procedure (steps 4 and 5) with test tubes 4, 3, 2 and 1 to fill the rinsing troughs and the test wells of rows 2, 3, 4 and 5 respectively.

7. Transfer of the rotifers into the test wells

1. Take the test plate out of the incubator and put it on the stage of the dissection microscope. The next steps are executed under a dissection microscope at magnification 10-12x.
2. Transfer approximately 50 rotifers with the micropipet from the hatching trough into the rinsing trough of control row (top row).
3. Transfer 5 rotifers from the rinsing trough to each of the six wells in top row. Take care during this operation to carry over as little as possible medium along with the rotifers. It is advised to count the rotifers as they exit the micropipet, to make sure that exactly five organisms are put in each test well.
4. Repeat this operation (steps 2 and 3) for rows 1, 2, 3, 4 and 5, in this sequence (i.e. in increasing order of concentration of the toxicant).
5. After completion of the last transfer, empty the hatching trough to avoid spilling of the hatching medium into the test wells during subsequent transportation of the test plate.

8. Incubation of the test plate

Put the strip of Parafilm back on the test plate, cover the plate and incubate at 25°C in darkness, for 24 hours.

9. Scoring of the results

1. Take the test plate out of the incubator and put it under the dissection microscope.

2. Check each test well of columns A, B, C, D and F, and record the number of dead* and living rotifers.

* The organisms are considered dead if they do not exhibit any movement in 5 seconds of observation even after gentle agitation of the liquid.

3. Score the mortality figures on the RESULT SHEET.
4. Total the number of dead rotifers for each concentration and calculate the % mortality*.
5. If the mortality in the controls exceeds 10%, the bioassay is considered invalid and the test must be repeated!

10. Estimation of the LC₅₀

1. Indicate the concentrations or dilutions used in the dilution series on the Y-axis.
2. Plot the percent mortality on the horizontal line at the height of each concentration.
3. Connect the plotted mortality points with a straight line.
4. Locate the two most adjacent points on the graph which are separated by the vertical 50% mortality line, and read the LC₅₀ at the intersect of the two lines.

Alternatively to graphical interpolation, appropriate software programme can be used.

11. Reference test

Weigh 100 mg potassium dichromate on an analytical balance, transfer it to a 100 ml volumetric flask and fill to the mark with deionized water.

1. Make dilutions series of the reference toxicant (e.g. in 10 ml plastic tubes). The dilution series for the reference tests ranges between 3.2 and 32 mg/l: 32, 18, 10, 5.6 and 3.2 mg/l.
2. Prepare five dilutions : C1 (56 mg/l), C2 (32 mg/l), C3 (18 mg/l), C4 (10 mg/l), and C5 (5.6 mg/l).
3. Proceed to the section 6: Filling of the Test Plate.

From the data obtained in the quality control test, a 24h LC₅₀ has to be calculate, the value of wich should be situated within the limits (range) stipulated in the specification sheet.

4.2.3 Protox

Chronic (multigeneration) freshwater toxicity microbiotest with protozoan ciliate *Tetrahymena thermophila* (PROTOXKIT F)

according to Standard Operational Procedure (MicroBioTests Inc., Belgium)

Test principle and test criterion

The Protoxkit assay is a multigeneration growth test which includes 5-6 generations and is completed in 24 hours (short term assessment of chronic toxicity). The assay is based on the measurement of the turnover of food substrate into ciliate biomass by optical density (OD) readings. Inhibited culture growth under toxic stress is reflected by remaining turbidity of the food suspension (and hence higher OD) in comparison to the control. Mean growth inhibition after 24h exposure at 30°C is determined and expressed as 24h EC₅₀.

1. Preparation of toxicant dilution series

A dilution series (100% - 50% - 25% - 12.5% and 6.25%) of the centrifuged (or filtered) effluent sample is prepared by serial 1:2 dilution with distilled water.

1. Take five 15 ml test tubes (preferably in glass) and label them from C1 to C5. C1 will contain the undiluted effluent, C5 the highest dilution.
2. Put 10 ml of the undiluted (treated) sample in tube C1 (=100%).
3. Fill C2 to C5 with 5 ml distilled water each.
4. Transfer 5 ml from C1 to C2 and mix (=50%).
5. Repeat this operation for C2 to C3 (=25 %), C3 to C4 (=12,5 %) and C4 to C5 (=6,25 %).

2. Preparation of ciliate inoculum

1. Take the stock culture vial and shake it gently to homogenize the contents.
2. Take 500 µl from the ciliate stock culture with a sterile syringe.

3. Transfer the 500 µl stock suspension into a 1.5 ml stock-culture cell and add 1 ml distilled water.
4. Cover the stock-culture cell with its lid, shake gently and measure the optical density at 440 nm.
5. Calculate the dilution factor needed to arrive at a 'theoretical' OD value of 0.040 with the formulas: $F = \text{ODvalue} / 0.040$ and $V = 0.5 \times (F - 1)$
(e.g., if the original OD is 0.090, the 1.5 ml ciliate suspension should be diluted by a factor $0.090/0.040 = 2.25$ times).
6. Transfer 500 µl of the diluted ciliate stock into the ciliate inoculum tube and add V ml distilled water.
7. Close the tube with the stopper and mix gently.

3. Preparation of the food suspension

1. Take one vial of reconstitution medium and one vial of food substrate out of the deepfreezer and defrost the content slowly.
2. Transfer (by micropipetting) the full contents of the vial with reconstitution medium into the food substrate tube.
3. Close the food substrate tube and mix thoroughly.

4. Inoculation of the test cells

6. Take 12 test cells and label them in pairs C0 to C5 (two cells for each dilution).
7. Add 2 ml distilled water to the two C0 cells (controls).
8. Add 2 ml from dilution tubes C1 to C5 to the respective C1 to C5 test cells.
9. Take the food substrate tube and mix the contents thoroughly. Add 40 µl food suspension to each of the 12 test cells.
10. Take the ciliate inoculum tube and homogenize the contents by gentle shaking. Transfer 40 µl food suspension to each of the 12 test cells. Close all the cells with their lids.

5. Optical density measurements and incubation of the test cells

1. Zero-calibrate the spectrophotometer at 440 nm, with a test cell containing 2 ml distilled water.
2. Subsequently, and after gentle shaking (by inverting the cells a few times), measure the optical density (OD) of each test cell at **440 nm** (= time T0 scorings).
3. Record the T0 data on the Result Sheet.
4. Put all the cells back in their holding tray and put the tray in an incubator at 30°C for 24 hours.
5. After 24 h incubation, recalibrate the measuring equipment with a test cell containing 2 ml distilled water. Gently shake each cell and determine again OD at 440 nm (= time T24 scorings).
6. Record the T24 data on the Result Sheet.

5. Validity of the test

For the toxicity test to be acceptable, the OD of the controls after 24h incubation must show a decrease of the T0 value by at least 60%.

6. Data treatment

1. Calculate the mean for the two parallels for each toxicant dilution and the control.
2. Calculate the difference between the mean OD at T0 and T24 for each toxicant dilution ($\Delta\text{OD}_{\text{C1-C5}}$) and controls ($\Delta\text{OD}_{\text{C0}}$).
3. Calculate the % inhibition for each toxicant dilution by the following equation:
$$\% \text{ inhibition}_{(\text{C1-C5})} = [1 - (\Delta\text{OD}_{\text{C1-C5}}) / (\Delta\text{OD}_{\text{C0}})] \times 100.$$
4. Calculate the 24h EC50 using appropriate software.

9. Reference test

1. Make a stock solution of 100 mg/l potassium dichromate by weighting 25 mg of the compound and dissolving it in distilled water in a 250 ml calibrated flask.
2. Prepare five dilutions : C1 (56 mg/l), C2 (32 mg/l), C3 (18 mg/l), C4 (10 mg/l), and C5 (5.6 mg/l).

From the data obtained in the quality control test, a 24h EC50 has to be calculate, the value of wich should be situated within the limits stipulated in the specification sheet.

4.2.4 Charatox

Freshwater toxicity microbiotest using charophyte alga cell electrophysiological reaction (CHARATOX)

according to Standard Operational Procedure (BI-VTS-01:2006, Lithuania)

Test principle and test criterion

The CHARATOX is a rapid assay based on electrophysiological response of green macrophytic alga cell of *Nitellopsis obtusa*, with calculation of the 90 min IC₅₀. Toxicity is defined as averaged 50% depolarization of resting potential (RP) of algal cells, i.e. decrease of absolute value of cell RP when the cells were affected by aquatic sample, at the end of 90 min exposure period in relation to the average value of the cells RP measured prior to the initiation of toxicant action.

The test is performed by means of hardware consisted of testing chamber for 32 cells and the measuring electrodes; interface which is connected to PC through serial port, and software allowing to store RP-values taken in required frequency and vizualize them as graphs or numbers in on-line regime.

1. Alga collection from the natural habitats and holding conditions in the laboratory

A freshwater charophyte, *Nitellopsis obtusa* (Desv.) J. Groves, is harvested by kedge anchor at a depth of approximately 5 m in freshwater lakes during vegetation period. The plants are transported to the laboratory in plastic bags filled with lake water. After separation from the bulk by cutting with the sizors, single internodal cells (each cell length 3-10 cm) are kept in 2 L glass vessels filled with 1/2 of tap water (unchlorinated) and 1/2 of lake water. Vessels with the cells are kept under dim light at room temperature, the holding medium has to be changed approximately every two months. So stored cells can be used for biotesting for 4-5 months.

2. Preparation of dilution medium

1. Fill a 1 liter volumetric flask with approximately 800 ml distilled (or deionized) water.
2. Pipet 1 ml of KCl, 5 ml CaCl₂, 10 ml NaCl and 10 ml HEPES-bufer from respective concentrated (100 mM) solutions and add to 1 liter flask.
3. Add distilled water up to the 1000 ml mark and shake to homogenize the medium.

The pH of the dilution (control) medium should be 7.5 (ajusted by 0.1 M HCl and 1 M NaOH).

3. Preparation of toxicant dilution series

A dilution series of the effluent sample is prepared according to standard dilution procedures prescribed for range-finding or definitive tests. It is required 70 ml of the testing solution for each 8-cell group.

4. Placememnt in the chamber and accomodation of algal cells

The cells are preadapted and exposed to toxicants in Petri dishes in darkness at room temperature 12-24 hours. Algal cell is placed into the chamber by positioning the node and small segment (about 1 mm) of each cell in the common central c-pool, and the larger part of the cell in a distinct yet identical for each cell testing a-pool. Electrical insulation between c-pool and a-pools is achieved with vaseline junctions.

1. Dry shortly the cell in the air before placing it into the chamber.
2. Dip gently each of the cell into the vaseline, wrap up carefully by using a flat part of the spatula to prevent small air bubbles between the cell surface and vaseline and drip several drops of control medium into the a-pool to let the water be in contact with the cell.
3. Fill up testing and reference pools with the dilution medium.
4. Fill up central pool with the KCl-medium (in mM): 100 KCl, 1 NaCl, 0,5 CaCl₂ and 1 HEPES-bufer, pH 7,5 (ajusted by 0.1 M HCl and 1 M NaOH).

5. Initiate software programme, which allows to monitor kinetics of RP of the cells and set the file name of the test.
6. Exclude the cell(s) which generates unstable resting potential or its value is more positive than – 140 mV from the visualization on the monitor and the statistics of the respective group. Elimination of up to three cells in each 8-cell group is tolerable.
7. Accommodate the cells in their experimental respective pools for 1-2 hours in control solution after filling the central pool with KCl. During this period the average RP value of the cell group(s) stabilizes and, if it is maintained for 0.5 h unchanged ($\pm 2.5\%$), the testing procedure will be initiated.

5. Exposure and measurement of cell resting potential

The test is carried out at room temperature (18-24°C) in dim light. The group of 8 cells is required per one sample concentration.

1. Record the T0-values of cell RP for each of the groups on the Result Sheet;
2. Change the control medium by respective dilution in each of the groups and indicate the time.
3. Record the T90(min)-values of cell RP for each of the groups on the Result Sheet.
4. Take out the cells and wash the pools with distilled water.

6. Data treatment

1. Calculate the mean RP values before (RP_0) and after (RP_{90}) treatments for each cell group.
2. Calculate the % inhibition for each toxicant dilution by the following equation:

$$\% \text{ depolarization} = (1 - RP_{90}/RP_0) \times 100\%.$$
3. Calculate the 90min IC_{50} using a non-linear (logistic) regression of the depolarization percentage with the logarithms of exposure concentration if all data points are used, and linear regression of the depolarization percentage with the logarithms of exposure concentration if concentrations that induce 20-80% depolarization are used.
4. If undiluted (100 %) effluent sample did not induce more than 50% depolarization of averaged RP of the cell group indicate an observed percentage of cell depolarization.

7. Validity of the test

When calculate percentage depolarization, the cell RP values that immediately before the treatment (at the end of accommodation) were more positive than -140 mV are not included. The same is truth for the occasionally dead cells that have been injured during the solution exchange procedures.

The mean RP-value of the group is valid if it was calculated from no less than five cells.

Estimation of IC_{50} value has to be based on no less than three data points that enfold 50% depolarization level.

8. Reference test

1. Make a stock solution of 10 g/l potassium dichromate by weighting 2.5 g of the compound and dissolving it in distilled water in a 250 ml calibrated flask.
2. Prepare four dilutions: C1 (0.45 g/l), C2 (0.6 g/l), C3 (0.9 g/l) and C4 (1.2 g/l).

Mean 90-min IC_{50} for the reference toxicant potassium dichromate ($K_2Cr_2O_7$): 0.77 g/l (0,62-0,92).

From the data obtained in the quality control test, a 90-min IC_{50} has to be calculated, the value of which should be situated within the limits.

Reference

[13] Manusadžianas L, Maksimov G, Darginavičienė J, Jurkonienė S, Sadauskas K, Vitkus R. 2002. Response of the charophyte *Nitellopsis obtusa* to heavy metals at the cell, cell membrane, and enzyme levels. *Environmental Toxicology* **17**:275-283.

4.2.5 Niteltox

Acute freshwater toxicity microbioassay with charophyte alga cells (NITELTOX) according to Standard Operational Procedure (BI-VTS-02:2006, Lithuania)

Test principle and test criterion

The acute NITELTOX is a 96 h assay based on mortality of green macrophytic alga cells of *Nitellopsis obtusa*, with calculation of the 96hLC₅₀. The test is performed in Petri dishes.

1. Alga collection from the natural habitats and holding conditions in the laboratory

A freshwater charophyte, *Nitellopsis obtusa* (Desv.) J. Groves, is harvested by kedge anchor at a depth of approximately 5 m in freshwater lakes during vegetation period. The plants are transported to the laboratory in plastic bags filled with lake water. After separation from the bulk by cutting with the sizers, single internodal cells (each cell length 3-10 cm) are kept in 2 L glass vessels filled with 1/2 of tap water (unchlorinated) and 1/2 of lake water. Vessels with the cells are kept under dim light at room temperature, the holding medium has to be changed approximately every two months. So stored cells can be used for biotesting for 4-5 months.

2. Preparation of dilution medium

1. Fill a 1 liter volumetric flask with approximately 800 ml distilled (or deionized) water.
2. Pipet 1 ml of KH₂PO₄, MgSO₄ and Mg(NO₃)₂ from respective concentrated salt (100 mM) solutions and add to 1 liter flask.
3. Pipet 4 ml of CaCl₂ from concentrated salt (100 mM) solution and add to 1 liter flask.
4. Pipet 10 ml of NaHCO₃ from concentrated salt (100 mM) solution and add to 1 liter flask.
5. Add distilled water up to the 1000 ml mark and shake to homogenize the medium.

The pH of the medium should be 7-7.4. No buffers are added.

3. Preparation of toxicant dilution series

A dilution series of the effluent sample is prepared according to standard dilution procedures prescribed for range-finding or definitive tests. It is required 3x50 ml of the solution for each dilution and controls (three replicates, 10 cells per Petri dish).

4. Preadaptation and exposure of the algal cells

The cells are preadapted and exposed to toxicants in Petri dishes in darkness at room temperature (18-24°C).

1. 12-24 hours prior to performing the test, take the cells from the glass vessels and place them into Petri dish with dilution medium, 10 cells/dish (use one additional Petri dish with the cells for the replacement of occasionally dead cells).
2. Mark control and testing dishes of respective concentrations.
3. Change the dilution medium in control dishes.
4. Change the dilution medium by the testing solutions of respective concentrations.
5. Indicate the beginning of the exposure.
6. Change all the solutions in all Petri dishes after 48 hours.

5. Observation of cell survival

Survival of the cells is checked daily by gently picking up each cell with a spatula. Cell death is judged by the disappearance of turgor pressure (the dead cell, when picked up, bends on the spatula and loses its cylindrical shape or even breaks into pieces)..The number of dead cells is recorded daily.

6. Estimation of the LC₅₀

1. After 96 hour exposure, count the number of dead cells in controls and testing dishes.
2. Calculate mortality percentage in controls and respective treatments by using equation:
$$\text{Mortality \%} = (N_{96}/N_0) \times 100\%$$
where N_{96} – a number of dead cells after 96 h, N_0 – initial cell number.
3. Calculate the 96h LC₅₀ using a non-linear (logistic) regression of the mortality percentage with the logarithms of exposure concentration.
4. If undiluted (100 %) effluent sample did not induce more than 50% death of the cells indicate an observed percentage of cell mortality.

7. Validity of the test

For the toxicity test to be acceptable, the mortality in controls should not be larger than 10% at the end of 96 hour exposure.

8. Reference test

1. Make a stock solution of 10 g/l potassium dichromate by weighting 2.5 g of the compound and dissolving it in distilled water in a 250 ml calibrated flask.

2. Prepare five dilutions : C1 (1.2 g/l), C2 (0.9 g/l), C3 (0.6 g/l), C4 (0.45 g/l), and C5 (0.3 g/l).

Mean 96-h LC₅₀ for the reference toxicant potassium dichromate (K₂Cr₂O₇): 0.76 g/l (0,60-0,93).

From the data obtained in the quality control test, a 96-h LC₅₀ has to be calculated, the value of which should be situated within the limits.

Reference

Manusadžianas L, Maksimov G, Darginavičienė J, Jurkonienė S, Sadauskas K, Vitkus R. 2002. Response of the charophyte *Nitellopsis obtusa* to heavy metals at the cell, cell membrane, and enzyme levels. *Environmental Toxicology* **17**:275-283.

5 Results of chemical analysis of waste water and sludge

5.1 Basic parameters

Flow rate

The flow rate in different treatment plants varied considerably, due to their size, handling capacity as well as seasonal fluctuations. At two MWWTP's the highest flow rates were observed in the spring and in the late autumn, when evaporation is low and snow covers are beginning to melt or form.

The highest flow rates in overall were observed at MWWTP1 and MWWTP2, the biggest treatment plants with the highest handling capacity (occasionally ca. $>17000 \text{ m}^3/\text{day}$ at MWWTP1 and ca. $>11000 \text{ m}^3/\text{day}$ at MWWTP2) (Figure 3, Figure 4). During 2009 May – 2010 August sampling period the total amount of the treated effluents was 1,6 million m^3 at MWWTP1 and 2,1 million m^3 at MWWTP2, respectively. Sampling dates are marked with red bars in the figures.

In case of industrial waste water treatment plans the lowest flow rates observed at IWWTP1 (max. approximately $600 \text{ m}^3/\text{day}$) (Figure 5). The flow rates reached approx. $2600 \text{ m}^3/\text{day}$ at IWWTP2 (Figure 6). The total effluent amount was about 0,5 million m^3 at IWWTP2 and 0,2 million m^3 at IWWTP1 that was $\frac{1}{4}$ and $\frac{1}{10}$ of capacity of MWWTP2.

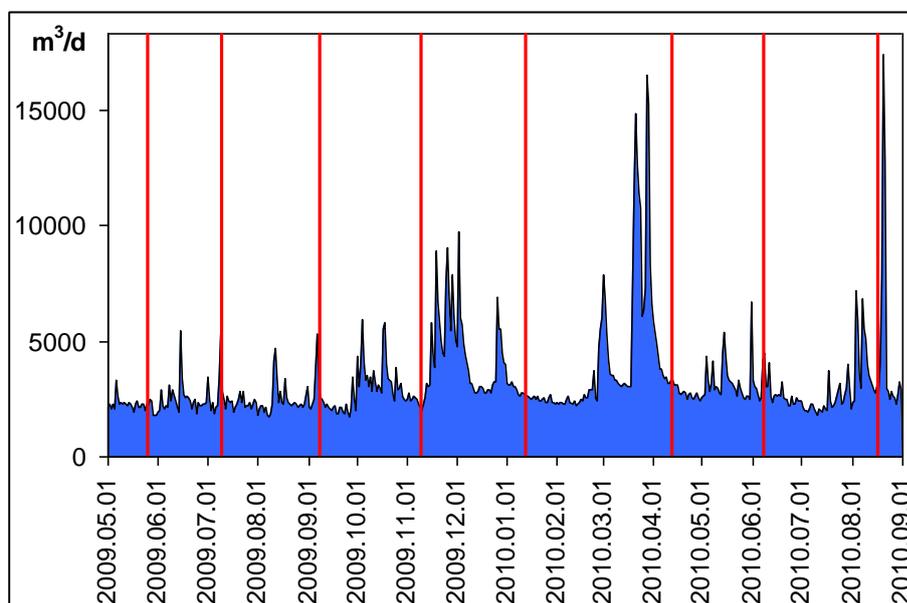


Figure 3. The daily flow rate during the years 2009 and 2010 in MWWTP1.

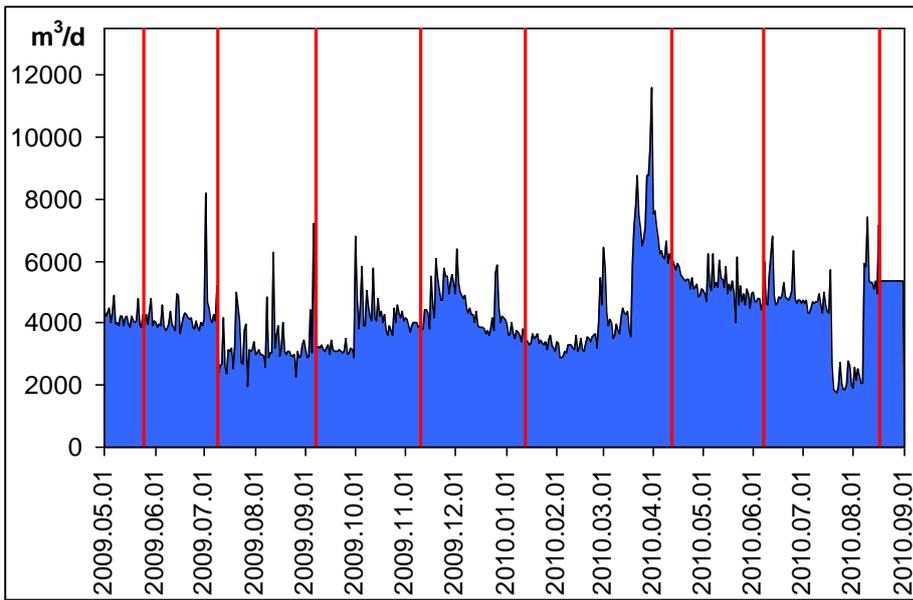


Figure 4. The daily flow rate during the years 2009 and 2010 in MWWTP2.

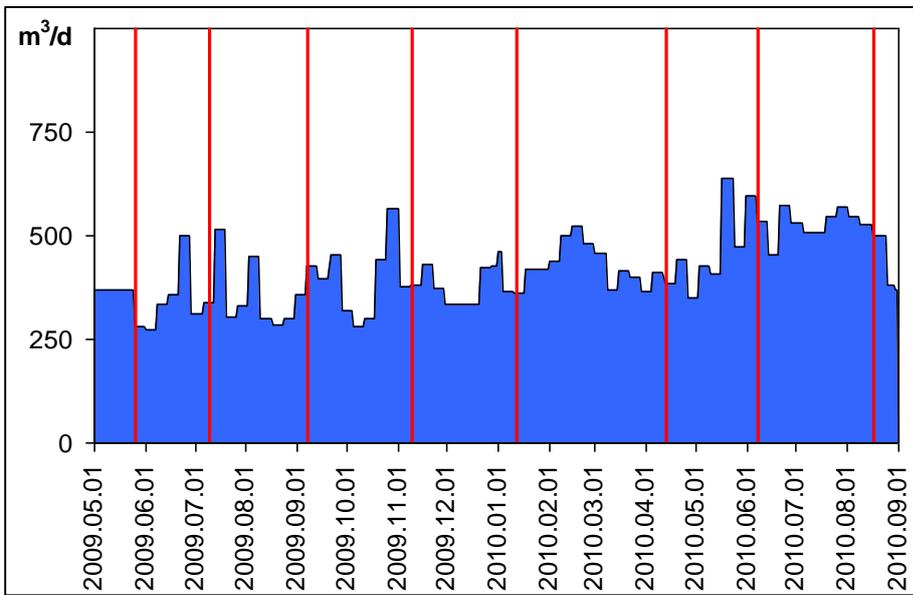


Figure 5. The daily flow rate during the years 2009 and 2010 in IWWTP1.

