Investigation of hazardous chemical discharges from two textile-manufacturing facilities in China

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Executive Summary

The manufacture of textile products is a global industry that employs a long and complex industrial manufacturing chain. A diverse range of techniques and often chemically intensive processes are used to produce the finished products. Some of the process and finishing chemicals used are known to have intrinsic hazardous properties, making their release to the environment of particular concern. Many others have never been properly tested in terms of their toxicology or other potential impacts should they be released to the environment.

Despite the quantities and range of chemicals used in a sector of this size and diversity, relatively few studies have investigated in significant depth the compositions of chemical mixtures in wastes produced by textile-manufacturing facilities, including those within wastewaters discharged to surface waters.

This study was undertaken to investigate the discharge of industrial chemicals from two textile-manufacturing facilities (located on the deltas of the Yangtze and Pearl rivers respectively), as part of a broader investigation of chemical discharges from textile-manufacturing facilities within China. The study therefore builds on previous work published in 2009, which investigated discharges from certain industrial facilities within the Pearl River Delta, including a textile-manufacturing facility.

The two facilities addressed in the current study were:

- Youngor Textile Complex, which discharges wastewaters to the Yangtze River Delta.
- Well Dyeing facility, which discharges wastewaters to the Pearl River Delta

It is not intended that these facilities should be taken as representative of this sector as a whole, but merely as two specific examples of such facilities in the region.

The Yangtze River Delta and Pearl River Delta are highly industrialised areas, both having undergone rapid development in recent decades. Previous studies have demonstrated contamination of these river systems with a range of hazardous chemicals, many of which are likely to be of industrial origin.

A total of 20 samples were collected in June 2010 (10 from the vicinity of each site). These included samples of water discharged from all identifiable and accessible discharge points located close to the perimeter of each site, as well as sediment samples from the river channels into which these waters were being directly released. The origins of the waste material in some of the samples collected in the vicinity of the Well Dyeing facility could not be verified. All samples were returned to the Greenpeace Research Laboratories (University of Exeter, UK) for analysis. The Youngor Textile Complex was revisited in March 2011 and additional wastewater samples were collected from the wastewater treatment plant discharge pipe. These samples were analysed at OMEGAM Laboratoria in the Netherlands.

Wastewaters discharged from both sites contained a diverse range of chemicals, some with known hazardous properties. Some similarities were found in the chemicals identified in wastewaters discharged from the two sites, as might be expected for facilities involved in related activities.

Key findings from this study can be summarised as follows:

Youngor Textile Complex (Yangtze River Delta).

Substances identified in wastewater samples included:

- Nonylphenol, a well known persistent environmental contaminant with hormone disrupting properties;
- Persistent and toxic perfluorinated compounds, including PFOS, PFOA and other perfluorinated carboxylic acids;
- Amine compounds, which may have arisen from partial breakdown of azo-dyes, including the carcinogenic form o-anisidine,
- Tributyl phosphate, a hazardous chemical used in the textile industry;
- Amino-anthraquinone, a toxic chemical used in dye manufacturing;
- Tris(2-ethylhexyl) ester of phosphoric acid (trioctyl phosphate), an irritant chemical used as an additive in adhesives and as a plasticiser in certain PVC formulations;
- Traces of toluene, chlorinated solvents, chlorophenols and naphthalene; and
- Other substances for which little information is available on their toxicity, including quinoline-related compounds that are associated with the use of dyes.

Concentrations of nonylphenol in the single sample of effluent collected at 11:00 on 8 March 2011 (14 µg/l) and of PFOA in all three samples collected on 8 and 9 March (0.13-0.14 µg/l, or 130-140 ng/l) were above the ranges most commonly reported for surface waters in Europe, United States and parts of Asia (including China), other than in waters immediately downstream from major centres of industrial activity, including metal-working and textile manufacture and processing. Concentrations of both these substances were, however, within the wide range reported for effluents discharged from wastewater treatment plants in many parts of the world, especially those receiving a high proportion of industrial wastewaters via the sewers.

Well Dyeing (Pearl River Delta)

Substances identified in wastewater samples included:

- Nonylphenol and octylphenol, well known persistent environmental contaminants that are hormone disrupting chemicals;
- Trialkyl phosphates, including tributyl phosphate, a hazardous chemical used in the textile industry;
- Dichloroaniline, a dye related chemical that is toxic to wide range of aquatic organisms; and
- A complex mixture of other substances including some previously reported to be environmental contaminants, and others for which little toxicity information is available.