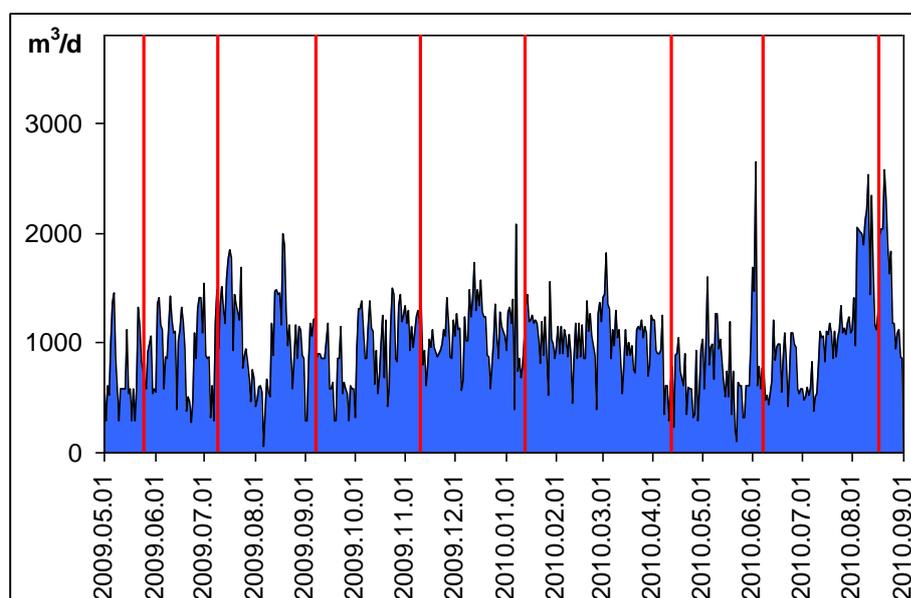


Figure 6. The daily flow rate during the years 2009 and 2010 in IWWTP2.

Other basic parameters

The other basic data measured from the effluents were pH, conductivity, temperature, biological oxygen demand (BOD_7), chemical oxygen demand (COD_{Cr}), suspended solids, total phosphorus (Tot-P), phosphates ($PO_4\text{-P}$), total nitrogen (Tot-N), ammonium ($NH_4\text{-N}$), total organic carbon (TOC), sulphates (SO_4^{2-}) and iron (Fe). The minimum-maximum and median values of basic parameters from the COHIBA samplings are presented in the Table 4. The limit values for basic parameters [12] are presented in Annex I.

It can be seen that the highest concentrations of TOC, BOD_7 , COD_{Cr} , suspended solids and sulphates were determined at IWWTP1.

The relatively large fluctuations in concentrations of total phosphorus (from 0,42 to 8,40 mgP/l) and phosphates (from 0,30 to 5,70 mgP/l) were determined at MWWTP2. High concentrations of these parameters were resulted by failure of biological treatment in the plant.

Table 4. Median, average and max-min values of basic parameters from the WWTPs effluents based on COHIBA samplings.

Parameter		MWWTP1	MWWTP2	IWWTP1	IWWTP2
BOD_7 (mg/l)	median	6,00	5,50	176,5	1,40
	average	6,53	136,5	317,9	1,40
	(min-max)	(3,40-14,0)	(<3,0-664)	(56,0-1025)	(1,00-1,80)
COD_{Cr} (mg/l)	median	48,5	49,0	589,0	39,0
	average	48,0	128,6	794,8	42,0
	(min-max)	(26,0-75,0)	(26,0-730)	(167,0-1910)	(25,0-75,0)
Suspended solids (mg/l)	median	7,20	5,50	38,0	<1,80
	average	7,55	21,3	44,1	1,46
	(min-max)	(2,00-16,0)	(2,80-128)	(5,60-108,0)	(<1,8-2,00)
Tot-P (mgP/l)	median	0,33	2,26	1,36	0,18
	average	0,46	2,58	1,42	0,19
	(min-max)	(0,18-0,96)	(0,42-8,40)	(0,20-3,36)	(0,13-0,30)
$PO_4\text{-P}$ (mgP/l)	median	0,04	2,01	0,30	0,17
	average	0,19	2,15	0,55	0,16

Parameter		MWWTP1	MWWTP2	IWWTP1	IWWTP2
	(min-max)	(0,01-0,66)	(0,30-5,70)	(0,01-1,70)	(0,10-0,25)
Tot-N (mgN/l)	median	12,5	10,9	8,70	4,09
	average	16,4	14,3	7,85	5,20
	(min-max)	(3,93-46,3)	(2,87-45,0)	(3,12-15,1)	(2,56-11,1)
NH ₄ -N (mgN/l)	median	8,30	0,34	1,15	0,15
	average	15,0	4,22	1,16	0,21
	(min-max)	(0,04-45,5)	(0,02-32,0)	(0,03-2,39)	(0,10-0,52)
Alkalinity (mmol/l)	median	7,98	7,12	3,91	3,30
	average	8,09	7,54	4,98	3,25
	(min-max)	(5,11-9,72)	(5,07-11,4)	(2,97-7,74)	(2,13-3,97)
pH	median	7,5	7,1	7,3	7,3
	average	7,5	7,1	7,7	7,3
	(min-max)	(7,30-7,85)	(6,75-7,37)	(6,96-9,00)	(7,01-7,43)
Conductivity (mS/m)	median	124,9	130,1	125,5	154,5
	average	120,5	126,3	124,9	200,9
	(min-max)	(84,4-148,0)	(56,5-174)	(44,8-238)	(81,2-514)
Fe (mg/l)	median	0,09	0,05	0,18	0,07
	average	0,10	0,21	0,15	0,10
	(min-max)	(0,06-0,16)	(0,03-1,30)	(<0,02-0,26)	(0,03-0,27)
t (°C)	median	14,0	16,0	26,0	24,0
	average	13,1	14,4	25,3	24,9
	(min-max)	(4,00-22,0)	(7,00-20,0)	(4,00-35,0)	(22,0-33,0)
SO ₄ ²⁻ (mg/l)	median	73,2	72,0	129,1	69,0
	average	75,9	67,1	122,9	60,4
	(min-max)	(64,0-90,0)	(35,0-114)	(27,0-278,0)	(25,6-90,0)
TOC (mgC/l)	median	10,3	9,52	195,5	8,02
	average	10,2	30,3	241,1	8,00
	(min-max)	(8,38-12,1)	(7,48-176)	(69,5-543)	(6,08-10,0)

5.2 Metals

In the frame of the COHIBA project analyses of cadmium and mercury were performed in waste water, sludge, landfill leachate and storm water samples. The analyses of metals were carried out by the Environmental Protection Agency.

The concentration of cadmium was below the limit of quantification, i.e. 0,050 µg/l, in all waste water and landfill leachate samples. Cadmium values in sludge samples taken at MWWTP2 were at the range from 0,43 to 0,52 mg/kg.

In most cases, the concentrations of mercury were below the limit of quantification of the method, i.e. 0,020 µg/l, in waste water, landfill leachate and storm water samples. Mercury concentrations determined in various samples are presented in Figure 7. The concentrations which were below the limit of quantification of the method are marked with unfilled bars. It can be seen from the Figure 7 that negligible amounts of mercury were found in the waste water samples taken at MWWTP1, IWWTP1, IWWTP2 and SW. In 75% of the samples, the concentrations of mercury were below the limit of quantification.

Determined concentrations of mercury in sludge samples taken at MWWTP2 were at the range of 0,17 – 0,25 mg/kg.

According to Lithuanian Waste water management regulation [12] the maximum allowable concentration (MAC) to the environment should not exceed for mercury 2 µg/l and for cadmium - 10 µg/l. The results of performed analyses showed that the concentrations of mercury and cadmium did not exceed the limit value at any monitored point in any sampling campaign.

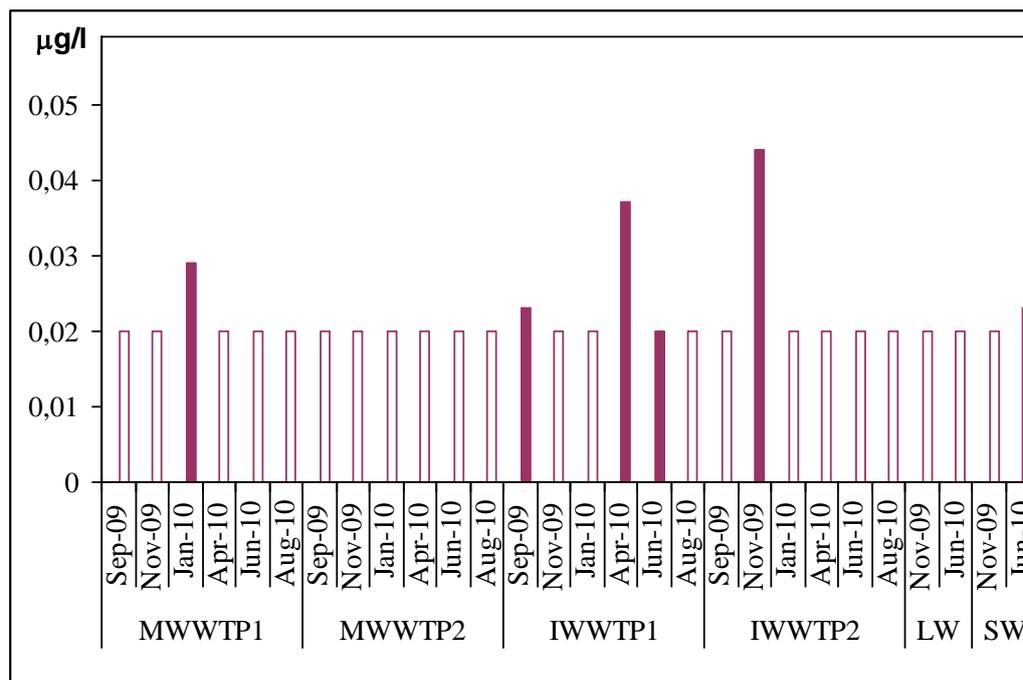


Figure 7. The concentrations of mercury in various samples.

5.3 Organotin

In the frame of the COHIBA project analyses of selected organotin compounds (monobutyltin (MBT), dibutyltin (DBT), tributyltin (TBT), tetrabutyltin (TTBT), mono-octyltin (MOT), dioctyltin (DOT), triphenyltin (TPhT) and tricyclohexyltin (TCyT)) were performed in waste water, sludge, landfill leachate and storm water samples. The analysis of organotin compounds were carried out by the Swedish Environmental Research Institute (IVL).

Observed concentrations of organotin compounds are presented in Figure 8. It should be noted that MBT was determined in all samples, except storm water. In the investigated period the obtained concentrations of MBT were at range from <1,0 to 59,0 ng/l. The highest concentration of MBT cation was determined in landfill leachate sample taken in June 2010. The recorded concentration of MBT in the sample was 59 ng/l. The highest mean value (10,4 ng/l) of MBT was observed at MWWTP1. The mean value of MTB was 6,1 ng/l at MWWTP2 and, respectively, 1,8 ng/l at IWWTP2. It can be seen from the Figure 8 MBT was not detected in waste water samples taken during the 1st and 2nd sampling campaigns (September and November 2010). In 64% of the samples MBT cation concentrations were below the limit of quantification.

Dibutyltin was found in the waste water samples taken at MWWTP1, MWWTP2 and IWWTP1. The concentrations of DBT were at the range 0,97 – 1,4 ng/l, the highest concentration of DBT (1,4 ng/l) was determined at IWWTP1. The concentration of DBT cation was below the limit of quantification both in storm water and landfill leachate samples as well as waste water samples taken at IWWTP2. In 75% of the samples DBT cation concentrations were below the limit of quantification.

Monoctyltin was determined only in single case – in landfill leachate sample taken in June 2010. The observed concentration of MOT was 9,3 ng/l.

All organotin compounds were determined in sludge samples taken at MWWTP2, except triphenyltin. The concentration of TPhT was below the quantification limit, i.e. 1 µg/kg. Considering the results of organotins in sludge samples taken in January and June 2010 it is evident that determined concentrations are comparable (Appendix C). The mean values of organotins were as follow: MBT – 450 µg/kg, DBT – 215 µg/kg, TBT – 3,95 µg/kg, MOT – 160 µg/kg and DOT – 82,5 µg/kg.

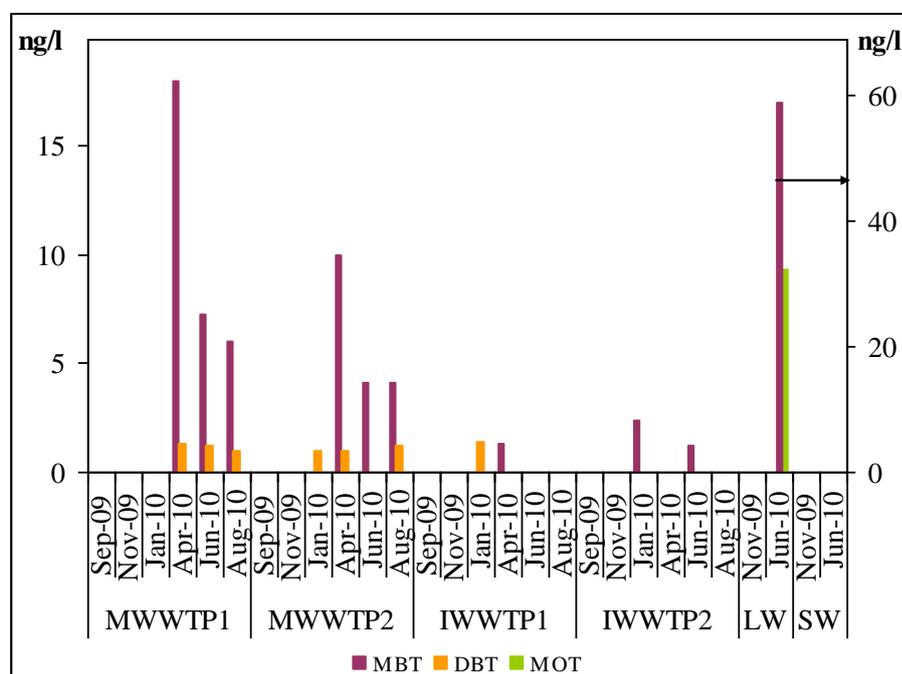


Figure 8. The concentrations of organotin compounds in various samples.

According to Lithuanian Waste water management regulation [12] the maximum allowable concentration (MAC) of tributyltin cation discharged into the environment should not exceed 20 ng/l. It should be noted that the MAC limit value for TBT was not exceeded.

5.4 Phenolic substances

In the frame of the COHIBA project analyses of selected phenolic substances: bisphenol A, 4-nonylphenol mix. (4-NP), 4-nonylphenol monoethoxylate mix. (4-NPEO1), 4-nonylphenol diethoxylate mix. (4-NPEO2), octylphenol (OP), octylphenol monoethoxylate (OPEO1) and octylphenol diethoxylate (OPEO2) were performed. The analyses of phenolic substances were carried out by the Finnish Environment Institute (SYKE).

Measured concentrations of alkylphenols and their ethoxylates are given in the Appendixes B-G. The results which exceeded limit of detection but were below limit of quantification are marked with red colour in bold. The values below limit of detection are marked as nd (=not detected).

Determined concentrations of 4-NP varied from <0,10 µg/l to 0,75 µg/l. It can be seen from Figure 9 the highest concentration of 4-NP was observed in sample taken at MWWTP1. The concentration was 0,75 µg/l. The mean values (medians) of 4-NP were as follow: 0,35 µg/l at MWTTP1, 0,22 µg/l at MWWTP2, 0,33 µg/l at IWWTP1, 0,24 µg/l at IWWTP2 and 0,22 µg/l landfill leachate, and medians, respectively: 0,22 µg/l at MWTTP1 and 0,17 µg/l at MWWTP2. 4-NP was

found only once in storm water sample taken in November 2009 (0.19 µg/l). In 82% of the samples 4-NP concentrations were below the limit of quantification.

Observed concentrations of 4-NPEO1 were at the range from 0,05 µg/l to 0,11 µg/l. The highest concentration of analyte was 0,11 µg/l at MWWTP1. The highest mean value (0,09 µg/l) was observed at MWWTP1, whereas the mean values of 4-NPEO1 at MWWTP2, IWWTP1 and IWWTP2 were comparable and varied from 0,05 µg/l to 0,06 µg/l. 4-NPEO1 was not detected in landfill leachate and storm water samples. It should be pointed out that all recorded concentrations of 4-NPEO1 were below the limit of quantification. In 64% of the samples the values were below the limit of detection.

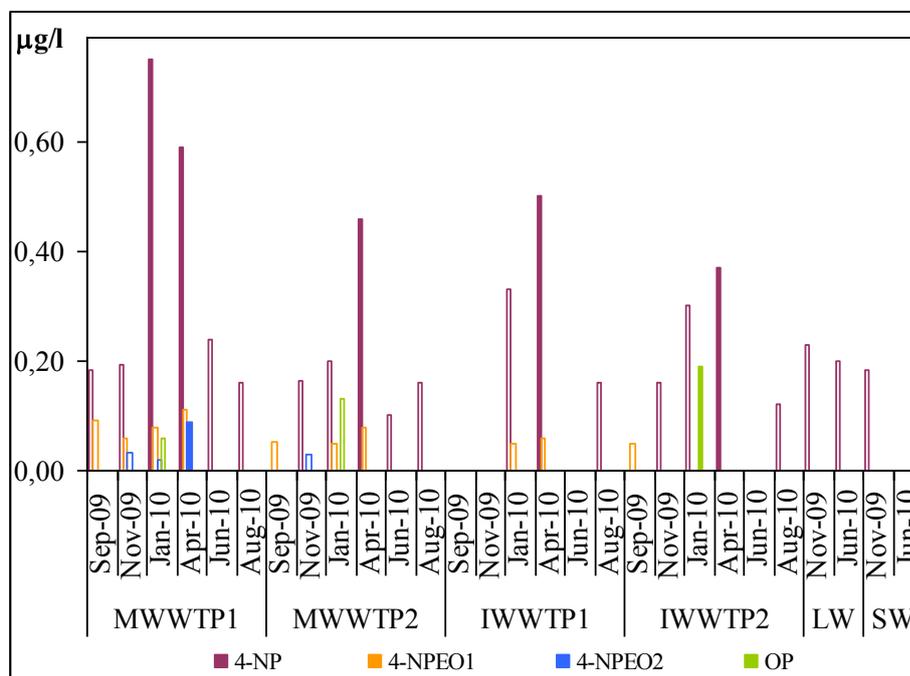


Figure 9. The concentrations of alkylphenols and ethoxylates.

4-NPEO2 was found in all samples taken at MWWTP1 while at MWWTP2 it was measured in only one sample taken in November 2009. The concentrations of 4-NPEO2 were at range from 0,02 µg/l to 0,09 µg/l. The highest concentration (0,09 µg/l) was measured at MWWTP1 and calculated mean value was 0,05 µg/l. The concentrations of 4-NPEO2 were below the limit of quantification in all determinations, except case at MWWTP1 (0,09 µg/l, April 2010).

In the investigated period OP was registered in 2 samples taken at MWWTP1 and MWWTP2 (January 2010). Determined concentrations were below the limit of quantification. Octylphenols ethoxylates were not detected in any water sample.

The results of bisphenol A are presented in Figure 10. It can be seen that in the majority of cases the concentration of the analyte was below the limit of quantification. The concentrations below the limit of quantification were marked with unfilled bars.

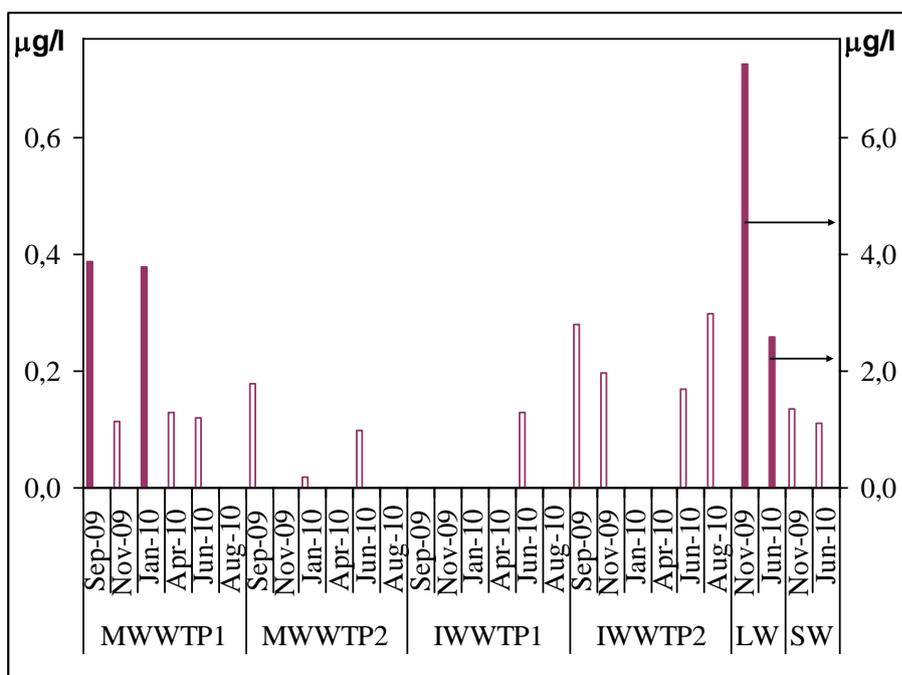


Figure 10. The concentrations of bisphenol A.

Determined concentrations of bisphenol A varied from 0,02 µg/l to 7,27 µg/l. The highest concentrations of bisphenol A (7.27 µg/l and 2.59 µg/l) were determined in landfill leachate samples.

The mean values of bisphenol A are as follow: 0,23 µg/l at MWTTP1, 0,10 µg/l at MWWTP2, 0,24 µg/l at IWWTP2, 4,93 µg/l in landfill effluent and 0,12 µg/l in storm water. Bisphenol A was determined in solitary instance, in waste water sample taken at IWWTP1 (June 2010) and measured concentration was 0,13 µg/l.

It should be noted that in 39% of the samples bisphenol A concentrations were below the limit of detection; in 86% of the samples the results were below the limit of quantification.

All phenolic substances were determined in sludge samples taken at MWWTP2, except bisphenol A (Appendix C). The mean values of the substances are as follow: 4-NP – 2,62 mg/kg, 4-NPEO1 – 0,44 mg/kg, 4-NPEO2 – 0,67 mg/kg and OP – 0,20 mg/kg. OPEO1 and OPEO2 were found only once in the sample taken in January 2010 (the concentrations 0,03 mg/kg and, respectively 0,31 mg/kg).

According to Lithuanian Waste water management regulation [12] the maximum allowable concentration (MAC) to the environment for 4-nonylphenol and octylphenol should not exceed 20 µg/l. The results of performed analyses showed that the concentrations of 4-nonylphenol and octylphenol did not exceed the limit values at any monitored point in any sampling campaign.

5.5 Pesticides

The analyses of pesticides were carried out by the Environmental Protection Agency.

The results of pesticides are given in Appendixes B-G. α -endosulfan, β -endosulfan and endosulfan sulphate concentrations found in waste water, landfill leachate and storm water samples were below the limit of quantification in all monitored points. The limit of quantification for both α -endosulfan and β -endosulfan is 0,004 µg/l, and for endosulfan sulphate - 0,005 µg/l.

It should be noted that determined amounts of pesticides in sludge samples taken at MWWTP2 were below 0,001 mg/kg, i.e. below the limit of quantification of the method, except α -endosulfan. α -endosulfan concentration was found at 0,0022 mg/kg.

According to Lithuanian Waste water management regulation [12] the maximum allowable concentration (MAC) of endosulfan to the environment is not defined.

5.6 Dioxins, furans and PCBs

The analyses of dioxins, furans and dioxin-like compounds were carried out by National Institute for Health and Welfare (THL).

Dioxins are generally found in mixtures containing several kinds of dioxins and dioxin-like compounds, each having its own degree of toxicity. International Toxic Equivalents (TEQ) expresses the overall toxicity of such a mixture as a single number.

TEQ method weighs the toxicity of the less toxic compounds as fractions of the toxicity of the most toxic TCDD. Each compound is credited a specific Toxic Equivalency Factor (TEF) which indicates the degree of toxicity compared to 2,3,7,8-TCDD, which is given a reference value of 1. The TEF values are given in Table 5.

Table 5. Toxic Equivalency Factors (TEF) for dioxins and dioxin-like compound [10].

Compound		WHO 2005 TEF
Chlorinated dibenzo-p-dioxins	2,3,7,8-TCDD	1
	1,2,3,7,8-PeCDD	1
	1,2,3,4,7,8-HxCDD	0,1
	1,2,3,6,7,8-HxCDD	0,1
	1,2,3,7,8,9-HxCDD	0,1
	1,2,3,4,6,7,8-HpCDD	0,01
	OCDD	0,0003
Chlorinated dibenzofurans	2,3,7,8-TCDF	0,1
	1,2,3,7,8-PeCDF	0,03
	2,3,4,7,8-PeCDF	0,3
	1,2,3,4,7,8-HxCDF	0,1
	1,2,3,6,7,8-HxCDF	0,1
	1,2,3,7,8,9-HxCDF	0,1
	2,3,4,6,7,8-HxCDF	0,1
	1,2,3,4,6,7,8-HpCDF	0,01
	1,2,3,4,7,8,9-HpCDF	0,01
	OCDF	0,0003
Non-ortho substituted PCBs	PCB 77	0,0001
	PCB 81	0,0003
	PCB 126	0,1
	PCB 169	0,03
Mono-ortho substituted PCBs	PCB 105	0,00003
	PCB 114	0,00003
	PCB 118	0,00003
	PCB 123	0,00003
	PCB 156	0,00003
	PCB 157	0,00003
	PCB 167	0,00003
	PCB 189	0,00003

For example, to calculate the total TCDD toxic equivalent of a dioxin mixture, the amounts of each toxic compound are multiplied by their TEF and then added together.

In the majority of cases the concentrations of individual congeners for PCDDs and PCDFs were below the limit of quantification (93% of assays (the total number being 315)) (see Appendixes B-G). In 44% of samples determined concentrations of dioxin-like compounds (PCBs and Co-PCBs) were below the limit of quantification.

Only concentrations of 2,3,7,8-TCDD (MWWTP1, IWWTP1), OCDD (MWWTP1, MWWTP2), 1,2,3,4,7,8-HxCDF (IWWTP1) and 1,2,3,4,6,7,8-HpCDF (IWWTP1) were above the limit of quantification. Measured concentrations of 2,3,7,8-TCDD were at range 0.1396-0.4818 pg/l, OCDD – 0.4329-1.2420 pg/l, 1,2,3,4,6,7,8-HpCDF – 0.4496-0.5359 pg/l.

Among CO-PCBs congeners the highest concentrations were observed for CO-PCB-77. The concentrations of that congener reached 8,76 pg/l. The majority of other congeners concentrations were below the limit of quantification.

The highest amounts of PCBs were determined in waste water samples taken at MWWTP1 and IWWTP1 (PCBs sum above 2 ng/l). The mean values of PCBs sum are comparable at MWWTP2 and IWWTP2.

In sludge sample taken at MWWTP2 in June 2010 only the concentrations of 1,2,3,4,7,8-HxCDD, 1,2,3,7,8,9-HxCDF, PCB 122 and PCB 189 were below the limit of quantification. The results are presented in Appendix C. The concentrations of dioxins varied from 0.3827 pg/g (2,3,7,8-TCDD) to 171.27 pg/g (OCDD), respectively, for furans – from 0.4133 pg/g (1,2,3,7,8-PeCDF) to 28.37 pg/g (OCDF). The concentrations of CO-PCBs varied from 2.2127 pg/g to 179,40 pg/g, respectively for PCBs – from 0.0225 ng/g (PCB 123) to 4.2842 ng/g (PCB 28/31).

5.7 Chloroparaffins (SCCP and MCCP)

Analyses of short chain chlorinated paraffins C₁₀-C₁₃ (SCCP) and medium chain chlorinated paraffins C₁₄-C₁₇ (MCCP) were carried out in all samples. Measurements of MCCP started from November 2009, i.e. from the 2nd sampling campaign. The analyses of SCCP were carried out by the Institute for Ecology of Industrial Areas (IETU), whereas analyses of MCCP were performed in Institute of Non-Ferrous Metals.

The results of SCCP analyses are presented in Figure 11. Short chain chlorinated paraffins were determined in all samples. Obtained concentrations of SCCP varied from 0.14 µg/l to 1.95 µg/l. Both the highest concentration value of SCCP (1.95 µg/l) and the highest mean value (1.14 µg/l) was found in sample taken at MWWTP1, whereas the lowest mean values were recorded at IWWTP1 (0.73 µg/l) and storm water sample (0.75 µg/l). The mean value of SCCP in samples taken at MWWTP2 and landfill sample are 0.83 µg/l.

It should be noted that the concentration of SCCP in storm water sample taken in November 2009 is approximately 3 times lower (0.36 µg/l) than in June 2010 (1.14 µg/l). The opposite situation was observed in the case of sludge sample analysis from MWWTP2 (Appendix C). The concentration of SCCP was about 2 times higher in sludge sample taken during the cold period (11,6 mg/kg, January 2010) in comparison with sample taken during the warm period (4.95 mg/kg, June 2010).

According to Lithuanian Waste water management regulation [12] the maximum allowable concentration (MAC) of C₁₀-C₁₃ chloroalkanes to the environment should not exceed 2 µg/l. The carried out research showed that the MAC limit value was not exceeded in all monitored sites.

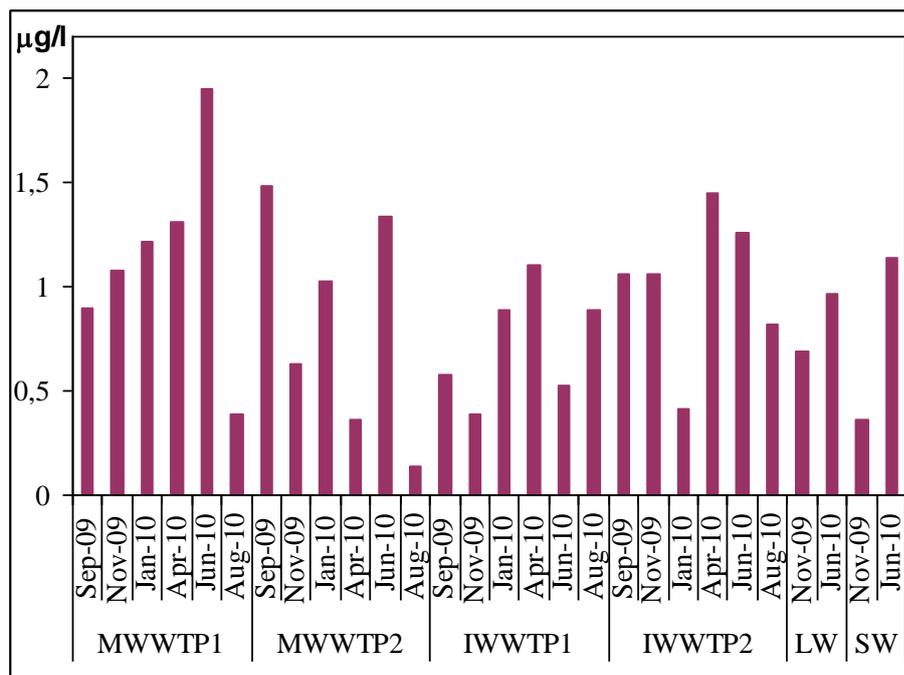


Figure 11. The concentrations of short chain chlorinated paraffins (SCCP).

Measured concentrations of medium chain chlorinated paraffins are shown in Figure 12. Observed concentrations of MCCP were at range from <0.6 µg/l to 31.5 µg/l. The highest concentration of MCCP (31.5 µg/l) was found in waste water from MWWTP1, whereas the lowest value (<0.6 µg/l) was recorded in the sample taken at MWWTP2. Comparing the results it is evident that the highest means values were found in samples taken at municipal waste water treatment plans (8.32 µg/l at MWWTP1 and 4.36 µg/l at MWWTP2). The mean values at industrial waste water treatment plans, storm water and leachate sample were comparable and varied from 2.25 µg/l to 3.11 µg/l.

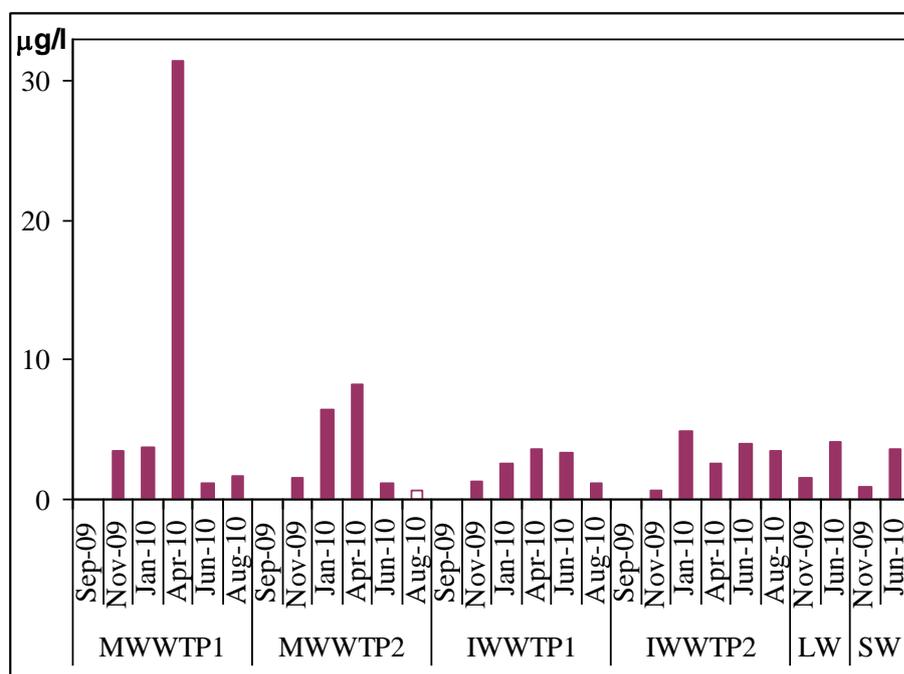


Figure 12. The concentrations of medium chain chlorinated paraffins (MCCP).

Measured concentrations of MCCP varied from <0.03 mg/kg (June 2010) to 0.123 mg/kg (January 2010) in sludge samples taken at MWWTP2.

5.8 Brominated flame retardants

The analyses of brominated flame retardants were carried out by the Finnish Environment Institute (SYKE).

Pentabrominated diphenyl ethers (pentaBDE). The sum of pentabrominated diphenyl ethers includes the following congeners: 2,2',4,4'-tetrabrominated diphenyl ether (BDE-47), 2,2',3,4,4'-pentabrominated diphenyl ether (BDE-85), 2,2',4,4',5-pentabrominated diphenyl ether (BDE-99), 2,2',4,4',6-pentabrominated diphenyl ether (BDE-100), 2,2',4,4',5,5'-hexabrominated diphenyl ether (BDE-153) and 2,2',4,4',5,6'-hexabrominated diphenyl ether (BDE-154).

Concentrations of pentabrominated diphenyl ethers obtained during measurement campaigns are shown in Figure 13 and Appendixes B-G. The values below the limit of detection were marked as nd (=not detected) in tables while the concentrations below the limit of quantification were marked with unfilled bars in the chart. Taking all monitored sites into consideration it can be seen that measured concentrations of pentaBDE were at range from <0.03 ng/l to 4.98 ng/l. The highest concentration (4.98 ng/l) was measured in the waste water sample taken at IWWTP1 (September 2009). The mean values of pentaBDE are as follow: 0.19 ng/l at MWTTP1, 0.28 ng/l at MWWTP2, 1.78 ng/l at IWWTP1 and 0.10 ng/l in storm water.

In 84% of samples the concentrations of pentaBDE were below the limit of detection.

According to Lithuanian Waste water management regulation [12] the maximum allowable concentration (MAC) of pentabrominated diphenyl ethers to the environment is not defined.

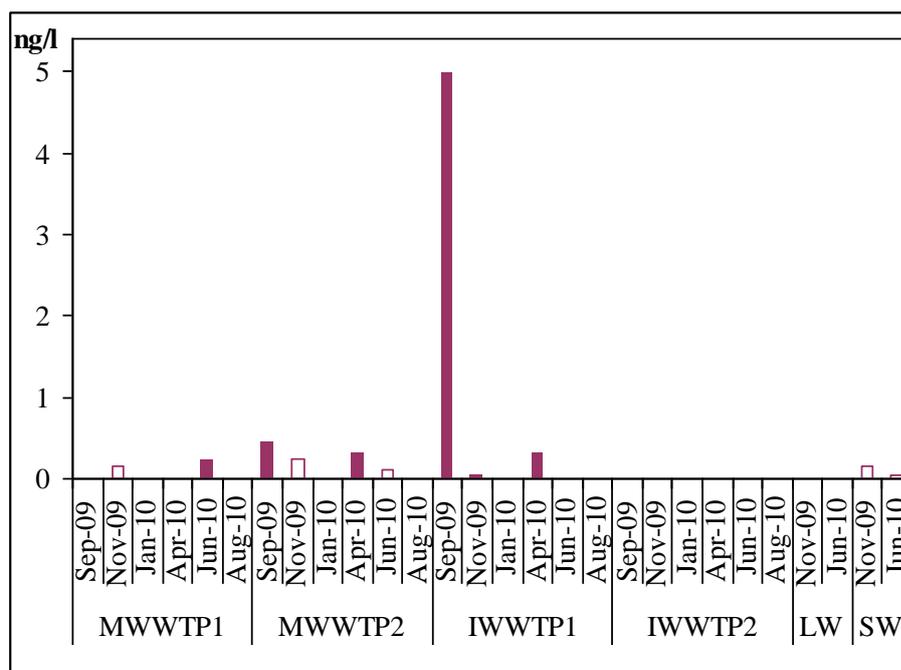


Figure 13. The concentrations of pentabrominated diphenyl ethers.

Octabrominated diphenyl ethers (octaBDE). The sum of octabrominated diphenyl ethers includes the following congeners: 2,2',3,4,4',5',6-heptabromodiphenyl ether (BDE-183) and 2,2',3,4,4',5,5',6-octabromodiphenyl ether (BDE-203). The concentrations of octaBDE are illustrated in Figure 14. It should be noted that octaBDE were determined only once at IWWTP1 (September 2009). Observed

concentration was 14.32 ng/l. In 96% of samples the concentration of octaBDE did not exceed the limit of detection, i.e. 0.03 ng/l.

Decabrominated diphenyl ethers (decaBDE). The results of decabrominated diphenyl ethers (2,2',3,3',4,4',5,5',6,6'-decabrominated diphenyl ether (BDE-209)) are presented in Figure 14. The concentrations of decaBDE varied at range from <0.03 ng/l to 10.24 ng/l, the mean value was 1.79 ng/l and median was 0.23 ng/l. The highest concentrations of the analyte (above 10 ng/l) were determined in samples taken at IWWTP1 (January and April 2010).

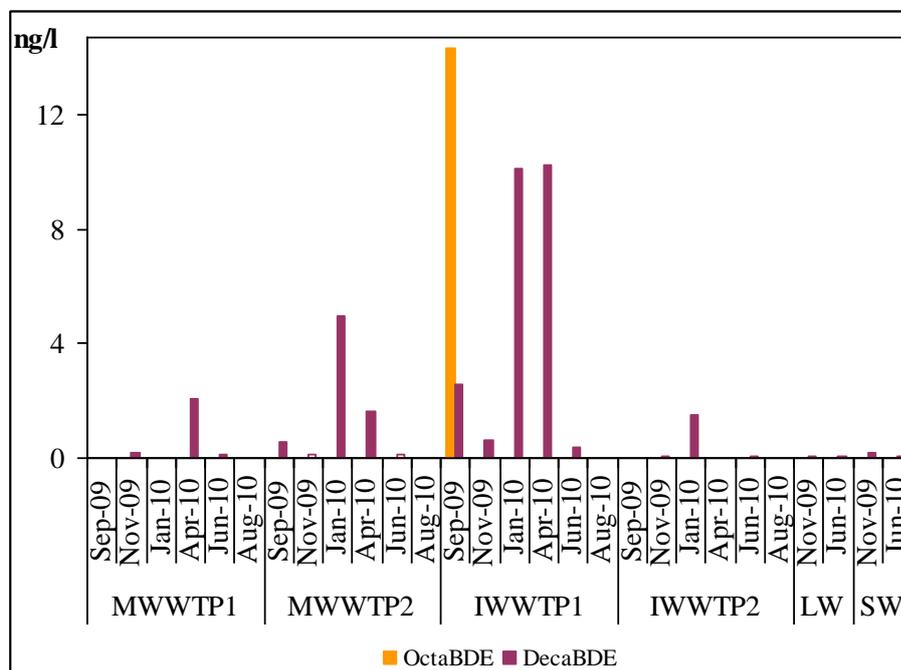


Figure 14. The concentrations of octabrominated and decabrominated diphenyl ethers.

The mean values of decaBDE are as follow: 0.80 ng/l at MWWTP1, 1.48 ng/l at MWWTP2, 4.79 ng/l at IWWTP1, 0.56 ng/l at IWWTP2, 0.075 ng/l in landfill effluent and 0.14 ng/l in storm water. In 31% the samples the concentration of decaBDE was below the limit of quantification.

In sludge samples taken at MWWTP2 the mean values of polybrominated diphenyl ethers were as follow: 18.72 µg/kg for pentaBDE, 2.09 µg/kg for octaBDE and 240 µg/kg for decaBDE.

Hexabromocyclododecanes (HBCD). 3 isomers of hexabromocyclododecanes were determined: α-HBCD, β-HBCD and γ-HBCD. The concentrations of isomers are presented in Appendixes B-G and Figure 15.

Observed concentrations of α-HBCD were at range from 0.02 ng/l to 32.1 ng/l, β-HBCD – from 0.03 ng/l to 7.27 ng/l and γ-HBCD – from 0.23 ng/l to 72.87 ng/l. The highest concentrations of the isomers were measured in waste water sample taken at IWWTP1 (January 2010). The mean values of hexabromocyclododecanes (α-HBCD – approximately 0.50 ng/l, β-HBCD – approximately 0.23 ng/l and γ-HBCD – 0.88 ng/l) were comparable in the samples taken at municipal waste water treatment plants. The highest mean values were at IWWTP1. It should be pointed that HBCD were not detected in landfill leachate samples.

In sludge sample taken at MWWTP2 the concentrations of HBCD isomers varied as follows: α-HBCD from 32.1 µg/kg to 89.1 µg/kg, β-HBCD – 4.3-11.5 µg/kg and γ-HBCD – 14.8-28.5 µg/kg. Higher concentrations of β-HBCD and γ-HBCD were determined in the sample taken in June 2010.

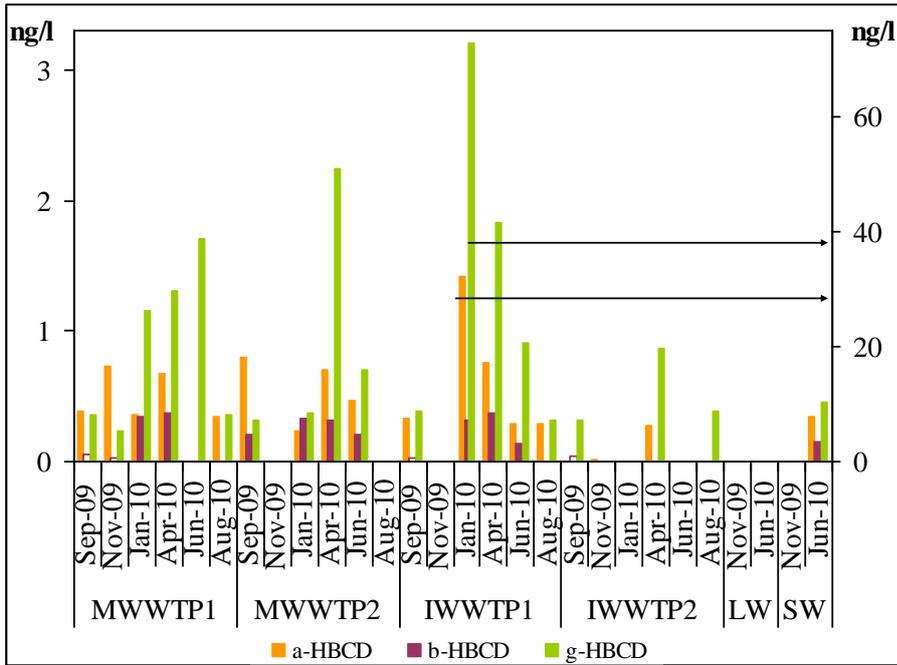


Figure 15. The concentrations hexabromocyclodecanes.

5.9 Perfluorinated compounds

The analyses of perfluorinated compounds were carried out by the Finnish Environment Institute (SYKE).

Analyses of selected perfluorinated compounds: perfluoro-n-hexanoic acid (PFHxA), perfluorooctane sulfonate (PFOS), perfluorooctanoic acid (PFOA) and perfluoro-n-decanoic acid (PFDA) were carried out in all samples.

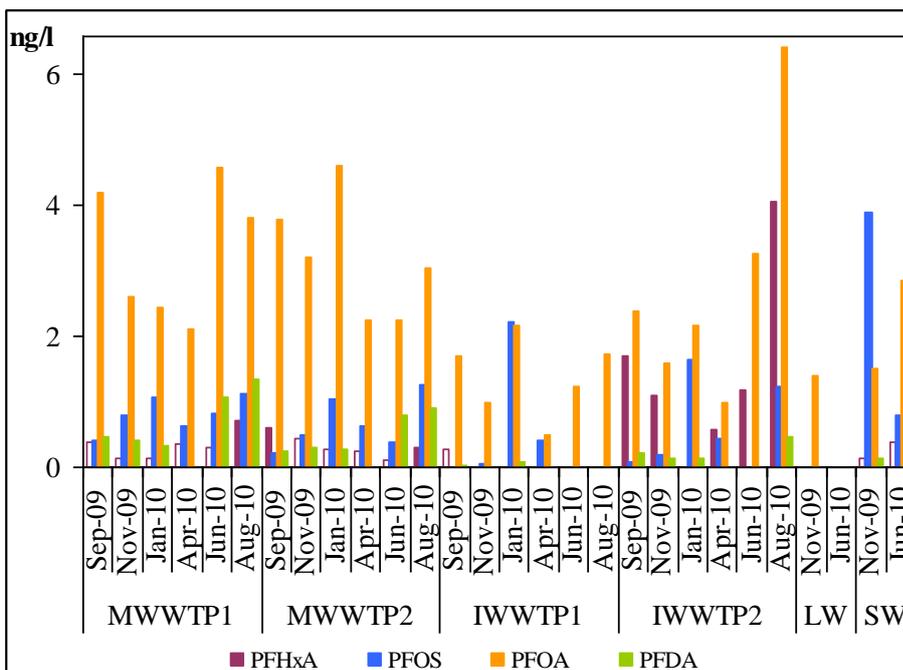


Figure 16. The concentration of perfluorinated compounds.

The concentrations of perfluorinated compounds are illustrated in Figure 16 and given Appendixes B-G. Taking all monitored points into consideration it can be seen that determined concentrations of PFHxA varied from 0,11 ng/l to 4,06 ng/l. The highest concentration of the analyte (4,06 ng/l) was determined in sample taken at IWWTP2.

The mean values (medians) of PFHxA are as follow: 0,34 ng/l (0,33 ng/l) at MWWTP1, 0,33 ng/l (0,29 ng/l) at MWWTP2 and 1,72 ng/l (1,18 ng/l) at IWWTP2. PFHxA was not detected in landfill leachate samples. The mean value of PFHxA is 0,26 ng/l in storm water samples. In 29% of samples PFHxA concentrations were below the limit of detection and 43% of the results were above the limit of detection but below the limit of quantification.

Measured concentrations of PFOS were at range from 0,06 ng/l to 3,90 ng/l. The highest concentration of PFOS (3,90 ng/l) was determined in storm water sample taken in November 2009, whereas the lowest value (0,06 ng/l) was found at IWWTP1.

The mean values of PFOS are as follow: 0.81 ng/l at MWWTP1, 0.67 ng/l at MWWTP2, 0.89 ng/l at IWWTP1, 0.72 ng/l at IWWTP2 and 2.35 ng/l in storm water, respectively, medians are 0.81 ng/l (MWWTP1), 0.56 ng/l (MWWTP2) and 0.44 ng/l (IWWTP2). PFOS was not detected in landfill leachate samples. In 21% of the samples the results were below the limit of detection.

Observed concentrations of PFOA were at range from 0.48 ng/l to 6.43 ng/l. The highest concentration (6.43 ng/l) was determined at IWWTP2. At IWWTP2 and municipal waste water treatment plants the mean values of PFOA were comparable (about 3.00 ng/l).

In monitored sites the concentrations of PFDA varied from 0.04 ng/l to 1.34 ng/l. The highest value (1.34 ng/l) was measured at MWWTP1. Medians and the mean values were comparable at municipal waste water treatment plants. PFDA was not detected in landfill leachate samples. In 36% of samples the results of PFDA were below the limit of detection.

In sludge samples the mean value of perfluorinated compounds were as follow: 3.4 µg/kg for PFOS, 0.7 µg/kg for PFOA and 1.95 µg/kg for PFDA. PFHxA was not detected in the sludge samples.

6 Results of biotests

6.1 Acute and short-term chronic tests

Toxicity data obtained with three obligatory (*V. fischeri*, *D. magna* and *P. subcapitata*) and five optional tests are represented on Table 7. The whole 8-test-battery included 3 algal (unicellular algae *Pseudokirshneriella subcapitata* growth inhibition test, macrophytic algae of *Nitellopsis obtusa* cell depolarization and cell mortality tests), 2 crustacean (*Daphnia magna* immobilization test, fairy shrimp *Thamnocephalus platyurus* mortality test), 1 rotifer *Brachyonus calyciflorus* mortality, 1 protozoa *Tetrahymena thermophila* growth inhibition and 1 marine bacteria *Vibrio fischeri* bioluminescent inhibition tests.

Table 7. Municipal (LT_MWWTP1 and LT_MWWTP2), industrial (LT_IWWTP1 and LT_IWWTP2) effluent, storm water (LT_SW) and landfill water (LT_LW) toxicity data obtained with acute and short-term chronic tests (endpoint values at 50% effect level are expressed as percentage of effluent dilution).

Table 7a
(LT_MWWTP1)

Acute and short-term chronic tests	Duration	End-point	2009 May	2009 Jul	2009 Sep	2009 Nov	2010 Jan	2010 Apr	2010 Jun	2010 Aug
<i>V. fischeri</i>	30-min	EC50	>100	>100	>100	>100	>100	>100	>100	>100
<i>D. magna</i> (1)*	48-h	EC50	<u>100</u>	<u>66.7</u>	>100	>100	>100	>100	>100	>100
<i>D. magna</i> (2)		EC50	>100	>100	>100	>100	>100	>100	>100	>100
<i>P.subcapitata</i> (1)	72-h	ErC50	<u>43.5</u>	<u>90.9</u>	>100	>100	>100	>100	>100	>100
<i>P.subcapitata</i> (2)		ErC50	<u>40.0</u>	<u>71.4</u>	>100	>100	<u>74.3</u>	<u>49.2</u>	>100	>100
Thamnotoxkit F (1)	24-h	LC50	<u>34.5</u>	<u>37.0</u>	>100	>100	<u>98.0</u>	<u>61.0</u>	>100	>100
Thamnotoxkit F (2)		LC50	<u>58.8</u>	<u>55.6</u>	>100	>100	>100	<u>54.5</u>	>100	>100
Charatox	90-min	IC50	<u>12.3</u>	<u>17.2</u>	>100	>100	<u>45.6</u>	<u>31.2</u>	>100	>100
Niteltox	96-h	LC50	<u>47.6</u>	<u>47.6</u>	>100	>100	<u>48.0</u>	<u>23.7</u>	>100	>100
Rotoxkit F	24-h	LC50	>100	>100	>100	>100	>100	>100	>100	>100
Protoxkit F	24-h	EC50	>100	>100	n.a.	>100	>100	>100	>100	>100

* Tests were conducted in two independent laboratories: (1) and (2);

Background in column 1 marks short-term chronic tests.

Table 7b
(LT_MWWTP2)

Acute and short-term chronic tests	Duration	End-point	2009 May	2009 Jul	2009 Sep	2009 Nov	2010 Jan	2010 Apr	2010 Jun	2010 Aug
<i>V. fischeri</i>	30-min	EC50	>100	>100	>100	>100	>100	>100	>100	>100
<i>D. magna</i> (1)	48-h	EC50	>100	>100	>100	>100	>100	>100	>100	>100
<i>D. magna</i> (2)		EC50	>100	>100	>100	>100	>100	>100	>100	>100
<i>P.subcapitata</i> (1)	72-h	ErC50	>100	>100	>100	>100	>100	>100	>100	>100
<i>P.subcapitata</i> (2)		ErC50	>100	>100	>100	>100	>100	>100	>100	>100
Thamnotoxkit F (1)	24-h	LC50	>100	<u>100</u>	>100	>100	>100	>100	>100	>100
Thamnotoxkit F (2)		LC50	>100	<u>58.8</u>	>100	>100	>100	>100	>100	>100
Charatox	90-min	IC50	>100	<u>20.4</u>	>100	>100	>100	>100	>100	>100
Niteltox	96-h	LC50	>100	<u>35.7</u>	>100	>100	>100	>100	>100	>100

Rotoxkit F	24-h	LC50	>100	>100	>100	>100	>100	>100	>100	>100
Protoxkit F	24-h	EC50	>100	<u>11.5</u>	n.a.	>100	>100	>100	>100	>100

Table 7c
(LT_IWWTP1)

Acute and short-term chronic tests	Duration	End-point	2009 May	2009 Jul	2009 Sep	2009 Nov	2010 Jan	2010 Apr	2010 Jun	2010 Aug
V. fischeri	30-min	EC50	>100	>100	>100	>100	>100	>100	>100	>100
D. magna (1)	48-h	EC50	>100	>100	>100	>100	>100	>100	>100	>100
D. magna (2)		EC50	>100	>100	>100	>100	>100	>100	>100	>100
P.subcapitata (1)	72-h	ErC50	>100	>100	>100	>100	>100	>100	>100	>100
P.subcapitata (2)		ErC50	<u>90.9</u>	>100	>100	>100	<u>49.7</u>	>100	>100	>100
Thamnotoxkit F (1)	24-h	LC50	>100	>100	>100	>100	>100	>100	>100	>100
Thamnotoxkit F (2)		LC50	>100	>100	>100	>100	>100	>100	>100	>100
Charatox	90-min	IC50	>100	100	>100	>100	>100	>100	>100	>100
Niteltox	96-h	LC50	>100	>100	>100	>100	>100	>100	>100	>100
Rotoxkit F	24-h	LC50	>100	>100	>100	>100	>100	>100	>100	>100
Protoxkit F	24-h	EC50	<u>32.3</u>	<u>31.0</u>	n.a.	>100	>100	>100	>100	>100

Table 7d
(LT_IWWTP2)

Acute and short-term chronic tests	Duration	End-point	2009 May	2009 Jul	2009 Sep	2009 Nov	2010 Jan	2010 Apr	2010 Jun	2010 Aug
V. fischeri	30-min	EC50	>100	>100	>100	>100	>100	>100	>100	>100
D. magna (1)	48-h	EC50	>100	>100	>100	>100	>100	>100	>100	>100
D. magna (2)		EC50	>100	>100	>100	>100	>100	>100	>100	>100
P.subcapitata (1)	72-h	ErC50	>100	>100	>100	>100	>100	>100	>100	>100
P.subcapitata (2)		ErC50	>100	<u>71.4</u>	>100	>100	>100	>100	>100	>100
Thamnotoxkit F (1)	24-h	LC50	>100	>100	>100	>100	>100	>100	>100	>100
Thamnotoxkit F (2)		LC50	>100	>100	>100	>100	>100	>100	>100	>100
Charatox	90-min	IC50	>100	>100	>100	>100	>100	>100	>100	>100
Niteltox	96-h	LC50	>100	>100	>100	>100	>100	>100	>100	>100
Rotoxkit F	24-h	LC50	>100	>100	>100	>100	>100	>100	>100	>100
Protoxkit F	24-h	EC50	>100	>100	n.a.	>100	>100	>100	>100	>100

Table 7e
(LT_SW)

Acute and short-term chronic tests	Duration	End-point	2009 May	2009 Jul	2009 Sep	2009 Nov	2010 Jan	2010 Apr	2010 Jun	2010 Aug
V. fischeri	30-min	EC50				>100			>100	
D. magna (1)	48-h	EC50				>100			>100	
D. magna (2)		EC50				>100			>100	
P.subcapitata (1)	72-h	ErC50				>100			>100	
P.subcapitata (2)		ErC50				>100			>100	
Thamnotoxkit F (1)	24-h	LC50				>100			>100	
Thamnotoxkit F (2)		LC50				>100			>100	

Charatox	90-min	IC50	>100	>100
Niteltox	96-h	LC50	>100	>100
Rotoxkit F	24-h	LC50	>100	>100
Protoxkit F	24-h	EC50	>100	>100

Table 7f
(LT_LW)

Acute and short-term chronic tests	Duration	End-point	2009 May	2009 Jul	2009 Sep	2009 Nov	2010 Jan	2010 Apr	2010 Jun	2010 Aug
V. fischeri	30-min	EC50				>100			>100	
D. magna (1)	48-h	EC50				>100			>100	
D. magna (2)		EC50				>100			>100	
P.subcapitata (1)	72-h	ErC50				>100			>100	
P.subcapitata (2)		ErC50				>100			>100	
Thamnotoxkit F (1)	24-h	LC50				>100			85.6	
Thamnotoxkit F (2)		LC50				>100			>100	
Charatox	90-min	IC50				93.0			>100	
Niteltox	96-h	LC50				>100			>100	
Rotoxkit F	24-h	LC50				>100			>100	
Protoxkit F	24-h	EC50				>100			>100	

Each effluent was tested by 11 tests (including three tests that were tested twice in two separate laboratories), i.e. during 8 biotesting sessions, from May 2009 to August 2010, 87x4=348 tests were performed, in total. When 50-% effect level is considered, a positive toxicity signal was calculated in 32 out of 348 cases.

Table 8 presents the same toxicity data as on Table 7, however toxicity data (EC50's, IC50's or LC50's) are hereby transformed into toxic units (TU) with the formula of Sprague and Ramsay (1965) [14]: $TU = 100\% / E(L)IC50$ (in % of dilution). In case of low toxicity (i.e. below the 50% and higher than 20% effect levels) detected with the undiluted 100%-effluent concentration, TUs were calculated as parts of 50%. For example, 40% effect equals $40/50=0.8$ TU. When the signals of low toxicity are considered, a detection of positive toxicity signals increased up to 67 out of 348 cases.

Table 8a-f. Municipal (LT_MWWTP1 and LT_MWWTP2), industrial (LT_IWWTP1 and LT_IWWTP2) effluent, storm water (LT_SW) and landfill water (LT_LW) toxicity data obtained with acute and short-term chronic tests (endpoint values are in toxic units, TU).

Table 8a
(LT_MWWTP1)

Acute and short-term chronic tests	Duration	End-point	2009 May	2009 Jul	2009 Sep	2009 Nov	2010 Jan	2010 Apr	2010 Jun	2010 Aug
V. fischeri	30-min	in TU	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
D. magna (1)*	48-h	in TU	1.0	1.5	0.0	0.0	0.0	0.0	0.0	0.0
D. magna (2)		in TU	0.0	0.0	0.0	<u>0.5</u>	0.0	0.0	0.0	0.0
P.subcapitata (1)	72-h	in TU	2.3	1.1	0.0	0.0	<u>0.4</u>	<u>0.7</u>	0.0	0.0
P.subcapitata (2)		in TU	2.5	1.4	0.0	0.0	1.3	2.0	0.0	0.0
Thamnotoxkit F (1)	24-h	in TU	2.9	2.7	0.0	0.0	1.0	1.6	0.0	0.0
Thamnotoxkit F (2)		in TU	1.7	1.8	0.0	0.0	0.0	1.8	0.0	0.0
Charatox	90-min	in TU	8.1	5.8	0.0	<u>0.5</u>	2.2	3.2	<u>0.7</u>	<u>0.8</u>
Niteltox	96-h	in TU	2.1	2.1	0.0	<u>0.5</u>	2.1	4.2	0.0	0.0
Rotoxkit F	24-h	in TU	<u>0.5</u>	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Protoxkit F	24-h	in TU	0.0	0.0	n.a.	0.0	0.0	0.0	0.0	0.0

* Tests were conducted in two independent laboratories: (1) and (2);

Background in column 1 marks short-term chronic tests.

Underlined are the values found at higher than 20% and below 50% effect levels (undiluted effluent)

Table 8b
(LT_MWWTP2)

Acute and short-term chronic tests	Duration	End-point	2009 May	2009 Jul	2009 Sep	2009 Nov	2010 Jan	2010 Apr	2010 Jun	2010 Aug
V. fischeri	30-min	in TU	0.0	<u>0.4</u>	0.0	0.0	0.0	0.0	0.0	0.0
D. magna (1)	48-h	in TU	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
D. magna (2)		in TU	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
P.subcapitata (1)	72-h	in TU	0.0	<u>0.8</u>	0.0	0.0	0.0	0.0	0.0	<u>0.5</u>
P.subcapitata (2)		in TU	0.0	<u>0.5</u>	0.0	0.0	0.0	0.0	0.0	0.0
Thamnotoxkit F (1)	24-h	in TU	0.0	1.4	0.0	0.0	0.0	0.0	0.0	0.0
Thamnotoxkit F (2)		in TU	0.0	1.7	0.0	0.0	0.0	<u>0.4</u>	0.0	0.0
Charatox	90-min	in TU	0.0	4.9	0.0	0.0	<u>0.6</u>	0.5	<u>0.4</u>	0.0
Niteltox	96-h	in TU	0.0	2.8	0.0	0.0	0.0	0.0	0.0	0.0
Rotoxkit F	24-h	in TU	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Protoxkit F	24-h	in TU	0.0	8.7	n.a.	0.0	0.0	0.0	0.0	0.0

Table 8c
(LT_IWWTP1)

Acute and short-term chronic tests	Duration	End-point	2009 May	2009 Jul	2009 Sep	2009 Nov	2010 Jan	2010 Apr	2010 Jun	2010 Aug
V. fischeri	30-min	<i>in TU</i>	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
D. magna (1)	48-h	<i>in TU</i>	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
D. magna (2)		<i>in TU</i>	0.0	0.0	0.0	0.0	0.0	0.0	<u>0.5</u>	0.0
P.subcapitata (1)	72-h	<i>in TU</i>	0.0	<u>0.5</u>	<u>0.5</u>	0.0	0.0	<u>0.4</u>	0.0	0.0
P.subcapitata (2)		<i>in TU</i>	1.1	0.0	0.0	0.0	2.0	0.0	<u>0.8</u>	0.0
Thamnotoxkit F (1)	24-h	<i>in TU</i>	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Thamnotoxkit F (2)		<i>in TU</i>	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Charatox	90-min	<i>in TU</i>	<u>0.7</u>	1.0	0.0	<u>0.8</u>	<u>0.4</u>	<u>0.5</u>	<u>0.7</u>	0.0
Niteltox	96-h	<i>in TU</i>	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Rotokit F	24-h	<i>in TU</i>	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Protoxkit F	24-h	<i>in TU</i>	3.1	3.2	n.a.	0.0	0.0	0.0	0.0	0.0

Table 8d
(LT_IWWTP2)

Acute and short-term chronic tests	Duration	End-point	2009 May	2009 Jul	2009 Sep	2009 Nov	2010 Jan	2010 Apr	2010 Jun	2010 Aug
V. fischeri	30-min	<i>in TU</i>	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
D. magna (1)	48-h	<i>in TU</i>	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
D. magna (2)		<i>in TU</i>	0.0	0.0	0.0	<u>0.6</u>	0.0	<u>0.6</u>	0.0	0.0
P.subcapitata (1)	72-h	<i>in TU</i>	<u>0.6</u>	<u>0.6</u>	0.0	0.0	0.0	<u>0.6</u>	0.0	0.0
P.subcapitata (2)		<i>in TU</i>	0.0	1.4	0.0	0.0	0.0	0.0	<u>0.7</u>	0.0
Thamnotoxkit F (1)	24-h	<i>in TU</i>	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Thamnotoxkit F (2)		<i>in TU</i>	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Charatox	90-min	<i>in TU</i>	<u>0.6</u>	<u>0.6</u>	0.0	0.0	0.0	<u>0.6</u>	0.0	0.0
Niteltox	96-h	<i>in TU</i>	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Rotokit F	24-h	<i>in TU</i>	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Protoxkit F	24-h	<i>in TU</i>	0.0	0.0	n.a.	0.0	0.0	0.0	0.0	0.0

Table 8e
(LT_SW)

Acute and short-term chronic tests	Duration	End-point	2009 May	2009 Jul	2009 Sep	2009 Nov	2010 Jan	2010 Apr	2010 Jun	2010 Aug
V. fischeri	30-min	<i>in TU</i>				0.0			0.0	
D. magna (1)	48-h	<i>in TU</i>				0.0			0.0	
D. magna (2)		<i>in TU</i>				0.0			0.0	
P.subcapitata (1)	72-h	<i>in TU</i>				0.0			0.0	
P.subcapitata (2)		<i>in TU</i>				0.0			0.0	
Thamnotoxkit F (1)	24-h	<i>in TU</i>				0.0			0.0	
Thamnotoxkit F (2)		<i>in TU</i>				0.0			0.0	
Charatox	90-min	<i>in TU</i>				0.0			<u>0.7</u>	
Niteltox	96-h	<i>in TU</i>				0.0			0.0	

Rotoxkit F	24-h	<i>in</i> TU	0.0	0.0
Protoxkit F	24-h	<i>in</i> TU	0.0	0.0

Table 8f
(LT_LW)

Acute and short-term chronic tests	Duration	End-point	2009 May	2009 Jul	2009 Sep	2009 Nov	2010 Jan	2010 Apr	2010 Jun	2010 Aug
V. fischeri	30-min	<i>in</i> TU				0.0			0.0	
D. magna (1)	48-h	<i>in</i> TU				<u>0.8</u>			0.0	
D. magna (2)		<i>in</i> TU				<u>0.5</u>			0.0	
P.subcapitata (1)	72-h	<i>in</i> TU				0.0			0.0	
P.subcapitata (2)		<i>in</i> TU				0.0			0.0	
Thamnotoxkit F (1)	24-h	<i>in</i> TU				0.0			1.2	
Thamnotoxkit F (2)		<i>in</i> TU				0.0			0.0	
Charatox	90-min	<i>in</i> TU				1.1			<u>0.8</u>	
Niteltox	96-h	<i>in</i> TU				0.0			0.0	
Rotoxkit F	24-h	<i>in</i> TU				0.0			0.0	
Protoxkit F	24-h	<i>in</i> TU				0.0			0.0	

Further analysis on toxicological data was conducted by attributing toxicity classes according to arbitrary toxicity classification system for effluents discharged into the aquatic environment proposed by Persoone et al. [15]. The AvTox index calculated from 3 obligatory test-battery data, B3 and B3', showed 7 and 19 toxic signals, respectively, out of 36 cases each (Tables 9a-c). Not surprisingly, the AvTox index calculated from extended 8-test-battery data, B8 and B8', was able to detect toxicity 19 and 27 times respectively, out of 36 cases each (Tables 9a-c). The shift from 3 to 8-test-battery, i.e. from B3 to B8 and from B3' to B8' (Table 9a-c) led to ascribe an effluent higher toxicity rank in 6 and 11 cases, respectively.

The evaluation of effluents by Potential Ecotoxic Effect Probe index (PEEP) developed by Costan et al. [16,17] when most powerful and relatively sensitive 8-test-battery data and effluent hourly rate (B8', Table 9) are considered revealed that harmful for aquatic biota could potentially be effluents from MWWTP1 (dates May-2009, July-2009, January-2010 and April-2010) and MWWTP2 (July-2010). The toxicity found in the latter effluent might be related with the damage of the biological treatment facilities. According to arbitrary effluent classification system proposed by Ronco et al. [18], MWWTP1 (May-2009) and MWWTP2 (July-2010) were classified as *moderately toxic* (Table 9a).

Table 9a. Integrated toxicity indices (Average Toxicity [AvTx], Toxic Print [TxPr] and PEEP), calculated for obligatory 3-test (TU3 and TU3') and extended 8-test (TU8 and TU8') battery data on municipal (LT_MWWTP1 and LT_MWWTP2) effluents investigated from May-2009 to August-2010 (8 sampling campaigns), toxicity classes appointed for 3-test (B3 and B3') and 8-test (B8 and B8') battery results, and effluent flow (Q, m³/h). Ecotoxicity results are expressed in TUs (apostrophe represents indices that include minor toxicity, i.e. more than 0.4 and less than 1.0 TU).

Table 9a

		AVERAGE TOXICITY ¹								TOXIC PRINT ²								Q, m3/h	PEEP ³							
		TU3	TU8	B3	B8	TU3'	TU8'	B3'	B8'	TU3	TU8	B3	B8	TU3'	TU8'	B3'	B8'		TU3	TU8	B3	B8	TU3'	TU8'	B3'	B8'
LT_MWWTP1	V-09	1.0	1.9	t ⁴	t	1.0	2.0	t	t	2.0	9.6	t	t	2.0	12.0	t	vt	94	2.27	2.95	ST	ST	2.27	3.05	ST ⁵	MT
	VII-09	0.7	1.5	st	t	0.7	1.5	st	t	1.4	7.6	t	t	1.4	7.5	t	t	88	2.09	2.83	ST	ST	2.09	2.82	ST	ST
	IX-09	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	127	0.00	0.00	NT	NT	0.00	0.00	NT	NT
	XI-09	0.0	0.0	nt	nt	0.1	0.2	st	st	0.0	0.0	nt	nt	0.1	0.6	st	st	95	0.00	0.00	NT	NT	1.02	1.76	NT	NT
	I-10	0.2	0.7	st	st	0.3	0.7	st	st	0.2	2.7	st	t	0.3	2.8	st	t	115	1.38	2.49	NT	ST	1.55	2.51	NT	ST
	IV-10	0.3	1.3	st	t	0.5	1.3	st	t	0.3	5.1	st	t	0.5	5.2	st	t	136	1.62	2.84	NT	ST	1.84	2.85	NT	ST
	VI-10	0.0	0.0	nt	nt	0.0	0.1	nt	st	0.0	0.0	nt	nt	0.0	0.1	nt	st	97	0.00	0.00	NT	NT	0.00	1.03	NT	NT
	VIII-10	0.0	0.0	nt	nt	0.0	0.1	nt	st	0.0	0.0	nt	nt	0.0	0.1	nt	st	109	0.00	0.00	NT	NT	0.00	1.07	NT	NT
LT_MWWTP2	V-09	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	271	0.00	0.00	NT	NT	0.00	0.00	NT	NT
	VII-09	0.0	2.2	nt	t	0.4	2.4	st	t	0.0	9.0	nt	t	0.7	14.3	st	vt	168	0.00	3.18	NT	MT	2.07	3.38	ST	MT
	IX-09	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	125	0.00	0.00	NT	NT	0.00	0.00	NT	NT
	XI-09	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	167	0.00	0.00	NT	NT	0.00	0.00	NT	NT
	I-10	0.0	0.0	nt	nt	0.0	0.1	nt	st	0.0	0.0	nt	nt	0.0	0.1	nt	st	158	0.00	0.00	NT	NT	0.00	1.23	NT	NT
	IV-10	0.0	0.0	nt	nt	0.0	0.1	nt	st	0.0	0.0	nt	nt	0.0	0.2	nt	st	258	0.00	0.00	NT	NT	0.00	1.72	NT	NT
	VI-10	0.0	0.0	nt	nt	0.0	0.1	nt	st	0.0	0.0	nt	nt	0.0	0.1	nt	st	183	0.00	0.00	NT	NT	0.00	1.29	NT	NT
	VIII-10	0.0	0.0	nt	nt	0.1	0.0	st	st	0.0	0.0	nt	nt	0.1	0.0	st	nt	206	0.00	0.00	NT	NT	1.33	0.00	NT	NT

¹ Average Toxicity (AvTx) index (sum of the endpoint values in TUs divided by the number of tests);

² Toxic Print (TxPr) index represents the AvTx multiplied by the number of tests exhibiting positive (toxic) responses [16];

³ Potential Ecotoxic Effect Probe, PEEP = log₁₀ (1+TxPr·Q) [16,17];

⁴ Arbitrary toxicity classification system for effluents discharged into the aquatic environment [15]: “not toxic” (nt, <0.4 TU), “slightly toxic” (0.4 ≤ st <1), “toxic” (1.0, < t <10), “very toxic” (10.0 ≤ vt <100) and “extremely toxic” (et ≥ 100);

⁵ PEEP-based toxicity classification system for effluents discharged into the aquatic environment (including effluent flow rate, Q) [18]: “practically non-toxic” (NT, ≤1.99 TU), “slightly toxic” (2 < ST ≤2.99), “moderately toxic” (3 < MT <3.99), “highly toxic” (4 ≤ HT <4.99) and “very highly toxic” (VHT > 5).

Table 9b. Integrated toxicity indices (Average Toxicity, Toxic Print and PEEP), calculated for obligatory 3-test (TU3 and TU3') and extended 8-test (TU8 and TU8') battery data on municipal (LT_IWWTP1 and LT_IWWTP2) effluents investigated from May-2009 to August-2010 (8 sampling campaigns), toxicity classes appointed for 3-test (B3 and B3') and 8-test (B8 and B8') battery results, and effluent flow (Q, m³/h). Ecotoxicity results are expressed in TUs (apostrophe represents indices that include minor toxicity, i.e. more than 0.4 and less than 1.0 TU).

Table 9b

		AVERAGE TOXICITY ¹								TOXIC PRINT ²								Q, m ³ /h	PEEP ³							
		TU3	TU8	B3	B8	TU3'	TU8'	B3'	B8'	TU3	TU8	B3	B8	TU3'	TU8'	B3'	B8'		TU3	TU8	B3	B8	TU3'	TU8'	B3'	B8'
LT_IWWTP1	V-09	0.2	0.5	st ⁴	st	0.2	0.5	st	st	0.2	0.9	st	st	0.2	1.6	st	t	20	0.70	1.28	NT	NT	0.70	1.52	NT ⁵	NT
	VII-09	0.0	0.5	nt	st	0.1	0.6	st	st	0.0	1.1	nt	t	0.1	1.7	st	t	17	0.00	1.29	NT	NT	0.43	1.47	NT	NT
	IX-09	0.0	0.0	nt	nt	0.1	0.0	st	st	0.0	0.0	nt	nt	0.1	0.0	st	nt	15	0.00	0.00	NT	NT	0.40	0.00	NT	NT
	XI-09	0.0	0.0	nt	nt	0.0	0.1	nt	st	0.0	0.0	nt	nt	0.0	0.1	nt	st	15	0.00	0.00	NT	NT	0.00	0.40	NT	NT
	I-10	0.3	0.1	st	st	0.3	0.2	st	st	0.3	0.1	st	st	0.3	0.4	st	st	13	0.70	0.37	NT	NT	0.70	0.80	NT	NT
	IV-10	0.0	0.0	nt	nt	0.1	0.1	st	st	0.0	0.0	nt	nt	0.1	0.2	st	st	17	0.00	0.00	NT	NT	0.43	0.65	NT	NT
	VI-10	0.0	0.0	nt	nt	0.2	0.2	st	st	0.0	0.0	nt	nt	0.4	0.5	st	st	16	0.00	0.00	NT	NT	0.86	0.95	NT	NT
	VIII-10	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	21	0.00	0.00	NT	NT	0.00	0.00	NT	NT
LT_IWWTP2	V-09	0.0	0.0	nt	nt	0.1	0.1	st	st	0.0	0.0	nt	nt	0.1	0.2	st	st	40	0.00	0.00	NT	NT	0.70	0.95	NT	NT
	VII-09	0.2	0.1	st	st	0.3	0.2	st	st	0.2	0.1	st	st	0.3	0.4	st	st	25	0.78	0.55	NT	NT	0.93	1.05	NT	NT
	IX-09	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	51	0.00	0.00	NT	NT	0.00	0.00	NT	NT
	XI-09	0.0	0.0	nt	nt	0.1	0.0	st	st	0.0	0.0	nt	nt	0.1	0.0	st	nt	49	0.00	0.00	NT	NT	0.77	0.00	NT	NT
	I-10	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	27	0.00	0.00	NT	NT	0.00	0.00	NT	NT
	IV-10	0.0	0.0	nt	nt	0.2	0.2	st	st	0.0	0.0	nt	nt	0.4	0.5	st	st	35	0.00	0.00	NT	NT	1.18	1.27	NT	NT
	VI-10	0.0	0.0	nt	nt	0.1	0.0	st	st	0.0	0.0	nt	nt	0.1	0.0	st	nt	21	0.00	0.00	NT	NT	0.50	0.00	NT	NT
	VIII-10	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	30	0.00	0.00	NT	NT	0.00	0.00	NT	NT

¹ Average Toxicity (AvTx) index (sum of the endpoint values in TUs divided by the number of tests);

² Toxic Print (TxPr) index represents the AvTx multiplied by the number of tests exhibiting positive (toxic) responses [16];

³ Potential Ecotoxic Effect Probe, PEEP = log₁₀ (1+TxPr·Q) [16,17];

⁴ Arbitrary toxicity classification system for effluents discharged into the aquatic environment [15]: “not toxic” (nt, <0.4 TU), “slightly toxic” (0.4 ≤ st <1), “toxic” (1.0, < t <10), “very toxic” (10.0 ≤ vt <100) and “extremely toxic” (et ≥ 100);

⁵ PEEP-based toxicity classification system for effluents discharged into the aquatic environment (including effluent flow rate, Q) [18]: “practically non-toxic” (NT, ≤1.99 TU), “slightly toxic” (2 < ST ≤2.99), “moderately toxic” (3 < MT <3.99), “highly toxic” (4 ≤ HT <4.99) and “very highly toxic” (VHT > 5).

Table 9c. Integrated toxicity indices (Average Toxicity and Toxic Print), calculated for obligatory 3-test (TU3 and TU3') and extended 8-test (TU8 and TU8') battery data on storm water (LT_SW) and landfill leachate (LT_LW) investigated in November-2009 and June-2010 (2 sampling campaigns), toxicity classes appointed for 3-test (B3 and B3') and 8-test (B8 and B8') battery results.

		AVERAGE TOXICITY ¹								TOXIC PRINT ²							
		TU3	TU8	B3	B8	TU3'	TU8'	B3'	B8'	TU3	TU8	B3	B8	TU3'	TU8'	B3'	B8'
LT_SW	V-09																
	VII-09																
	IX-09																
	XI-09	0.0	0.0	nt ³	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt	0.0	0.0	nt	nt
	I-10																
	IV-10																
	VI-10	0.0	0.0	nt	nt	0.0	0.1	<u>nt</u>	<u>st</u>	0.0	0.0	nt	nt	0.0	0.1	<u>nt</u>	<u>st</u>
	VIII-10																
LT_LW	V-09																
	VII-09																
	IX-09																
	XI-09	0.0	0.1	<u>nt</u>	<u>st</u>	0.2	0.2	st	st	0.0	0.1	<u>nt</u>	<u>st</u>	0.2	0.4	st	st
	I-10																
	IV-10																
	VI-10	0.0	0.1	<u>nt</u>	<u>st</u>	0.0	0.2	<u>nt</u>	<u>st</u>	0.0	0.1	<u>nt</u>	<u>st</u>	0.0	0.4	<u>nt</u>	<u>st</u>
	VIII-10																

Ecotoxicity results are expressed in TUs (apostrophe represents indices that include minor toxicity, i.e. more than 0.4 and less than 1.0 TU);

¹ Average Toxicity (AvTx) index (sum of the endpoint values in TUs divided by the number of tests);

² Toxic Print (TxPr) index represents the AvTx multiplied by the number of tests exhibiting positive (toxic) responses [16];

³ Arbitrary toxicity classification system for effluents discharged into the aquatic environment [15]: "not toxic" (*nt*, <0.4 TU), "slightly toxic" (0.4 ≤ *st* <1), "toxic" (1.0, < *t* <10), "very toxic" (10.0 ≤ *vt* <100) and "extremely toxic" (*et* ≥ 100).

Table 10. Linear regression (r^2) between toxicity index (Average Toxicity) and respective chemical parameter for municipal (LT_MWWTP1 and LT_MWWTP2), industrial (LT_IWWTP1 and LT_IWWTP2) effluents, storm water (LT_SW) and landfill water (LT_LW).

	BOD ₇ , mg/l	COD _{Cr} , mg/l	SS mg/l	Tot-P mgP/l	PO ₄ -P mgP/l	Tot-N mgN/l	NH ₄ -N mgN/l	Alkalinity, mmol/l	SO ₄ ²⁻ mg/l	TOC mgC/l
TU3	0.01	0.01	0.00	0.00	0.03	0.29	0.66	0.07	0.02	0.01
TU8	0.04	0.00	0.14	0.23	0.28	0.67	0.90	0.31	0.05	0.00
TU3'	0.00	0.00	0.04	0.03	0.00	0.46	0.81	0.05	0.01	0.00
TU8'	0.06	0.01	0.04	0.26	0.30	0.69	0.88	0.31	0.06	0.00

Average Toxicity was calculated for obligatory 3-test (TU3 and TU3') and extended 8-test (TU8 and TU8') batteries; apostrophe represents indices that include minor toxicity, i.e. more than 0.4 and less than 1.0 TU.

Linear regression between toxicity results obtained from various number of tests in the battery (TU3 and TU8) or different levels of percentage endpoint levels in the test (TU3 or TU3' and TU8 or TU8'), and general wastewater chemical parameters revealed that main possible reason of observed toxicity could be ammonium (the highest value of r^2 on Table 10). The MAC value stated for effluents discharged in Lithuanian aquatic environment for this chemical (5 mgN/l) was exceeded in 6 tested effluents.

6.2 Chronic tests and biomarkers

In addition to acute and short-term chronic tests, effluent samples from LT_MWWTP1 and LT_IWWTP1 were analysed by chronic tests and biomarkers in Finnish Environmental Institute (SYKE). The tests were conducted on the samples taken on January 12, 2010. The first long-term test, 7-day growth inhibition of aquatic vascular plant *Lemna minor* test (ISO 20079), showed no decrease in growth, instead, approximately 10-25 % increase in frond number and leaf area was found for both effluents. The second long-term test, 14-day *D. magna* reproduction test, revealed just insignificant decrease in survival of offsprings (up to 5%) treated by both effluents. It can be mentioned that LT_MWWTP1 and LT_IWWTP1 effluents collected on January 14, 2010 at the same WWTP, were evaluated as slightly toxic (based on AvTx values, Tables 9a and b) with the 8-test-battery. Toxic responses have been detected by 4 tests for LT_MWWTP1 (Table 8a) and by 2 tests for LT_IWWTP1 (Table 8c).

The presence of endocrine-disrupting contaminants in effluents were explored by fish hepatocyte vitellogenin induction assay conducted by SYKE. Both effluents, LT_MWWTP1 and LT_IWWTP1 showed an induction of vitellogenin up to 300 pg/ml.

Table 11. LT_MWWTP1 and LT_IWWTP1 induced alterations of vitellogenin (pg/ml) in fish hepatocytes.

Report			
%		MWWTP1	IWWTP 1
,0	Mean pg/ml	,00	,00
	N	4	4
	Std. Deviation	,000	,000
6,3	Mean pg/ml	71,43	236,61
	N	4	4
	Std. Deviation	43,741	101,408
12,5	Mean pg/ml	272,32	116,07
	N	4	4
	Std. Deviation	93,785	69,160
25,0	Mean pg/ml	267,86	232,14
	N	4	4
	Std. Deviation	97,807	88,688
50,0	Mean pg/ml	290,18	316,96
	N	4	4
	Std. Deviation	89,136	36,813

Another biomarker used in the study represented cytochrome P450-1A (CYP1A) family of monooxygenases which is known to perform vital enzymatic functions in animals by detoxifying lipophilic environmental contaminants. Hepatocyte EROD activity assay conducted on LT_MWWTP1 and LT_IWWTP1 effluent samples showed remarkable suppression of the enzyme activity (Table 12).

Table 12. LT_MWWTP1 and LT_IWWTP1 induced hepatocyte ethoxy-and methoxyresorufin-O-deethylase (EROD) activity alterations.

Lithuania; Hepatocyte EROD activity (pmol/mg prot./min); mean ± SD						
		%	MWWTP1	IWWTP1		
		0	2,8 ± 0,66	2,8 ± 0,66		
		6,3	0,35 ± 0,07	0,4 ± 0,28		
		12,5	0,30 ± 0,14	0,0 ± 0,0		
		25	0,1 ± 0,0	0,0 ± 0,0		
		50	0,0 ± 0,0	0,0 ± 0,0		

7 Conclusions

1. Data of chemical analysis revealed that the concentrations of 11 priority hazardous substances in selected Lithuanian effluents sites did not exceed the maximum allowable concentrations (MAC) according to Lithuanian Waste water management regulation [12].
2. Toxicological screening of two WWTPs located near the coastal zone revealed rather high variability of effluents quality discharged into environment in terms of nitrogen containing compounds, in particular, ammonium. The contaminant exceeded Lithuanian MAC limits in several effluent samples. This was reflected by good correlation between ammonium concentration and integral toxicity index calculated from the test-battery data.
3. The toxicity of majority of the effluent samples assessed by various tests did not reach 50% endpoint level. To increase relative sensitivity of the test-battery-based evaluation it was useful to include lower endpoint levels. Then measured positive toxic signals increased from approximately 10 to 20%.
4. Biomarkers (vitellogenin induction, EROD activity assays) applied on two effluents (LT_MWWTP1 and LT_IWWTP1, sampling date 12-01-2010) revealed their potential toxicity, while chronic tests with *L. minor* growth inhibition and long-term *D. magna* reproduction did not show toxicity of these effluents.

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Appendices

Appendix A: COHIBA project partners

Finnish Environment Institute (SYKE)
Baltic Marine Environment Protection Commission (HELCOM)
Baltic Environmental Forum Latvia (BEF - LV)
Latvian Institute of Aquatic Ecology (LHEI)
Baltic Environmental Forum Estonia (BEF - EE)
Estonian Marine Institute, University of Tartu
Estonian Environmental Research Centre
Tallinn University of Technology
Municipality of Copenhagen
Copenhagen Waste Water Treatment Plants
Copenhagen Energy (KE)
Technical University of Denmark (DTU)
Baltic Environmental Forum Lithuania (BEF - LT)
Environmental Protection Agency, Ministry of Environment of Lithuania (EPA)
Institute of Botany, Nature Research Center (IB)
Institute for Ecology of Industrial Areas (IETU)
Swedish Environmental Research Institute (IVL)
Swedish Chemicals Agency (KEMI)
City of Stockholm, Environment and Health Administration
Federal Environment Agency (UBA)
State of Mecklenburg-Vorpommern (MV) (Ministry of Agriculture, Environment and Consumer Protection)

Appendix B: Municipal waste water treatment plant 1 (MWWTP1)

Table B-1. Basic data from WWTP.

Parameter	MWWTP1							
Sampling site								
Sampling date	May-2009	Jul-2009	Sep-2009	Nov-2009	Jan-2010	Apr-2010	Jun-2010	Aug-2010
Sampling time	10:45	10:00	13:00	10:15	11:15	9:40	10:15	10:35
Flow rate (m ³ /d)	2245	2108	3048	2269	2761	3268	2318	2611
BOD ₇ (mg/l)	14	6.6	6.1	4.7	5.4	6.1	3.4	5.9
COD _{Cr} (mg/l)	75	53	26	44	40	29	57	60
Suspended solids (mg/l)	16	9.6	6.4	8.0	5.6	4.4	2.0	8.4
Tot-P (mgP/l)	0.96	0.96	0.267	0.18	0.4	0.25	0.18	0.52
PO ₄ -P (mgP/l)		0.657	0.039	0.008	0.35	0.015	0.011	0.230
Tot-N (mgN/l)	46.3	11.9	3.93	7.92	17.6	25.3	13.0	5.15
NH ₄ -N (mgN/l)	45.5	33.4	0.238	0.036	15.4	24.5	0.046	1.20
Alkalinity (mmol/l)		9.72	5.11	7.31	7.98	9.57	7.88	9.08
pH	7.85	7.5	7.3	7.37	7.42	7.59	7.56	7.70
Conductivity (mS/m)	119.5	148	84.4	93.3	130.3	115.8	139	134
Fe (mgFe/l)	0.16	0.12	0.057	0.088	0.12	0.089	0.099	0.069
t (°C)	14	16	17	9	4	9	14	22
SO ₄ ²⁻ (mg/l)		80	67.6	73.2	67.3	90	89	64
TOC (mgC/l)	12.1	11.1	8.38	11.3	9.83	10.8	9.63	8.42

Table B-2. Results of chemical analyses.

Waste water								
Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
PBDEs								
BDE-17	147217-75-2	ng/l	-	nd	nd	nd	nd	nd
BDE-28	41318-75-6	ng/l	-	nd	nd	nd	nd	nd
BDE-47	5436-43-1	ng/l	-	0.05	nd	nd	0.07	0.,22
BDE-66	189084-61-5	ng/l	-	nd	nd	nd	nd	nd
BDE-85	182346-21-0	ng/l	-	nd	nd	nd	nd	nd
BDE-99	60348-60-9	ng/l	-	0.10	nd	nd	0.14	nd
BDE-100	189084-64-8	ng/l	-	nd	nd	nd	nd	nd
BDE-153	68631-49-2	ng/l	-	nd	nd	nd	nd	nd
BDE-154	207122-15-4	ng/l	-	nd	nd	nd	nd	nd
BDE-183	207122-16-5	ng/l	-	nd	nd	nd	nd	nd
BDE-203		ng/l	-	nd	nd	nd	nd	nd
BDE-209	1163-19-5	ng/l	-	0.19	nd	2.05	0.15	1.59
pentaBDE		ng/l	-	0.15	nd	nd	0.21	0.22
octaBDE		ng/l	-	nd	nd	nd	nd	nd
decaBDE		ng/l	-	0.19	nd	2.05	0.15	1.59
HBCDs								
α -HBCD		ng/l	0.39	0.73	0.36	0.68	nd	0.35
β -HBCD		ng/l	0.05	0.03	0.34	0.37	nd	nd
γ -HBCD		ng/l	0.36	0.23	1.16	1.31	1.,70	0.36
Perfluorinated substances								
perfluoro-n-hexanoic acid (PFHxA)	307-24-4	ng/l	0.4	0.2	0.1	0.4	0.3	0.7
perfluorooctane sulfonate (PFOS)	1763-23-1	ng/l	0.4	0.8	1.1	0.6	0.,8	1.1
perfluorooctanoic acid (PFOA)	335-67-1	ng/l	4.2	2.6	2.4	2.1	4.6	3.8
perfluoro-n-decanoic acid (PFDA)	335-76-2	ng/l	0.5	0.4	0.3	nd	1.1	1.3
Phenolic substances								
bisphenol a	80-05-7	μ g/l	0.39	0.12	0.38	0.13	0.12	nd
4-nonylphenol (mix.)	84852-15-3	μ g/l	0.18	0.19	0.75	0.59	0.24	0.16
4-nonylphenol monoethoxylate (mix.)	-	μ g/l	0.09	0.06	0.08	0.11	nd	nd
4-nonylphenol diethoxylate (mix.)	-	μ g/l	nd	0.03	0.02	0.09	nd	nd
octylphenol	140-66-9	μ g/l	nd	nd	0.06	nd	nd	nd
octylphenol monoethoxylate	-	μ g/l	nd	nd	nd	nd	nd	nd

Waste water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
octylphenol diethoxylate	-	µg/l	nd	nd	nd	nd	nd	nd
Dioxins								
2,3,7,8-TCDD	1746-01-6	pg/l				0,1396	<0,23	<0,24
2,3,7,8-TCDF	51207-31-9	pg/l				<0,053	<0,14	<0,024
1,2,3,7,8-PeCDD	40321-76-4	pg/l				<0,17	<0,27	<0,047
1,2,3,7,8-PeCDF	57117-41-6	pg/l				<0,060	<0,12	<0,031
2,3,4,7,8-PeCDF	57117-31-4	pg/l				<0,072	<0,18	<0,065
1,2,3,4,7,8-HxCDD	39227-28-6	pg/l				<0,26	<0,44	<0,088
1,2,3,6,7,8-HxCDD	57653-85-7	pg/l				<0,26	<0,43	<0,087
1,2,3,7,8,9-HxCDD	19408-74-3	pg/l				<0,25	<0,42	<0,085
1,2,3,4,7,8-HxCDF	70648-26-9	pg/l				<0,11	<0,18	<0,078
1,2,3,6,7,8-HxCDF	57117-44-9	pg/l				<0,085	<0,17	<0,055
1,2,3,7,8,9-HxCDF	72918-21-9	pg/l				<0,48	<0,65	<0,083
2,3,4,6,7,8-HxCDF	60851-34-5	pg/l				<0,16	<0,26	<0,095
1,2,3,4,6,7,8-HpCDD	35822-46-9	pg/l				<0,35	<0,56	<0,089
1,2,3,4,6,7,8-HpCDF	67562-39-4	pg/l				<0,11	<0,21	<0,084
1,2,3,4,7,8,9-HpCDF	55673-89-7	pg/l				<0,57	<0,98	<0,076
OCDD	3268-87-9	pg/l				1,242	<1,4	0,43
OCDF	39001-02-0	pg/l				<1,8	<3,6	<0,19
Sum		pg/l				<6,2	<10	<1,9
WHO-TEQ 2005 (upperbound)		pg/l				0,5119	0,8467	0,3736
WHO-TEQ 2005 (mediumbound)		pg/l				0,3259	0,4233	0,1869
WHO-TEQ 2005 (lowerbound)		pg/l				0,14	0,0000	0,0001
CO-PCB-77		pg/l				8,7609	2,7730	2,10
CO-PCB-81		pg/l				0,5752	<0,17	0,10
CO-PCB-126		pg/l				0,0628	<0,11	<0,076
CO-PCB-169		pg/l				<0,078	<0,12	<0,055
Sum CO-PCB		pg/l				< 9,5	< 3,2	<2,3
WHO-TEQ 2005 (upperbound)		pg/l				0,0097	0,0147	0,0095
PCB-18		ng/l				0,3141	0,1274	0,02
PCB-28/31		ng/l				0,6184	0,2482	0,07
PCB-33		ng/l				0,1596	0,0265	<0,0070
PCB-47		ng/l				1,5255	<0,043	<0,067
PCB-49		ng/l				0,2713	0,0301	0,01
PCB-51		ng/l				0,2435	<0,0060	<0,0063

Waste water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
PCB-52		ng/l				0,6612	0,0488	0,02
PCB-60		ng/l				0,0536	0,0120	0,00
PCB-66		ng/l				0,2422	0,0447	0,02
PCB-74		ng/l				0,1484	0,0258	0,01
PCB-99		ng/l				0,0562	0,0142	0,01
PCB-101		ng/l				0,1476	0,0304	<0,018
PCB-105		ng/l				0,0170	0,0117	<0,0069
PCB-110		ng/l				0,0663	0,0303	<0,019
PCB-114		ng/l				<0,0037	<0,0015	<0,0004
PCB-118		ng/l				0,0674	0,0317	<0,018
PCB-122		ng/l				<0,0045	<0,0017	<0,0004
PCB-123		ng/l				<0,0032	<0,0013	<0,0003
PCB-128		ng/l				0,0149	0,0145	<0,0036
PCB-138		ng/l				0,0724	0,0884	<0,017
PCB-141		ng/l				0,0217	0,0230	<0,0034
PCB-153		ng/l				0,0657	0,0814	<0,015
PCB-156		ng/l				0,0144	0,0176	<0,0018
PCB-157		ng/l				<0,0039	<0,0018	0,00
PCB-167		ng/l				<0,0033	0,0045	<0,0005
PCB-170		ng/l				0,0418	0,0737	<0,0032
PCB-180		ng/l				0,0618	0,0980	<0,0061
PCB-183		ng/l				0,0142	0,0181	<0,0019
PCB-187		ng/l				0,0211	0,0253	<0,0034
PCB-189		ng/l				<0,0047	0,0035	<0,0003
PCB-194		ng/l				0,0104	0,0180	<0,0010
PCB-206		ng/l				<0,010	<0,0027	<0,0005
PCB-209		ng/l				<0,0062	<0,0021	<0,0005
Sum PCBs		ng/l				<5,0	< 1,2	<0,36
WHO-TEQ 2005 pg/l (upperbound)						0,0135	0,0173	0,0104
WHO-TEQ 2005 pg/l (mediumbound)						0,0121	0,0100	0,0053
WHO-TEQ 2005 pg/l (lowerbound)						0,0107	0,0027	0,0003
Organotins								
Monobutyltin cation, MBT		ng/l	<1	<1	<1	18	7,3	6
Dibutyltin cation, DBT		ng/l	<1	<1	<1	1,3	1,2	0,97
Tributyltin cation, TBT		ng/l	<1	<1	<1	<1	<1	<1

Waste water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
Tetrabutyltin, TTBT		ng/l	<1	<1	<1	<1	<1	<1
Monooctyltin cation, MOT		ng/l	<1	<1	<1	<1	<1	<1
Diocetyl tin cation, DOT		ng/l	<1	<1	<1	<1	<1	<1
Triphenyltin cation, TPhT		ng/l	<1	<1	<1	<1	<1	<1
Tricyclohexyltin cation, TCyT		ng/l	<1	<1	<1	<1	<1	<1
Chlorinated paraffins								
SCCP	85535-84-8	µg/l	0,90	1,08	1,22	1,31	1,95	0,39
MCCP		µg/l		3,53	3,69	31,50	1,22	1,68
Endosulfane								
α-Endosulfane	959-98-8	µg/l	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004
β-Endosulfane	33213-65-9	µg/l	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004
Endosulfane sulphate	1031-07-8	µg/l	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Metals								
Cadmium	7440-43-9	µg/l	<0,050	<0,050	<0,050	<0,050	<0,050	<0,050
Mercury	7439-97-6	µg/l	<0,020	<0,020	0,029	<0,020	<0,020	<0,020

The values above the detection limit but below the quantification limit are marked with **bold**.

The values below the detection limit are marked as nd (=not detected).

The values below the quantification limit (LOQ) are marked as <LOQ.

Appendix C: Municipal waste water treatment plant 2 (MWWTP2)

Table C-1. Basic data from WWTP.

Parameter	MWWTP2							
Sampling site								
Sampling date	May-2009	Jul-2009	Sep-2009	Nov-2009	Jan-2010	Apr-2010	Jun-2010	Aug-2010
Sampling time	10:40	12:30	15:25	15:25	11:45	16:15	10:30	10:50
Flow rate (m ³ /d)	6500	4025	3000	4000	3800	6200	4400	4950
BOD ₇ (mg/l)	5.7	664	4.1	5.5	3.2	<3.0	<3.0	<3.0
COD _{Cr} (mg/l)	55	730	33	33	50	26	54	48
Suspended solids (mg/l)	7	128	14	4.0	3.6	3.6	2.8	7.2
Tot-P (mgP/l)	1.01	8.4	3.26	2.39	2.12	0.5	0.42	2.51
PO ₄ -P (mgP/l)		5.7	2.83	2.08	2.01	0.37	0.30	1.78
Tot-N (mgN/l)	2.87	45	10.2	19.9	11.5	5.9	14.6	4.56
NH ₄ -N (mgN/l)	0.41	32	0.59	0.29	0.029	0.02	0.038	0.38
Alkalinity (mmol/l)		11.42	5.07	6.45	6.84	7.12	8.35	7.56
pH	7.37	6.75	6.93	7.03	6.80	7.06	7.21	7.32
Conductivity (mS/m)	148.3	174	106.2	126.2	142.6	122.6	134	56.5
Fe (mgFe/l)	0.14	1.3	0.04	0.063	0.047	0.05	0.041	0.031
t (°C)	18	18	17	10	7	10	15	20
SO ₄ ²⁻ (mg/l)		114	54.3	75.4	73.8	72	35	45
TOC (mgC/l)	11.8	176	8.87	10.2	9.5	9.31	9.54	7.48

Table C-2. Results of chemical analyses.

Name of substance	CAS	Unit	Waste water						Sludge			
			Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10	Unit	Jan-10	Jun-10	
PBDEs												
BDE-17	147217-75-2	ng/l	nd	nd	nd	nd	nd	nd	nd	µg/kg	0,46	nd
BDE-28	41318-75-6	ng/l	nd	nd	nd	nd	nd	nd	nd	µg/kg	nd	nd
BDE-47	5436-43-1	ng/l	0,15	0,08	nd	0,30	0,04	nd	nd	µg/kg	9,50	6,72
BDE-66	189084-61-5	ng/l	nd	nd	nd	nd	nd	nd	nd	µg/kg	nd	0,85
BDE-85	182346-21-0	ng/l	nd	nd	nd	nd	nd	nd	nd	µg/kg	nd	nd
BDE-99	60348-60-9	ng/l	0,14	0,12	nd	nd	0,08	nd	nd	µg/kg	13,1	8,60
BDE-100	189084-64-8	ng/l	0,06	0,05	nd	nd	nd	nd	nd	µg/kg	2,88	1,17
BDE-153	68631-49-2	ng/l	0,08	nd	nd	nd	nd	nd	nd	µg/kg	3,15	2,23
BDE-154	207122-15-4	ng/l	nd	nd	nd	nd	nd	nd	nd	µg/kg	nd	nd
BDE-183	207122-16-5	ng/l	nd	nd	nd	nd	nd	nd	nd	µg/kg	1,17	0,98
BDE-203		ng/l	nd	nd	nd	nd	nd	nd	nd	µg/kg	1,21	1,11
BDE-209	1163-19-5	ng/l	0,58	0,14	4,95	1,62	0,1	1,25	nd	µg/kg	222	240
pentaBDE		ng/l	0,43	0,25	nd	0,30	0,12	nd	nd	µg/kg	28,59	18,72
octaBDE		ng/l	nd	nd	nd	nd	nd	nd	nd	µg/kg	2,38	2,09
decaBDE		ng/l	0,58	0,14	4,95	1,62	0,1	1,25	nd	µg/kg	222	240
HBCDs												
α-HBCD		ng/l	0,80	nd	0,24	0,70	0,47	nd	nd	µg/kg	32,1	89,1
β-HBCD		ng/l	0,20	nd	0,33	0,32	0,20	nd	nd	µg/kg	11,5	4,3
γ-HBCD		ng/l	0,32	nd	0,37	2,24	0,70	nd	nd	µg/kg	28,6	14,8
Perfluorinated substances												
perfluoro-n-hexanoic acid (PFHxA)	307-24-4	ng/l	0,6	0,5	0,3	0,3	0,1	0,3	nd	µg/kg	nd	nd
perfluorooctane sulfonate (PFOS)	1763-23-1	ng/l	0,2	0,5	1,0	0,6	0,4	1,3	nd	µg/kg	3,3	3,5
perfluorooctanoic acid (PFOA)	335-67-1	ng/l	3,8	3,2	4,6	2,3	2,2	3,1	nd	µg/kg	1,0	0,4
perfluoro-n-decanoic acid (PFDA)	335-76-2	ng/l	0,3	0,3	0,3	nd	0,8	0,9	nd	µg/kg	2,2	1,7
Phenolic substances												
bisphenol a	80-05-7	µg/l	0,18	nd	0,02	nd	0,10	nd	nd	mg/kg	nd	nd
4-nonylphenol (mix.)	84852-15-3	µg/l	nd	0,17	0,20	0,46	0,10	0,16	nd	mg/kg	4,28	0,95
4-nonylphenol monoethoxylate (mix.)	-	µg/l	0,05	nd	0,05	0,08	nd	nd	nd	mg/kg	0,72	0,16
4-nonylphenol diethoxylate (mix.)	-	µg/l	nd	0,03	nd	nd	nd	nd	nd	mg/kg	0,90	0,44
octylphenol	140-66-9	µg/l	nd	nd	0,13	nd	nd	nd	nd	mg/kg	0,20	0,19
octylphenol monoethoxylate	-	µg/l	nd	nd	nd	nd	nd	nd	nd	mg/kg	0,03	nd
octylphenol diethoxylate	-	µg/l	nd	nd	nd	nd	nd	nd	nd	mg/kg	0,31	nd

Dioxins											
2,3,7,8-TCDD	1746-01-6	pg/l				<0,11	<0,25	<0,24	ng/kg		0,3827
2,3,7,8-TCDF	51207-31-9	pg/l				<0,067	<0,14	<0,024	ng/kg		1,4874
1,2,3,7,8-PeCDD	40321-76-4	pg/l				<0,18	<0,25	<0,059	ng/kg		1,1993
1,2,3,7,8-PeCDF	57117-41-6	pg/l				<0,056	<0,12	<0,034	ng/kg		0,4133
2,3,4,7,8-PeCDF	57117-31-4	pg/l				<0,061	<0,19	<0,065	ng/kg		1,2454
1,2,3,4,7,8-HxCDD	39227-28-6	pg/l				<0,20	<0,41	<0,096	ng/kg		<0,30
1,2,3,6,7,8-HxCDD	57653-85-7	pg/l				<0,20	<0,40	<0,093	ng/kg		1,1101
1,2,3,7,8,9-HxCDD	19408-74-3	pg/l				<0,19	<0,40	<0,092	ng/kg		0,743
1,2,3,4,7,8-HxCDF	70648-26-9	pg/l				<0,086	<0,16	<0,078	ng/kg		1,4538
1,2,3,6,7,8-HxCDF	57117-44-9	pg/l				<0,071	<0,17	<0,055	ng/kg		1,0137
1,2,3,7,8,9-HxCDF	72918-21-9	pg/l				<0,43	<0,68	<0,083	ng/kg		<0,32
2,3,4,6,7,8-HxCDF	60851-34-5	pg/l				<0,14	<0,28	<0,14	ng/kg		1,407
1,2,3,4,6,7,8-HpCDD	35822-46-9	pg/l				<0,31	<0,69	<0,11	ng/kg		15,2825
1,2,3,4,6,7,8-HpCDF	67562-39-4	pg/l				<0,10	<0,24	<0,084	ng/kg		7,8268
1,2,3,4,7,8,9-HpCDF	55673-89-7	pg/l				<0,62	<1,1	<0,11	ng/kg		0,8487
OCDD	3268-87-9	pg/l				<0,83	<1,6	0,55	ng/kg		171,2667
OCDF	39001-02-0	pg/l				<2,3	<3,5	<0,27	ng/kg		28,3717
Sum		pg/l				<6,0	<11	<2,2	ng/kg		<295
WHO-TEQ 2005 (upperbound)		pg/l				0,4525	0,8386	0,3932	ng/kg		3,0504
WHO-TEQ 2005 (mediumbound)		pg/l				0,2262	0,4193	0,1967	ng/kg		3,0197
WHO-TEQ 2005 (lowerbound)		pg/l				0,0000	0,0000	0,0002	ng/kg		2,9889
CO-PCB-77		pg/l				3,5487	0,9481	1,00	ng/kg		179,3964
CO-PCB-81		pg/l				0,2758	<0,13	<0,055	ng/kg		11,035
CO-PCB-126		pg/l				<0,051	<0,086	<0,076	ng/kg		5,6306
CO-PCB-169		pg/l				<0,058	<0,14	<0,059	ng/kg		2,2127
Sum CO-PCB		pg/l				< 3,9	< 1,3	< 1,2	ng/kg		198,2747
WHO-TEQ 2005 (upperbound)		pg/l				0,0073	0,0130	0,0095	ng/kg		0,6507
PCB-18		ng/l				0,1746	0,0474	0,05	µg/kg		2,1012
PCB-28/31		ng/l				0,1943	0,0549	0,06	µg/kg		4,2842
PCB-33		ng/l				0,0499	0,0055	<0,0070	µg/kg		0,9726
PCB-47		ng/l				0,1415	<0,043	<0,067	µg/kg		0,4436
PCB-49		ng/l				0,0548	0,0148	0,01	µg/kg		1,2037
PCB-51		ng/l				0,0191	<0,0060	0,01	µg/kg		0,0772
PCB-52		ng/l				0,1360	0,0274	0,02	µg/kg		1,8336
PCB-60		ng/l				0,0146	0,0037	0,00	µg/kg		0,4343

PCB-66		ng/l				0,0592	0,0148	0,01	µg/kg		1,5084
PCB-74		ng/l				0,0385	0,0102	0,01	µg/kg		1,0313
PCB-99		ng/l				0,0242	0,0104	<0,0075	µg/kg		1,1172
PCB-101		ng/l				0,0700	0,0190	<0,018	µg/kg		1,7192
PCB-105		ng/l				0,0144	0,0075	<0,0069	µg/kg		0,7222
PCB-110		ng/l				0,0428	0,0184	<0,019	µg/kg		1,7533
PCB-114		ng/l				<0,0030	<0,0017	<0,0004	µg/kg		0,0938
PCB-118		ng/l				0,0423	0,0213	<0,018	µg/kg		2,0682
PCB-122		ng/l				<0,0036	<0,0019	<0,0004	µg/kg		<0,024
PCB-123		ng/l				<0,0027	<0,0016	<0,0004	µg/kg		0,0225
PCB-128		ng/l				0,0086	0,0064	<0,0036	µg/kg		0,3189
PCB-138		ng/l				0,0513	0,0394	<0,017	µg/kg		2,2237
PCB-141		ng/l				0,0158	0,0089	<0,0034	µg/kg		0,2786
PCB-153		ng/l				0,0596	0,0338	<0,015	µg/kg		2,8969
PCB-156		ng/l				0,0110	0,0070	<0,0018	µg/kg		0,343
PCB-157		ng/l				<0,0040	<0,0019	<0,0003	µg/kg		0,0589
PCB-167		ng/l				<0,0029	<0,0015	<0,0005	µg/kg		0,0706
PCB-170		ng/l				0,0214	0,0262	<0,0032	µg/kg		0,6032
PCB-180		ng/l				0,0332	0,0360	<0,0061	µg/kg		1,1313
PCB-183		ng/l				0,0106	<0,0043	<0,0019	µg/kg		0,299
PCB-187		ng/l				0,0099	0,0096	<0,0034	µg/kg		0,5058
PCB-189		ng/l				<0,0047	<0,0027	<0,0003	µg/kg		<0,031
PCB-194		ng/l				<0,0078	0,0062	<0,0010	µg/kg		0,1109
PCB-206		ng/l				<0,0084	<0,0032	<0,0007	µg/kg		0,0705
PCB-209		ng/l				<0,0050	<0,0023	<0,0006	µg/kg		0,0641
Sum PCBs		ng/l				<1,3	<0,50	<0,37	µg/kg		<31
WHO-TEQ 2005 pg/l (upperbound)						0,0101	0,0145	0,0104	µg/kg		0,7612
WHO-TEQ 2005 pg/l (mediumbound)						0,0064	0,0080	0,0053	µg/kg		0,7607
WHO-TEQ 2005 pg/l (lowerbound)						0,0028	0,0014	0,0001	µg/kg		0,7602
Organotins											
Monobutyltin cation, MBT		ng/l	<1	<1	<1	10	4,1	4,1	µg/kg	560	340
Dibutyltin cation, DBT		ng/l	<1	<1	1	1	<1	1,2	µg/kg	240	190
Tributyltin cation, TBT		ng/l	<1	<1	<1	<1	<1	<1	µg/kg	3,3	4,6
Tetrabutyltin, TTBT		ng/l	<1	<1	<1	<1	<1	<1	µg/kg	*	*
Monooctyltin cation, MOT		ng/l	<1	<1	<1	<1	<1	<1	µg/kg	180	140

Diocetyl tin cation, DOT		ng/l	<1	<1	<1	<1	<1	<1	<1	µg/kg	80	85
Triphenyl tin cation, TPhT		ng/l	<1	<1	<1	<1	<1	<1	<1	µg/kg	<5	<1
Tricyclohexyl tin cation, TCyT		ng/l	<1	<1	<1	<1	<1	<1	<1	µg/kg	<5	1,2
Chlorinated paraffins												
SCCP	85535-84-8	µg/l	1,48	0,63	1,03	0,36	1,34	0,14		mg/kg	11,6	4,95
MCCP		µg/l		1,55	6,48	8,19	1,22	<0,6		mg/kg	0,123	<0,03
Endosulfane												
α-Endosulfane	959-98-8	µg/l	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004	mg/kg	0,0022	<0,001
β-Endosulfane	33213-65-9	µg/l	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004	mg/kg	<0,001	<0,001
Endosulfane sulphate	1031-07-8	µg/l	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	mg/kg	<0,001	<0,001
Metals												
Cadmium	7440-43-9	µg/l	<0,050	<0,050	<0,050	<0,050	<0,050	<0,050	<0,050	mg/kg	0,43	0,52
Mercury	7439-97-6	µg/l	<0,020	<0,020	<0,020	<0,020	<0,020	<0,020	<0,020	mg/kg	0,25	0,17

* - TTBT is not included in the method for sludge

The values above the detection limit but below the quantification limit are marked with **bold**.

The values below the detection limit are marked as nd (=not detected).

The values below the quantification limit (LOQ) are marked as <LOQ.

Appendix D: Industrial waste water treatment plant 1 (IWWTP1)

Table D-1. Basic data from WWTP.

Parameter	IWWTP1							
Sampling site								
Sampling date	May-2009	Jul-2009	Sep-2009	Nov-2009	Jan-2010	Apr-2010	Jun-2010	Aug-2010
Sampling time	15:50	11:15	14:30	13:40	13:20	10:45	9:10	9:05
Flow rate (m ³ /d)	480	400	360	360	320	410	377	500
BOD ₇ (mg/l)	402	90	56	102	135	1025	515	218
COD _{Cr} (mg/l)	764	414	167	231	380	1910	1438	1054
Suspended solids (mg/l)	62	13	12	21	55	108	76	5.6
Tot-P (mgP/l)	1.94	1.63	0.92	1.09	1.62	0.61	3.36	0.20
PO ₄ -P (mgP/l)		0.86	0.011	0.300	0.84	0.038	1.70	0.076
Tot-N (mgN/l)	9.54	3.12	3.7	4.14	9.8	9.45	15.1	7.94
NH ₄ -N (mgN/l)	0.34	2	0.03	1.51	1.04	2.39	1.26	0.68
Alkalinity (mmol/l)		7.12	5.88	7.74	3.65	3.59	2.97	3.91
pH	8.4	7.09	8.02	9.00	7.31	6.96	7.14	7.35
Conductivity (mS/m)	189.2	238	123.9	165.0	59	52.3	44.8	127
Fe (mgFe/l)	0.19	0.11	0.204	<0.020	0.082	0.26	0.180	0.036
t (°C)	26	26	25	20	4	31	35	35
SO ₄ ²⁻ (mg/l)		278	129.1	172	175	44	35	27
TOC (mgC/l)	239	152	69.5	73.3	96.1	543	437	319

Table D-2. Results of chemical analyses.

Name of substance	CAS	Unit	Waste water					
			Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
PBDEs								
BDE-17	147217-75-2	ng/l	0,03	nd	nd	nd	nd	nd
BDE-28	41318-75-6	ng/l	0,04	nd	nd	nd	nd	nd
BDE-47	5436-43-1	ng/l	0,17	0,05	nd	0,30	nd	nd
BDE-66	189084-61-5	ng/l	0,14	nd	nd	nd	nd	nd
BDE-85	182346-21-0	ng/l	0,55	nd	nd	nd	nd	nd
BDE-99	60348-60-9	ng/l	0,52	nd	nd	nd	nd	nd
BDE-100	189084-64-8	ng/l	0,40	nd	nd	nd	nd	nd
BDE-153	68631-49-2	ng/l	3,34	nd	nd	nd	nd	nd
BDE-154	207122-15-4	ng/l	nd	nd	nd	nd	nd	nd
BDE-183	207122-16-5	ng/l	8,82	nd	nd	nd	nd	nd
BDE-203		ng/l	5,50	nd	nd	nd	nd	nd
BDE-209	1163-19-5	ng/l	2,55	0,62	10,13	10,24	0,40	2,64
pentaBDE		ng/l	4,47	0,05	nd	0,30	nd	nd
octaBDE		ng/l	14,32	nd	nd	nd	nd	nd
decaBDE		ng/l	2,55	0,62	10,13	10,24	0,40	2,64
HBCDs								
α -HBCD		ng/l	0,33	nd	32,1	0,75	0,29	0,29
β -HBCD		ng/l	0,03	nd	7,27	0,37	0,14	nd
γ -HBCD		ng/l	0,39	nd	72,87	1,83	0,91	0,31
Perfluorinated substances								
perfluoro-n-hexanoic acid (PFHxA)	307-24-4	ng/l	0,3	nd	nd	nd	nd	nd
perfluorooctane sulfonate (PFOS)	1763-23-1	ng/l	nd	0,1	2,2	0,4	nd	nd
perfluorooctanoic acid (PFOA)	335-67-1	ng/l	1,7	1,0	2,2	0,5	1,2	1,7
perfluoro-n-decanoic acid (PFDA)	335-76-2	ng/l	0,0	nd	0,1	nd	nd	nd
Phenolic substances								
bisphenol a	80-05-7	μ g/l	nd	nd	nd	nd	0,13	nd
4-nonylphenol (mix.)	84852-15-3	μ g/l	nd	nd	0,33	0,50	nd	0,16
4-nonylphenol monoethoxylate (mix.)	-	μ g/l	nd	nd	0,05	0,06	nd	nd
4-nonylphenol diethoxylate (mix.)	-	μ g/l	nd	nd	nd	nd	nd	nd
octylphenol	140-66-9	μ g/l	nd	nd	nd	nd	nd	nd
octylphenol monoethoxylate	-	μ g/l	nd	nd	nd	nd	nd	nd

Waste water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
octylphenol diethoxylate	-	µg/l	nd	nd	nd	nd	nd	nd
Dioxins								
2,3,7,8-TCDD	1746-01-6	pg/l				0,4818	<0,24	<0,24
2,3,7,8-TCDF	51207-31-9	pg/l				<0,15	<0,096	<0,024
1,2,3,7,8-PeCDD	40321-76-4	pg/l				<0,52	<0,26	<0,035
1,2,3,7,8-PeCDF	57117-41-6	pg/l				<0,16	<0,13	<0,031
2,3,4,7,8-PeCDF	57117-31-4	pg/l				<0,19	<0,20	<0,065
1,2,3,4,7,8-HxCDD	39227-28-6	pg/l				<0,71	<0,46	<0,054
1,2,3,6,7,8-HxCDD	57653-85-7	pg/l				<0,59	<0,47	<0,053
1,2,3,7,8,9-HxCDD	19408-74-3	pg/l				<0,71	<0,44	<0,051
1,2,3,4,7,8-HxCDF	70648-26-9	pg/l				<0,37	0,4445	<0,078
1,2,3,6,7,8-HxCDF	57117-44-9	pg/l				<0,24	<0,18	<0,055
1,2,3,7,8,9-HxCDF	72918-21-9	pg/l				<1,2	<0,80	<0,083
2,3,4,6,7,8-HxCDF	60851-34-5	pg/l				<0,55	<0,31	<0,071
1,2,3,4,6,7,8-HpCDD	35822-46-9	pg/l				<1,3	<0,63	<0,089
1,2,3,4,6,7,8-HpCDF	67562-39-4	pg/l				0,4496	0,5359	<0,084
1,2,3,4,7,8,9-HpCDF	55673-89-7	pg/l				<1,2	<1,2	<0,063
OCDD	3268-87-9	pg/l				<1,2	<1,5	<0,35
OCDF	39001-02-0	pg/l				<3,2	<2,8	<0,15
Sum		pg/l				<13	<11	<1,6
WHO-TEQ 2005 (upperbound)		pg/l				1,5447	0,9104	0,3488
WHO-TEQ 2005 (mediumbound)		pg/l				1,0155	0,4801	0,1744
WHO-TEQ 2005 (lowerbound)		pg/l				0,4862	0,0498	0,0000
CO-PCB-77		pg/l				5,6065	1,7502	<0,96
CO-PCB-81		pg/l				0,2337	<0,27	<0,055
CO-PCB-126		pg/l				0,1638	<0,21	<0,076
CO-PCB-169		pg/l				<0,17	<0,17	<0,055
Sum CO-PCB		pg/l				< 6,2	< 2,4	< 1,1
WHO-TEQ 2005 (upperbound)		pg/l				0,0221	0,0261	0,0094
PCB-18		ng/l				0,3546	0,1146	0,0462
PCB-28/31		ng/l				0,6649	0,1757	0,0906
PCB-33		ng/l				0,2430	0,0474	0,0189
PCB-47		ng/l				0,8841	<0,043	<0,067
PCB-49		ng/l				0,0641	0,0282	<0,0038
PCB-51		ng/l				0,1721	0,0073	<0,0063

Waste water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
PCB-52		ng/l				0,1298	0,0497	<0,011
PCB-60		ng/l				0,0250	0,0088	0,0024
PCB-66		ng/l				0,0735	0,0304	<0,0079
PCB-74		ng/l				0,0459	0,0171	0,0059
PCB-99		ng/l				<0,021	0,0124	<0,0075
PCB-101		ng/l				0,1301	0,0332	<0,018
PCB-105		ng/l				0,0277	0,0101	<0,0069
PCB-110		ng/l				0,0993	0,0371	<0,019
PCB-114		ng/l				<0,018	<0,0015	<0,0004
PCB-118		ng/l				0,0851	0,0287	<0,018
PCB-122		ng/l				<0,020	<0,0018	<0,0004
PCB-123		ng/l				<0,015	<0,0014	<0,0003
PCB-128		ng/l				0,0702	0,0149	0,0063
PCB-138		ng/l				0,3671	0,0916	0,0442
PCB-141		ng/l				0,1128	0,0217	0,0133
PCB-153		ng/l				0,3274	0,0759	0,0389
PCB-156		ng/l				0,1084	0,0171	0,0102
PCB-157		ng/l				<0,019	<0,0015	0,0007
PCB-167		ng/l				0,0267	0,0037	0,0023
PCB-170		ng/l				0,3319	0,0737	0,0518
PCB-180		ng/l				0,4964	0,0974	0,0755
PCB-183		ng/l				0,0828	0,0152	0,0182
PCB-187		ng/l				0,1495	0,0231	0,0234
PCB-189		ng/l				<0,024	0,0049	0,0026
PCB-194		ng/l				0,0885	0,0155	0,0123
PCB-206		ng/l				<0,030	<0,0021	<0,0007
PCB-209		ng/l				<0,033	<0,0018	<0,0004
Sum PCBs		ng/l				< 5,3	< 1,1	<0,63
WHO-TEQ 2005 (upperbound)		ng/l				0,0342	0,0286	0,0109
WHO-TEQ 2005 (mediumbound)		ng/l				0,0306	0,0156	0,0058
WHO-TEQ 2005 (lowerbound)		ng/l				0,0269	0,0025	0,0007
Organotins								
Monobutyltin cation, MBT		ng/l	<1	<1	<1	1,3	<1	<1
Dibutyltin cation, DBT		ng/l	<1	<1	1,4	<1	<1	<1
Tributyltin cation, TBT		ng/l	<1	<1	<1	<1	<1	<1

Waste water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
Tetrabutyltin, TTBT		ng/l	<1	<1	<1	<1	<1	<1
Monooctyltin cation, MOT		ng/l	<1	<1	<1	<1	<1	<1
Dioctyltin cation, DOT		ng/l	<1	<1	<1	<1	<1	<1
Triphenyltin cation, TPhT		ng/l	<1	<1	<1	<1	<1	<1
Tricyclohexyltin cation, TCyT		ng/l	<1	<1	<1	<1	<1	<1
Chlorinated paraffins								
SCCP	85535-84-8	µg/l	0,58	0,39	0,89	1,10	0,53	0,89
MCCP		µg/l		1,30	2,64	3,61	3,31	1,16
Endosulfane								
α-Endosulfane	959-98-8	µg/l	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004
β-Endosulfane	33213-65-9	µg/l	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004
Endosulfane sulphate	1031-07-8	µg/l	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Metals								
Cadmium	7440-43-9	µg/l	<0,050	<0,050	<0,050	<0,050	<0,050	<0,050
Mercury	7439-97-6	µg/l	0,023	<0,020	<0,020	0,037	0,020	<0,020

The values above the detection limit but below the quantification limit are marked with **bold**.

The values below the detection limit are marked as nd (=not detected).

The values below the quantification limit (LOQ) are marked as <LOQ.

Appendix E: Industrial waste water treatment plant 2 (IWWTP2)

Table E-1. Basic data from WWTP.

Parameter Sampling site	IWWTP2							
	May- 2009	Jul- 2009	Sep- 2009	Nov- 2009	Jan- 2010	Apr- 2010	Jun- 2010	Aug- 2010
Sampling date								
Sampling time	9:30	9:15	10:50	9:00	9:20	8:45	9:20	9:00
Flow rate (m ³ /d)	960	608	1220	1164	657	850	514	712
BOD ₇ (mg/l)	<3.0	1	1.8	<3.0	<3.0	<3.0	<3.0	<3.0
COD _{Cr} (mg/l)	75	29	25	52	34	30	44	47
Suspended solids (mg/l)	2	1	0.85	2.0	<1.8	<1.9	<1.9	<1.9
Tot-P (mgP/l)	0.18	0.22	0.3	0.13	0.16	0.17	0.21	0.17
PO ₄ -P (mgP/l)		0.16	0.25	0.103	0.17	0.17	0.17	0.130
Tot-N (mgN/l)	4.18	4.00	2.9	8.19	11.1	2.56	5.45	3.23
NH ₄ -N (mgN/l)	0.12	0.18	0.52	0.098	0.1	0.1	0.23	0.32
Alkalinity (mmol/l)		3.3	3.39	3.11	2.13	3.62	3.97	3.23
pH	7.43	7.2	7.22	7.22	7.01	7.43	7.31	7.34
Conductivity (mS/m)	514.3	277	205	90.5	101.9	81.2	252	84.9
Fe (mgFe/l)	0.040	0.066	0.072	0.066	0.031	0.062	0.270	0.21
t (°C)	25	26	33	24	24	22	22	23
SO ₄ ²⁻ (mg/l)		90	71.2	25.6	69.9	55	81	30
TOC (mgC/l)	8.31	10.0	9.61	9.34	6.67	6.08	7.73	6.10

Table E-2. Results of chemical analyses.

Waste water								
Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
PBDEs								
BDE-17	147217-75-2	ng/l	-	nd	nd	nd	nd	nd
BDE-28	41318-75-6	ng/l	-	nd	nd	nd	nd	nd
BDE-47	5436-43-1	ng/l	-	nd	nd	nd	nd	nd
BDE-66	189084-61-5	ng/l	-	nd	nd	nd	nd	nd
BDE-85	182346-21-0	ng/l	-	nd	nd	nd	nd	nd
BDE-99	60348-60-9	ng/l	-	nd	nd	nd	nd	nd
BDE-100	189084-64-8	ng/l	-	nd	nd	nd	nd	nd
BDE-153	68631-49-2	ng/l	-	nd	nd	nd	nd	nd
BDE-154	207122-15-4	ng/l	-	nd	nd	nd	nd	nd
BDE-183	207122-16-5	ng/l	-	nd	nd	nd	nd	nd
BDE-203		ng/l	-	nd	nd	nd	nd	nd
BDE-209	1163-19-5	ng/l	-	0,07	1,52	nd	0,08	0,87
pentaBDE		ng/l	-	nd	nd	nd	nd	nd
octaBDE		ng/l	-	nd	nd	nd	nd	nd
decaBDE		ng/l	-	0,07	1,52	nd	0,08	0,87
HBCDs								
α -HBCD		ng/l	nd	0,02	nd	0,28	nd	nd
β -HBCD		ng/l	0,04	nd	nd	nd	nd	nd
γ -HBCD		ng/l	0,32	nd	nd	0,87	nd	0,38
Perfluorinated substances								
perfluoro-n-hexanoic acid (PFHxA)	307-24-4	ng/l	1,7	1,1	nd	0,6	1,2	4,1
perfluorooctane sulfonate (PFOS)	1763-23-1	ng/l	0,1	0,2	1,7	0,4	nd	1,2
perfluorooctanoic acid (PFOA)	335-67-1	ng/l	2,4	1,6	2,2	1,0	3,3	6,4
perfluoro-n-decanoic acid (PFDA)	335-76-2	ng/l	0,2	0,2	0,1	nd	nd	0,5
Phenolic substances								
bisphenol a	80-05-7	μ g/l	0,28	0,20	nd	nd	0,17	0,30
4-nonylphenol (mix.)	84852-15-3	μ g/l	nd	0,16	0,30	0,37	nd	0,12
4-nonylphenol monoethoxylate (mix.)	-	μ g/l	0,05	nd	nd	nd	nd	nd
4-nonylphenol diethoxylate (mix.)	-	μ g/l	nd	nd	nd	nd	nd	nd
octylphenol	140-66-9	μ g/l	nd	nd	0,19	nd	nd	nd
octylphenol monoethoxylate	-	μ g/l	nd	nd	nd	nd	nd	nd

Waste water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
octylphenol diethoxylate	-	µg/l	nd	nd	nd	nd	nd	nd
Dioxins								
2,3,7,8-TCDD	1746-01-6	pg/l				<0,23	<0,21	<0,24
2,3,7,8-TCDF	51207-31-9	pg/l				<0,10	<0,13	<0,024
1,2,3,7,8-PeCDD	40321-76-4	pg/l				<0,27	<0,24	<0,049
1,2,3,7,8-PeCDF	57117-41-6	pg/l				<0,10	<0,13	<0,031
2,3,4,7,8-PeCDF	57117-31-4	pg/l				<0,12	<0,14	<0,065
1,2,3,4,7,8-HxCDD	39227-28-6	pg/l				<0,36	<0,31	<0,057
1,2,3,6,7,8-HxCDD	57653-85-7	pg/l				<0,33	<0,31	<0,057
1,2,3,7,8,9-HxCDD	19408-74-3	pg/l				<0,35	<0,30	<0,054
1,2,3,4,7,8-HxCDF	70648-26-9	pg/l				<0,21	<0,15	<0,078
1,2,3,6,7,8-HxCDF	57117-44-9	pg/l				<0,18	<0,15	<0,055
1,2,3,7,8,9-HxCDF	72918-21-9	pg/l				<0,69	<0,63	<0,083
2,3,4,6,7,8-HxCDF	60851-34-5	pg/l				<0,26	<0,25	<0,091
1,2,3,4,6,7,8-HpCDD	35822-46-9	pg/l				<0,35	<0,59	<0,089
1,2,3,4,6,7,8-HpCDF	67562-39-4	pg/l				<0,12	<0,23	<0,084
1,2,3,4,7,8,9-HpCDF	55673-89-7	pg/l				<0,57	<0,92	<0,064
OCDD	3268-87-9	pg/l				<0,72	<0,92	<0,35
OCDF	39001-02-0	pg/l				<1,3	<2,8	<0,19
Sum		pg/l				<6,2	<8,4	<1,7
WHO-TEQ 2005 (upperbound)		pg/l				0,7966	0,7390	0,3658
WHO-TEQ 2005 (mediumbound)		pg/l				0,3983	0,3695	0,1829
WHO-TEQ 2005 (lowerbound)		pg/l				0,0000	0,0000	0,0000
CO-PCB-77		pg/l				2,2795	<0,47	<0,96
CO-PCB-81		pg/l				0,1990	<0,13	0,0631
CO-PCB-126		pg/l				<0,11	<0,092	<0,076
CO-PCB-169		pg/l				<0,15	<0,098	<0,055
Sum CO-PCB		pg/l				< 2,7	< 0,80	< 1,1
WHO-TEQ 2005 (upperbound)		pg/l				0,0153	0,0122	0,0094
PCB-18		ng/l				0,0968	0,0112	<0,012
PCB-28/31		ng/l				0,1356	0,0257	0,0238
PCB-33		ng/l				0,0470	0,0082	0,0077
PCB-47		ng/l				0,0488	<0,043	<0,067
PCB-49		ng/l				0,0553	0,0087	0,0042
PCB-51		ng/l				0,0101	<0,0060	0,0085

Waste water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
PCB-52		ng/l				0,1158	0,0125	0,0155
PCB-60		ng/l				0,0128	0,0013	0,0027
PCB-66		ng/l				0,0445	0,0051	0,0115
PCB-74		ng/l				0,0266	0,0039	0,0086
PCB-99		ng/l				0,0142	<0,0019	<0,0075
PCB-101		ng/l				0,0603	0,0069	<0,018
PCB-105		ng/l				0,0073	<0,0018	<0,0069
PCB-110		ng/l				0,0282	0,0043	<0,019
PCB-114		ng/l				<0,0033	<0,0014	<0,0004
PCB-118		ng/l				0,0248	<0,0039	<0,018
PCB-122		ng/l				<0,0039	<0,0015	<0,0004
PCB-123		ng/l				<0,0027	<0,0012	<0,0004
PCB-128		ng/l				<0,0063	<0,0022	<0,0036
PCB-138		ng/l				0,0244	<0,0042	0,0170
PCB-141		ng/l				0,0141	<0,0026	0,0044
PCB-153		ng/l				0,0357	<0,0043	0,0167
PCB-156		ng/l				<0,0046	<0,0014	0,0031
PCB-157		ng/l				<0,0045	<0,0015	<0,0004
PCB-167		ng/l				<0,0037	<0,0015	<0,0005
PCB-170		ng/l				<0,0084	<0,0033	0,0164
PCB-180		ng/l				<0,0069	<0,0026	0,0247
PCB-183		ng/l				<0,0087	<0,0035	0,0052
PCB-187		ng/l				<0,0067	<0,0030	0,0064
PCB-189		ng/l				<0,0054	<0,0024	0,0007
PCB-194		ng/l				<0,0070	<0,0024	0,0037
PCB-206		ng/l				<0,0081	<0,0023	<0,0006
PCB-209		ng/l				<0,0057	<0,0020	<0,0003
Sum PCBs		ng/l				< 0,89	< 0,19	<0,34
WHO-TEQ 2005 (upperbound)		ng/l				0,0170	0,0127	0,0104
WHO-TEQ 2005 (mediumbound)		ng/l				0,0091	0,0063	0,0053
WHO-TEQ 2005 (lowerbound)		ng/l				0,0013	0,0000	0,0002
Organotins								
Monobutyltin cation, MBT		ng/l	<1	<1	2,4	<1	1,2	<1
Dibutyltin cation, DBT		ng/l	<1	<1	<1	<1	<1	<1
Tributyltin cation, TBT		ng/l	<1	<1	<1	<1	<1	<1

Waste water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
Tetrabutyltin, TTBT		ng/l	<1	<1	<1	<1	<1	<1
Monooctyltin cation, MOT		ng/l	<1	<1	<1	<1	<1	<1
Diocetyl tin cation, DOT		ng/l	<1	<1	<1	<1	<1	<1
Triphenyltin cation, TPhT		ng/l	<1	<1	<1	<1	<1	<1
Tricyclohexyltin cation, TCyT		ng/l	<1	<1	<1	<1	<1	<1
Chlorinated paraffins								
SCCP	85535-84-8	µg/l	1,06	1,06	0,41	1,45	1,26	0,82
MCCP		µg/l		0,60	4,91	2,56	4,00	3,47
Endosulfane								
α-Endosulfane	959-98-8	µg/l	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004
β-Endosulfane	33213-65-9	µg/l	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004
Endosulfane sulphate	1031-07-8	µg/l	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
Metals								
Cadmium	7440-43-9	µg/l	<0,050	<0,050	<0,050	<0,050	<0,050	<0,050
Mercury	7439-97-6	µg/l	<0,020	0,044	<0,020	<0,020	<0,020	0,020

The values above the detection limit but below the quantification limit are marked with **bold**.

The values below the detection limit are marked as nd (=not detected).

The values below the quantification limit (LOQ) are marked as <LOQ.

Appendix F: Storm water (SW)

Table F-1. Basic data from storm water discharger.

Parameter Sampling site	SW	
	Nov-2009	Jun-2010
Sampling date	Nov-2009	Jun-2010
Sampling time	10:25	17:20
Flow rate (m ³ /d)	---	---
BOD ₇ (mg/l)	<3.0	<3.0
COD _{Cr} (mg/l)	33	36
Suspended solids (mg/l)	9.6	4.8
Tot-P (mgP/l)	0.13	0.24
PO ₄ -P (mgP/l)	0.056	0.11
Tot-N (mgN/l)	3.88	2.51
NH ₄ -N (mgN/l)	0.14	0.15
Alkalinity (mmol/l)	6.92	7.51
pH	7.44	7.66
Conductivity (mS/m)	85.8	115
Fe (mgFe/l)	0.6	0.530
t (°C)	7	14
SO ₄ ²⁻ (mg/l)	71.7	105
TOC (mgC/l)	6.38	7.68

Table F-2. Results of chemical analyses.

Name of substance	CAS	Unit	Storm water					
			Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
PBDEs								
BDE-17	147217-75-2	ng/l		nd			nd	
BDE-28	41318-75-6	ng/l		nd			nd	
BDE-47	5436-43-1	ng/l		0,05			0,05	
BDE-66	189084-61-5	ng/l		nd			nd	
BDE-85	182346-21-0	ng/l		nd			nd	
BDE-99	60348-60-9	ng/l		0,10			nd	
BDE-100	189084-64-8	ng/l		nd			nd	
BDE-153	68631-49-2	ng/l		nd			nd	
BDE-154	207122-15-4	ng/l		nd			nd	
BDE-183	207122-16-5	ng/l		nd			nd	
BDE-203		ng/l		nd			nd	
BDE-209	1163-19-5	ng/l		0,19			0,09	
pentaBDE		ng/l		0,15			0,05	
octaBDE		ng/l		nd			nd	
decaBDE		ng/l		0,19			0,09	
HBCDs								
α -HBCD		ng/l		nd			0,34	
β -HBCD		ng/l		nd			0,15	
γ -HBCD		ng/l		nd			0,46	
Perfluorinated substances								
perfluoro-n-hexanoic acid (PFHxA)	307-24-4	ng/l		0,1			0,4	
perfluorooctane sulfonate (PFOS)	1763-23-1	ng/l		3,9			0,8	
perfluorooctanoic acid (PFOA)	335-67-1	ng/l		1,5			2,9	
perfluoro-n-decanoic acid (PFDA)	335-76-2	ng/l		0,2			0,9	
Phenolic substances								
bisphenol a	80-05-7	μ g/l		0,14			0,11	
4-nonylphenol (mix.)	84852-15-3	μ g/l		0,19			nd	
4-nonylphenol monoethoxylate (mix.)	-	μ g/l		nd			nd	
4-nonylphenol diethoxylate (mix.)	-	μ g/l		nd			nd	
octylphenol	140-66-9	μ g/l		nd			nd	

Storm water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
octylphenol monoethoxylate	-	µg/l		nd			nd	
octylphenol diethoxylate	-	µg/l		nd			nd	
Dioxins								
2,3,7,8-TCDD	1746-01-6	pg/l					<0,30	
2,3,7,8-TCDF	51207-31-9	pg/l					<0,15	
1,2,3,7,8-PeCDD	40321-76-4	pg/l					<0,29	
1,2,3,7,8-PeCDF	57117-41-6	pg/l					<0,17	
2,3,4,7,8-PeCDF	57117-31-4	pg/l					<0,17	
1,2,3,4,7,8-HxCDD	39227-28-6	pg/l					<0,44	
1,2,3,6,7,8-HxCDD	57653-85-7	pg/l					<0,42	
1,2,3,7,8,9-HxCDD	19408-74-3	pg/l					<0,42	
1,2,3,4,7,8-HxCDF	70648-26-9	pg/l					<0,19	
1,2,3,6,7,8-HxCDF	57117-44-9	pg/l					<0,20	
1,2,3,7,8,9-HxCDF	72918-21-9	pg/l					<0,83	
2,3,4,6,7,8-HxCDF	60851-34-5	pg/l					<0,32	
1,2,3,4,6,7,8-HpCDD	35822-46-9	pg/l					<0,62	
1,2,3,4,6,7,8-HpCDF	67562-39-4	pg/l					<0,30	
1,2,3,4,7,8,9-HpCDF	55673-89-7	pg/l					<1,1	
OCDD	3268-87-9	pg/l					<1,3	
OCDF	39001-02-0	pg/l					<3,8	
Sum		pg/l					<11	
WHO-TEQ 2005 (upperbound)		pg/l					0,9577	
WHO-TEQ 2005 (mediumbound)		pg/l					0,4788	
WHO-TEQ 2005 (lowerbound)		pg/l					0,0000	
CO-PCB-77		pg/l					8,2237	
CO-PCB-81		pg/l					0,3156	
CO-PCB-126		pg/l					<0,12	
CO-PCB-169		pg/l					<0,15	
Sum CO-PCB		pg/l					< 8,8	
WHO-TEQ 2005 (upperbound)		pg/l					0,0172	
PCB-18		ng/l					0,1064	
PCB-28/31		ng/l					0,1593	
PCB-33		ng/l					0,0180	
PCB-47		ng/l					0,0464	
PCB-49		ng/l					0,0487	

Storm water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
PCB-51		ng/l					0,0103	
PCB-52		ng/l					0,0736	
PCB-60		ng/l					0,0203	
PCB-66		ng/l					0,0745	
PCB-74		ng/l					0,0331	
PCB-99		ng/l					0,0126	
PCB-101		ng/l					0,0297	
PCB-105		ng/l					0,0113	
PCB-110		ng/l					0,0324	
PCB-114		ng/l					<0,0015	
PCB-118		ng/l					0,0239	
PCB-122		ng/l					<0,0017	
PCB-123		ng/l					<0,0014	
PCB-128		ng/l					0,0081	
PCB-138		ng/l					0,0348	
PCB-141		ng/l					0,0090	
PCB-153		ng/l					0,0323	
PCB-156		ng/l					0,0063	
PCB-157		ng/l					<0,0015	
PCB-167		ng/l					0,0017	
PCB-170		ng/l					0,0198	
PCB-180		ng/l					0,0293	
PCB-183		ng/l					0,0066	
PCB-187		ng/l					0,0092	
PCB-189		ng/l					<0,0024	
PCB-194		ng/l					0,0066	
PCB-206		ng/l					<0,0028	
PCB-209		ng/l					<0,0017	
Sum of PCBs		ng/l					<0,89	
WHO-TEQ 2005 (upperbound)		ng/l					0,0188	
WHO-TEQ 2005 (mediumbound)		ng/l					0,0106	
WHO-TEQ 2005 (lowerbound)		ng/l					0,0023	
Organotins								
Monobutyltin cation, MBT		ng/l		<1			<1	
Dibutyltin cation, DBT		ng/l		<1			<1	

Storm water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
Tributyltin cation, TBT		ng/l		<1			<1	
Tetrabutyltin, TTBT		ng/l		<1			<1	
Monooctyltin cation, MOT		ng/l		<1			<1	
Dioctyltin cation, DOT		ng/l		<1			<1	
Triphenyltin cation, TPhT		ng/l		<1			<1	
Tricyclohexyltin cation, TCyT		ng/l		<1			<1	
Chlorinated paraffins								
SCCP	85535-84-8	µg/l		0,36			1,14	
MCCP				0,86			3,64	
Endosulfane								
α-Endosulfane	959-98-8	µg/l		<0,004			<0,004	
β-Endosulfane	33213-65-9	µg/l		<0,004			<0,004	
Endosulfane sulphate	1031-07-8	µg/l		<0,005			<0,005	
Metals								
Cadmium	7440-43-9	µg/l		<0,050			<0,050	
Mercury	7439-97-6	µg/l		<0,020			0,023	

The values above the detection limit but below the quantification limit are marked with **bold**.

The values below the detection limit are marked as nd (=not detected).

The values below the quantification limit (LOQ) are marked as <LOQ.

Appendix G: Landfill leachate (LW)

Table G-1. Basic data from landfill.

Parameter	LW	
Sampling site		
Sampling date	Nov-2009	Jun-2010
Sampling time	9:20	11:30
Flow rate (m ³ /d)	---	7
BOD ₇ (mg/l)	13	52
COD _{Cr} (mg/l)	18	239
Suspended solids (mg/l)	<1.8	5.2
Tot-P (mgP/l)	0.024	0.046
PO ₄ -P (mgP/l)	0.021	0.015
Tot-N (mgN/l)	6.48	16.0
NH ₄ -N (mgN/l)	1.79	5.96
Alkalinity (mmol/l)	0.97	0.36
pH	4.70	4.72
Conductivity (mS/m)	90.0	23.6
Fe (mgFe/l)	0.043	0.032
t (°C)	18	18
SO ₄ ²⁻ (mg/l)	20.5	16
TOC (mgC/l)	<1.01	38.0

Table G-2. Results of chemical analyses.

Landfill water								
Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
PBDEs								
BDE-17	147217-75-2	ng/l		nd			nd	
BDE-28	41318-75-6	ng/l		nd			nd	
BDE-47	5436-43-1	ng/l		nd			nd	
BDE-66	189084-61-5	ng/l		nd			nd	
BDE-85	182346-21-0	ng/l		nd			nd	
BDE-99	60348-60-9	ng/l		nd			nd	
BDE-100	189084-64-8	ng/l		nd			nd	
BDE-153	68631-49-2	ng/l		nd			nd	
BDE-154	207122-15-4	ng/l		nd			nd	
BDE-183	207122-16-5	ng/l		nd			nd	
BDE-203		ng/l		nd			nd	
BDE-209	1163-19-5	ng/l		0,07			0,08	
pentaBDE		ng/l		nd			nd	
octaBDE		ng/l		nd			nd	
decaBDE		ng/l		0,07			0,08	
HBCDs								
α -HBCD		ng/l		nd			nd	
β -HBCD		ng/l		nd			nd	
γ -HBCD		ng/l		nd			nd	
Perfluorinated substances								
perfluoro-n-hexanoic acid (PFHxA)	307-24-4	ng/l		nd			nd	
perfluorooctane sulfonate (PFOS)	1763-23-1	ng/l		nd			nd	
perfluorooctanoic acid (PFOA)	335-67-1	ng/l		1,4			nd	
perfluoro-n-decanoic acid (PFDA)	335-76-2	ng/l		nd			nd	
Phenolic substances								
bisphenol a	80-05-7	μ g/l		7,27			2,59	
4-nonylphenol (mix.)	84852-15-3	μ g/l		0,23			0,20	
4-nonylphenol monoethoxylate (mix.)	-	μ g/l		nd			nd	
4-nonylphenol diethoxylate (mix.)	-	μ g/l		nd			nd	
octylphenol	140-66-9	μ g/l		nd			nd	
octylphenol monoethoxylate	-	μ g/l		nd			nd	

Landfill water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
octylphenol diethoxylate	-	µg/l		nd			nd	
Dioxins								
2,3,7,8-TCDD	1746-01-6	pg/l					<0,21	
2,3,7,8-TCDF	51207-31-9	pg/l					<0,10	
1,2,3,7,8-PeCDD	40321-76-4	pg/l					<0,24	
1,2,3,7,8-PeCDF	57117-41-6	pg/l					<0,13	
2,3,4,7,8-PeCDF	57117-31-4	pg/l					<0,15	
1,2,3,4,7,8-HxCDD	39227-28-6	pg/l					<0,36	
1,2,3,6,7,8-HxCDD	57653-85-7	pg/l					<0,37	
1,2,3,7,8,9-HxCDD	19408-74-3	pg/l					<0,35	
1,2,3,4,7,8-HxCDF	70648-26-9	pg/l					<0,18	
1,2,3,6,7,8-HxCDF	57117-44-9	pg/l					<0,17	
1,2,3,7,8,9-HxCDF	72918-21-9	pg/l					<0,73	
2,3,4,6,7,8-HxCDF	60851-34-5	pg/l					<0,29	
1,2,3,4,6,7,8-HpCDD	35822-46-9	pg/l					<0,54	
1,2,3,4,6,7,8-HpCDF	67562-39-4	pg/l					<0,26	
1,2,3,4,7,8,9-HpCDF	55673-89-7	pg/l					<1,3	
OCDD	3268-87-9	pg/l					<1,1	
OCDF	39001-02-0	pg/l					<3,0	
Sum		pg/l					<9,5	
WHO-TEQ 2005 (upperbound)		pg/l					0,7783	
WHO-TEQ 2005 (mediumbound)		pg/l					0,3892	
WHO-TEQ 2005 (lowerbound)		pg/l					0,0000	
CO-PCB-77		pg/l					0,6035	
CO-PCB-81		pg/l					<0,10	
CO-PCB-126		pg/l					<0,097	
CO-PCB-169		pg/l					<0,12	
Sum CO-PCB		pg/l					< 0,92	
WHO-TEQ 2005 (upperbound)		pg/l					0,0132	
PCB-18		ng/l					0,1099	
PCB-28/31		ng/l					0,0630	
PCB-33		ng/l					0,0201	
PCB-47		ng/l					<0,043	
PCB-49		ng/l					0,0099	
PCB-51		ng/l					<0,0060	

Landfill water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
PCB-52		ng/l					0,0249	
PCB-60		ng/l					0,0028	
PCB-66		ng/l					0,0115	
PCB-74		ng/l					0,0073	
PCB-99		ng/l					0,0053	
PCB-101		ng/l					0,0159	
PCB-105		ng/l					0,0039	
PCB-110		ng/l					0,0165	
PCB-114		ng/l					<0,0016	
PCB-118		ng/l					0,0114	
PCB-122		ng/l					<0,0018	
PCB-123		ng/l					<0,0014	
PCB-128		ng/l					0,0056	
PCB-138		ng/l					0,0249	
PCB-141		ng/l					0,0079	
PCB-153		ng/l					0,0215	
PCB-156		ng/l					0,0040	
PCB-157		ng/l					<0,0014	
PCB-167		ng/l					<0,0015	
PCB-170		ng/l					0,0144	
PCB-180		ng/l					0,0199	
PCB-183		ng/l					0,0060	
PCB-187		ng/l					0,0062	
PCB-189		ng/l					<0,0023	
PCB-194		ng/l					<0,0023	
PCB-206		ng/l					<0,0027	
PCB-209		ng/l					<0,0025	
Sum PCBs		ng/l					< 0,48	
WHO-TEQ 2005 (upperbound)		ng/l					0,0141	
WHO-TEQ 2005 (mediumbound)		ng/l					0,0074	
WHO-TEQ 2005 (lowerbound)		ng/l					0,0008	
Organotins								
Monobutyltin cation, MBT		ng/l		<1			59	
Dibutyltin cation, DBT		ng/l		<1			<5	
Tributyltin cation, TBT		ng/l		<1			<5	

Landfill water

Name of substance	CAS	Unit	Sep-09	Nov-09	Jan-10	Apr-10	Jun-10	Aug-10
Tetrabutyltin, TTBT		ng/l		<1			<5	
Monooctyltin cation, MOT		ng/l		<1			9,3	
Diocetyl tin cation, DOT		ng/l		<1			<10	
Triphenyltin cation, TPhT		ng/l		<1			<5	
Tricyclohexyltin cation, TCyT		ng/l		<1			<5	
Chlorinated paraffins								
SCCP	85535-84-8	µg/l		0,69			0,97	
MCCP		µg/l		1,54			4,11	
Endosulfane								
α-Endosulfane	959-98-8	µg/l		<0,004			<0,004	
β-Endosulfane	33213-65-9	µg/l		<0,004			<0,004	
Endosulfane sulphate	1031-07-8	µg/l		<0,005			<0,005	
Metals								
Cadmium	7440-43-9	µg/l		<0,050			<0,050	
Mercury	7439-97-6	µg/l		<0,020			<0,020	

The values above the detection limit but below the quantification limit are marked with **bold**.

The values below the detection limit are marked as nd (=not detected).

The values below the quantification limit (LOQ) are marked as <LOQ.

Appendix H: Directive 2008/105/EC on environmental quality standards

Environmental quality standards for priority substances and certain other pollutants

Part A: Environmental quality standards (EQS)

AA: annual average;

MAC: maximum allowable concentration.

Unit [$\mu\text{g/l}$]

Name of substance	CAS number ¹	AA-EQS ² Inland surface waters ⁽³⁾	AA-EQS ² Other surface waters	MAC-EQS ⁴ Inland surface waters ⁽³⁾	MAC-EQS ⁴ Other surface waters
Alachlor	15972-60-8	0.3	0.3	0.7	0.7
Anthracene	120-12-7	0.1	0.1	0.4	0.4
Atrazine	1912-24-9	0.6	0.6	2.0	2.0
Benzene	71-43-2	10	8	50	50
Brominated diphenylether ⁵	32534-81-9	0.0005	0.0002	not applicable	not applicable
Cadmium and its compounds (depending on water hardness classes) ⁶	7440-43-9	≤ 0.08 (Class 1) 0.08 (Class 2) 0.09 (Class 3) 0.15 (Class 4) 0.25 (Class 5)	0.2	≤ 0.45 (Class 1) 0.45 (Class 2) 0.6 (Class 3) 0.9 (Class 4) 1.5 (Class 5)	≤ 0.45 (Class 1) 0.45 (Class 2) 0.6 (Class 3) 0.9 (Class 4) 1.5 (Class 5)
Carbon-tetrachloride ⁷	56-23-5	12	12	not applicable	not applicable
C10-13 Chloroalkanes	85535-84-8	0.4	0.4	1.4	1.4
Chlorfenvinphos	470-90-6	0.1	0.1	0.3	0.3
Chlorpyrifos (Chlorpyrifos-ethyl)	2921-88-2	0.03	0.03	0.1	0.1
Cyclodiene pesticides: Aldrin ⁷ Dieldrin ⁷ Endrin ⁷ Isodrin ⁷	309-00-2 60-57-1 72-20-8 465-73-6	$\Sigma = 0.01$	$\Sigma = 0.005$	not applicable	not applicable
DDT total ^{7, 8}	not applicable	0.025	0.025	not applicable	not applicable
para-para-DDT ⁷	50-29-3	0.01	0.01	not applicable	not applicable
1,2-Dichloroethane	107-06-2	10	10	not applicable	not applicable
Dichloromethane	75-09-2	20	20	not applicable	not applicable
Di(2-ethylhexyl)-phthalate (DEHP)	117-81-7	1.3	1.3	not applicable	not applicable
Diuron	330-54-1	0.2	0.2	1.8	1.8
Endosulfan	115-29-7	0.005	0.0005	0.01	0.004
Fluoranthene	206-44-0	0.1	0.1	1	1
Hexachlorobenzene	118-74-1	0.01 ⁹	0.01 ⁹	0.05	0.05
Hexachlorobutadiene	87-68-3	0.1 ⁹	0.1 ⁹	0.6	0.6
Hexachlorocyclohexane	608-73-1	0.02	0.002	0.04	0.02
Isoproturon	34123-59-6	0.3	0.3	1.0	1.0
Lead and its compounds	7439-92-1	7.2	7.2	not applicable	not applicable
Mercury and its compounds	7439-97-6	0.05 ⁹	0.05 ⁹	0.07	0.07
Naphthalene	91-20-3	2.4	1.2	not applicable	not applicable
Nickel and its compounds	7440-02-0	20	20	not applicable	not applicable

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Nonylphenol (4-Nonylphenol)	104-40-5	0.3	0.3	2.0	2.0
Octylphenol ((4-(1,1',3,3'-tetramethylbutyl)-phenol))	140-66-9	0.1	0.01	not applicable	not applicable
Pentachlorobenzene	608-93-5	0.007	0.0007	not applicable	not applicable
Pentachlorophenol	87-86-5	0.4	0.4	1	1
Polyaromatic hydrocarbons (PAH) ¹⁰	not applicable				
Benzo(a)pyrene	50-32-8	0.05	0.05	0.1	0.1
Benzo(b)fluoranthene	205-99-2	Σ= 0.03	Σ= 0.03	not applicable	not applicable
Benzo(k)fluoranthene	207-08-9				
Benzo(g,h,i)perylene	191-24-2	Σ= 0.002	Σ= 0.002	not applicable	not applicable
Indeno(1,2,3-cd)pyrene	193-39-5				
Simazine	122-34-9	1	1	4	4
Tetrachloroethylene ⁷	127-18-4	10	10	not applicable	not applicable
Trichloroethylene ⁷	79-01-6	10	10	not applicable	not applicable
Tributyltin compounds (Tributyltin-cation)	36643-28-4	0.0002	0.0002	0.0015	0.0015
Trichlorobenzenes	12002-48-1	0.4	0.4	not applicable	not applicable
Trichloromethane	67-66-3	2.5	2.5	not applicable	not applicable
Trifluralin	1582-09-8	0.03	0.03	not applicable	not applicable

¹CAS: Chemical Abstracts Service.

² This parameter is the EQS expressed as an annual average value (AA-EQS). Unless otherwise specified, it applies to the total concentration of all isomers.

³ Inland surface waters encompass rivers and lakes and related artificial or heavily modified water bodies.

⁴ This parameter is the EQS expressed as a maximum allowable concentration (MAC-EQS). Where the MAC-EQS are marked as 'not applicable', the AA-EQS values are considered protective against short-term pollution peaks in continuous discharges since they are significantly lower than the values derived on the basis of acute toxicity.

⁵ For the group of priority substances covered by brominated diphenylethers listed in Decision No 2455/2001/EC, an EQS is established only for congener numbers 28, 47, 99, 100, 153 and 154.

⁶ For cadmium and its compounds (No 6) the EQS values vary depending on the hardness of the water as specified in five class categories (Class 1: < 40 mg CaCO₃/l, Class 2: 40 to < 50 mg CaCO₃/l, Class 3: 50 to < 100 mg CaCO₃/l, Class 4: 100 to < 200 mg CaCO₃/l and Class 5: ≥ 200 mg CaCO₃/l).

⁷ This substance is not a priority substance but one of the other pollutants for which the EQS are identical to those laid down in the legislation that applied prior to 13 January 2009.

⁸ DDT total comprises the sum of the isomers 1,1,1-trichloro-2,2 bis (p-chlorophenyl) ethane (CAS number 50-29-3; EU number 200-024-3); 1,1,1-trichloro-2 (o-chlorophenyl)-2-(p-chlorophenyl) ethane (CAS number 789-02-6; EU number 212-332-5); 1,1-dichloro-2,2 bis (p-chlorophenyl) ethylene (CAS number 72-55-9; EU number 200-784-6); and 1,1-dichloro-2,2 bis (p-chlorophenyl) ethane (CAS number 72-54-8; EU number 200-783-0).

⁹ If Member States do not apply EQS for biota they shall introduce stricter EQS for water in order to achieve the same level of protection as the EQS for biota set out in Article 3(2) of this Directive. They shall notify the Commission and other Member States, through the Committee referred to in Article 21 of Directive 2000/60/EC, of the reasons and basis for using this approach, the alternative EQS for water established, including the data and the methodology by which the alternative EQS were derived, and the categories of surface water to which they would apply.

¹⁰ For the group of priority substances of polyaromatic hydrocarbons (PAH) (No 28), each individual EQS is applicable, i.e. the EQS for benzo(a)pyrene, the EQS for the sum of benzo(b)fluoranthene and benzo(k)fluoranthene and the EQS for the sum of benzo(g,h,i)perylene and indeno(1,2,3-cd)pyrene must be met.

Appendix I: Waste water management regulation

Table 1. General requirements for waste water discharged to the environment

Parameter	Unit	Limit value
Maximum temperature	°C	Not higher than 30
pH ¹	-	6,5–8,5
Mineralization	g/l	Not higher than 2
Waste water should not be ²		

¹ Instantaneous pH values 4,0-6,4 ir 8,4-10,0 are allowable if their duration in an hour period does not exceed 6 min. (10% of time).

² Assessment of acute toxicity for *Daphia magna* (LAND 45-2001, Lithuanian Minister of Environment Order No. 4, 3 January 2002).

Table 2. The contamination limits of waste water discharged to the environment

Parameter	Size of agglomeration (quantity of discharged waste water/ contamination source)	Unit	MAC of average 24-h sample ¹ (the highest level of cleaning)	Instantaneous MAC value (the highest level of cleaning)	An annual average MAC value (the highest level of cleaning)	Minimum efficiency of cleaning, %	
BOD ₅ /BOD ₇ (without nitrification)	<5 m ³ /d	mgO ₂ /l	-	35/40	25/29	-	
	>5 m ³ /d	<2000 GE	mgO ₂ /l	-	30/34(15/17)	20/23 (10/12)	-
		2000–10000 GE	mgO ₂ /l	25/29 (10/12)	-	Defined individually	70–90
		>10000 GE	mgO ₂ /l	15/17 (8/10)	-	Defined individually	70–90
COD	>2000 GE	mgO ₂ /l	125	-	-	75	
Total phosphorous	>5 m ³ /d	<10000 GE	mgP/l	-	-	2	80
		10000–100000 GE	mgP/l	-	-	2 (1)	
		>100000 GE	mgP/l	-	-	1 (0,5)	
Total nitrogen	>5 m ³ /d	<10000 GE	mgN/l	-	-	20	70–80
		10000–100000 GE	mgN/l	-	-	15 (10)	
		>100000 GE	mgN/l	-	-	10 (10)	

¹ Concentration in average 24-h sample. ² Inhabitant equivalent (GE).

Table 3. General requirements for industrial waste water discharged to sewage system

Parameter	Unit	Limit value
Maximum temperature	°C	45
pH ¹	-	6,5–9,5 ²
COD/BOD ₇ ratio	-	<3
BOD ₇	mg/l	800 ²

¹ Instantaneous pH values 4,0-6,4 ir 8,4-10,0 are allowable, if their duration in an hour period does not exceed 6 min. (10% of time).

² Concentration in average 24-h sample.

Annex 1. Maximum allowable concentration (MAC) of priority hazardous substances in waste water and environmental quality standards (EQS)

Unit [$\mu\text{g/l}$]

Name of substance	CAS number ¹	MAC to sewage system	MAC to the environment	AA-EQS ²		MAC-EQS ³	
				Inland surface waters ⁴	Other surface waters	Inland surface waters ⁴	Other surface waters
Mercury and its compounds	7439-97-6	10	2	0.05	0.05	0.07	0.07
Cadmium and its compounds (depending on water hardness classes) ⁵	7440-43-9	100	40	≤ 0.08 (class 1) 0.08 (class 2) 0.09 (class 3) 0.15 (class 4) 0.25 (class 5)	0.2	≤ 0.45 (class 1) 0.45(class 2) 0.6 (class 3) 0.9 (class 4) 1.5 (class 5)	≤ 0.45 (class 1) 0.45(class 2) 0.6 (class 3) 0.9 (class 4) 1.5 (class 5)
Brominated diphenylether Pentabromodiphenylether ⁶	32534-81-9	-	-	0.0005	0.0002	-	-
Hexachlorocyclohexane (HCH)	608-73-1	40	2	0.02	0.002	0.04	0.02
Hexachlorobenzene (HCB)	118-74-1	12	0.6	0.01	0.01	0.05	0.05
Hexachlorobutadiene (HCBd)	87-68-3	40	2	0.1	0.1	0.6	0.6
Tributyltin compounds (Tributyltin-cation)	36643-28-4	0.4	0.02	0.0002	0.0002	0.0015	0.0015
Polyaromatic hydrocarbons (PAH) ⁷							
Benzo(a)pyrene	50-32-8	20	1	0.05	0.05	0.1	0.1
Benzo(b)fluoroanthene	205-99-2	16	0.8	$\Sigma=0.03$	$\Sigma=0.03$	-	-
Benzo(k)fluoroanthene	207-08-9	16	0.8				
Benzo(g,h,i)perylene	191-24-2	12	0.6	$\Sigma=0.002$	$\Sigma=0.002$	-	-
Indeno(1,2,3-cd)pyrene	193-39-5	16	0.8				
Nonylphenols (4-nonylphenol)	25154-52-3 (104-40-5)	400	20	0.3	0.3	2.0	2.0
C10-C13 chloroalkanes	85535-84-8	40	2	0.4	0.4	1.4	1.4
Anthracene	120-12-7	4	0.2	0.1	0.1	0.4	0.4
Endosulfan	115-29-7	-	-	0.005	0.0005	0.01	0.004
Pentachlorobenzene	608-93-5	12	0.6	0.007	0.0007	-	-

¹ CAS - chemical abstract service.

² This parameter is the EQS expressed as an annual average value (AA-EQS). Unless otherwise specified, it applies to the total concentration of all isomers.

³ This parameter is the EQS expressed as a maximum allowable concentration (MAC-EQS). Where the MAC-EQS are marked as „not applicable“, the AA-EQS values are considered protective against short-term pollution peaks in continuous discharges since they are significantly lower than the values derived on the basis of acute toxicity.

⁴ Surface waters encompass rivers and lakes and related artificial or heavily modified water bodies.

⁵ For cadmium and its compounds (No 6) the EQS values vary depending on the hardness of the water as specified in five class categories (Class 1: <40 mg CaCO₃/l, Class 2: 40 to <50 mg CaCO₃/l, Class 3: 50 to <100 mg CaCO₃/l, Class 4: 100 to <200 mg CaCO₃/l, and Class 5: ≥200 mg CaCO₃/l).

⁶ For the group of priority substances covered by brominated diphenylethers listed in Decision No 2455/2001/EC, an EQS is established only for congener numbers 28, 47, 99, 100, 153 and 154.

⁷ For the group of priority substances of polyaromatic hydrocarbons (PAH), each individual EQS is applicable, i.e. the EQS for benzo(a)pyrene, the EQS for the sum of benzo(b)fluoranthene and benzo(k)fluoranthene and the EQS for the sum of benzo(g,h,i)perylene and indeno(1,2,3-cd)pyrene must be met.

Annex 2. Maximum allowable concentration (MAC) of hazardous substances

Part A. Maximum allowable concentration (MAC) of priority substances and other pollutants in waster water and environmental quality standards (EQS).

Part A lists the priority substances (except priority hazardous substances listed in Annex 1 of Waste water management regulation) and other pollutants.

Unit: [µg/l]

Name of substance	CAS number ¹	MAC to sewage system	MAC to the environment	AA-EQS ³		MAC-EQS ⁴		Limit concentration to sewage system ²	Limit concentration to the environment ²
				Inland surface waters ⁵	Other surface waters	Inland surface waters ⁵	Other surface waters		
Alachlor ⁸	15972-60-8	-	-	0.3	0.3	0.7	0.7	-	-
Atrazine ⁸	1912-24-9	-	-	0.6	0.6	2.0	2.0	-	-
Benzene	71-43-2	800	40	10	8	50	50	160	8
Tetrachloromethane (CCl ₄) ⁶	56-23-5	1500	240	12	12	-	-	300	48
Chlorfenvinphos ⁸	470-90-6	-	-	0.1	0.1	0.3	0.3	-	-
Chlorpyrifos ⁸	2921-88-2	-	-	0.03	0.03	0.1	0.1	-	-
Cyclodiene pesticides ⁸ : Aldrin ⁶ Dieldrin ⁶ Endrin ⁶ Isodrin ⁶	309-00-2 60-57-1 72-20-8 465-73-6	-	-	Σ=0.01	Σ=0.005	-	-	-	-
DDT ^{6 7}	-	-	-	0.025	0.025	-	-	-	-
Para-para-DDT ⁶	CAS 50-29-3	-	-	0.01	0.01	-	-	-	-
1,2-dichloroethane (EDC)	107-06-2	200	200	10	10	-	-	40	40
Methylene chloride (dichloromethane)	75-09-2	4000	200	20	20	-	-	800	40
Di(2-ethylhexyl)phtalate	117-81-7	40	2	1.3	1.3	-	-	8	0.4
Diuron ⁸	330-54-1	-	-	0.2	0.2	1.8	1.8	-	-
Fluoroanthenene	206-44-0	120	6	0.1	0.1	1	1	24	1.2
Isoproturon ⁸	34123-59-6	-	-	0.3	0.3	1.0	1.0	-	-
Lead and its compounds	7439-92-1	500	100	7.2	7.2	-	-	100	20

Name of substance	CAS number ¹	MAC to sewage system	MAC to the environment	AA-EQS ³		MAC-EQS ⁴		Limit concentration to sewage system ²	Limit concentration to the environment ²
				Inland surface waters ⁵	Other surface waters	Inland surface waters ⁵	Other surface waters		
Naphtalene	91-20-3	400	20	2.4	1.2	-	-	80	4
Nickel and its compounds	7440-02-0	500	200	20	20	-	-	100	40
Octylphenol ((4-(1,1',3,3'-tetramethylbutyl)phenol)	140-66-9	400	20	0.1	0.01	-	-	80	4
Pentachlorophenol (PCP)	87-86-5	800	40	0.4	0.4	1	1	160	8
Simazine ⁸	122-34-9	-	-	1	1	4	4	-	-
Tetrachloroethylene ⁶	127-18-4	-	200	10	10	-	-	-	40
Trichloroethylene ⁶	79-01-6	-	200	10	10	-	-	-	40
Trichlorobenzene	12002-48-1	100	8	0.4	0.4	-	-	20	1.6
Trichloromethane	67-66-3	1000	200	2.5	2.5	-	-	200	40
Trifluralin	1582-09-8	40	2	0.03	0.03	-	-	8	0.4

¹ CAS: chemical abstract service.

² Limit concentration – maximum limit concentration is calculated, measured or planned concentration of substance, till which the control of substances is not needed yet.

³ This parameter is the EQS expressed as an annual average value (AA-EQS). Unless otherwise specified, it applies to the total concentration of all isomers.

⁴ This parameter is the EQS expressed as a maximum allowable concentration (MAC-EQS). Where the MAC-EQS are marked as „not applicable“, the AA-EQS values are considered protective against short-term pollution peaks in continuous discharges since they are significantly lower than the values derived on the basis of acute toxicity.

⁵ Surface waters encompass rivers and lakes and related artificial or heavily modified water bodies.

⁶ This substance is not a priority substance but one of the other pollutants for which the EQS are identical to those laid down in the legislation that applied prior to 13 January 2009.

⁷ DDT total comprises the sum of the isomers 1,1,1-trichloro-2,2 bis (p-chlorophenyl) ethane (CAS number 50-29-3; EU number 200-024-3); 1,1,1-trichloro-2 (o-chlorophenyl)-2-(p-chlorophenyl) ethane (CAS number 789-02-6; EU number 212-332-5); 1,1-dichloro-2,2 bis (p-chlorophenyl) ethylene (CAS number 72-55-9; EU number 200-784-6); and 1,1-dichloro-2,2 bis (p-chlorophenyl) ethane (CAS number 72-54-8; EU number 200-783-0).

⁸ Pesticides usually reach the environment from diffuse sources of pollution; therefore the limits are not specified.

Part B. Maximum allowable concentration (MAC) of other controlled substances.

Unit [mg/l]

Name of substances group	Name of substance	CAS number ¹	MAC to sewage system	MAC to the environment	MAC-EQS	Limit concentration to sewage system ²	Limit concentration to the environment ²
List B1							
Metals	Chromium (total)	7440-47-3	2	0.5	0.01	0.4	0.1
	Chromium (VI)		0.2	0.1	0.001	0.04	0.04
	Copper	7440-50-8	2	0.5	0.01	0.4	0.1
	Tin	2406-52-2	5	1	-	1	0.4
	Zinc	7440-66-6	3	0.4	0.1	0.6	0.16
	Vanadium	7440-62-2	10	2	-	2	0.8
	Aluminium	7429-90-5	2	0.5	-	0.4	0.2
	Arsenic	7440-38-2	0.15	0.05	-	0.03	0.02
Other substances	Oil hydrocarbons (total)		25	5	0.2	5	1
	Phenols		3	0.2	0.001	0.6	0.08
	Monochloroacetic acid	79-11-8	-	-	-	-	-
	3,4-dichloroaniline	95-76-1	-	-	-	-	-
	Dibutylphtalate	84-74-2	-	-	-	-	-
	Ethylenediaminetetra-acetic acid	60-00-4	-	-	-	-	-
	Ethylenediaminetetra-acetic acid disodium salt	64-02-8	-	-	-	-	-
	Sulphides (mineral) ³		2	0.5	-	0.4	0.2
	Chlorine (active)		0.6	0.1	-	0.12	0.04
Cyanides		0.5	0.1	-	0.1	0.04	
List B2							
Other substances	Total nitrogene		100	30	*	50	12
	Nitrites (NO ₂ -N)/NO ₂		-	0.45/1.5	*	-	0.09/0.3
	Nitrates (NO ₃ -N)/NO ₃		-	23/100	*	-	9/39
	Amonium (NH ₄ -N)/NH ₄		-	5/6.43	*	-	2/2.57
	Total phosphorous		20	4	*	10	1.6
	Phosphates (PO ₄ -P)/PO ₄		-	-	*	-	-
	Chlorides		2000	1000	300	1000	500
	Fluorides		10	8	-	2	3.2
Sulphates		1000	300	100	300	200	

Name of substances group	Name of substance	CAS number ¹	MAC to sewage system	MAC to the environment	MAC-EQS	Limit concentration to sewage system ²	Limit concentration to the environment ²
List B1							
Metals	Chromium (total)	7440-47-3	2	0.5	0.01	0.4	0.1
	Chromium (VI)		0.2	0.1	0.001	0.04	0.04
	Copper	7440-50-8	2	0.5	0.01	0.4	0.1
	Tin	2406-52-2	5	1	-	1	0.4
	Anion surfactants		10	1.5	-	2	0.6
	Non-ionic surfactants		15	2	-	3	0.8
	Fat		100	10	-	50	5

¹ CAS: chemical abstract service.

² Limit concentration – maximum limit concentration is calculated, measured or planned concentration of substance, till which the control of substances is not needed yet.

³ Preliminary values applied after the methods of determination of mineral sulphides.

* The average annual values of these materials in surface water (according to the distribution of ecological condition classes) are specified in the procedure of determination surface water status approved by the Lithuanian Minister of Environment in 2010 4 March Order No. D1-178 (Žin., 2010, Nr. 29-1363).

This is the report of Lithuanian results of COHIBA Work Package 3 work. Goals in this WP 3 were to identify sources of hazardous substances, for which there is scarce information available. Also the participating countries were to perform a toxicity survey in the case studies by using the Whole Effluent Assessment (WEA) method.



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