This study has therefore demonstrated that hazardous substances were, at the time of sampling, being discharged within wastewaters from both the Youngor Textile Complex and the Well Dyeing facility, to the receiving freshwater environments of the Yangtze River basin or Pearl River basin, respectively. In addition, many other substances were identified in the discharged wastewaters about which little is known in terms of their toxicology or potential impacts following release to the environment. Furthermore, the majority of chemicals isolated from some wastewater samples could not be reliably identified, and therefore their properties and potential impacts, whether alone or as components of complex mixture of contaminants, simply cannot be assessed.

These two sites provide an indication of the types of chemicals currently being used and released by parts of the textile-manufacturing sector and, while it cannot be assumed that these are representative of the sector as a whole, they nonetheless illustrate the nature of what is likely to be a much wider problem within China and elsewhere.

This study also highlights instances in which wastewater was discharged sporadically from an outfall and where the composition of wastewater from an outfall varied considerably at different times. These situations raise additional concerns regarding the monitoring of hazardous chemical discharges from industrial facilities.

Some of the substances identified are known to be highly persistent within aquatic environments, and able to bioaccumulate within organisms. In fact, Greenpeace has also recently found some chemicals identified in wastewaters discharged from these two sites (alkylphenols and some perfluorinated chemicals) in tissues of wild fish collected from the Yangtze River (Brigden et al. 2010b). Ongoing releases of such substances are likely to lead to ever-increasing levels in the receiving environment, which in some cases will not significantly decrease for long periods of time, even after controls on their release have been introduced. For many hazardous substances, the use of ‘end-of-pipe’ measures, including through the use of conventional wastewater treatment plants, cannot effectively address their presence in waste streams, as the substances either pass through in treated effluents or accumulate in sludges which can subsequently act as diffuse sources to the environment when disposed of.

Regulations addressing wastewater discharges from industrial facilities in China, including the two facilities investigated in this study, set maximum permissible levels for a limited number of chemical pollutants. In most cases, however, regulations do not prohibit their use. The majority of substances identified in the wastewater samples analysed for this study are not regulated within China, with regard to their use or discharge.

Differences exist between regulations dealing with the use and release of industrial chemicals in different countries, particularly for hazardous chemicals. Some hazardous substances that are not subject to such regulations within China are specifically listed as priority substances in one or more regulations or conventions developed to protect the environment and/or human health in other regions. This includes some of the hazardous substances identified in this study.

There is an urgent need to address the concerns related to the use and release of hazardous chemicals that this study has raised. Solutions are required which seek alternatives to the use of hazardous substances in manufacturing processes, progressively replacing them with less hazardous, and preferably non-hazardous, alternatives in order to bring about rapid reductions and ultimate cessation in their discharges, emissions and losses. Systematic rethinking and redesigning of products and processes to enable these changes can play a part in building a more sustainable industry, and in eliminating both the waste of resources and the pervasive threats to the environment and human health that result from the ongoing use and release of hazardous chemicals.

1. Introduction

The textile industry has one of the longest and most complicated industrial chains in the manufacturing industry. The production of a finished fabric commonly employs a diverse range of techniques and processes, many of which make use of numerous chemicals (IPPC 2003).

Despite the size and diversity of this sector, and the range of chemicals used in the various manufacturing processes employed, relatively few studies have investigated the presence of hazardous chemicals in wastes produced by textile manufacturing facilities, including chemicals within wastewaters discharged to surface waters. Furthermore, those studies which have been carried out have tended to focus on a limited number of specific chemicals or groups of chemicals, rather than attempt to investigate as far as possible the compositions of complex chemical mixtures that can be characteristic of industrial effluents (IPPC 2003, Lacasse & Baumann 2004).

Many of the man-made chemicals used to manufacture textiles are known to have intrinsic hazardous properties, making their release to the environment of particular concern. Many others have never been properly tested in terms of their toxicology or other potential impacts should they be released to the environment. Properties of hazardous chemicals include being persistent (do not readily breakdown in the environment), bio-accumulative (able to accumulate in organisms), and toxic, including carcinogenic (chemicals which can cause cancer), mutagenic (chemicals with capacity to induce mutations and gene-defects), toxic to reproduction (chemicals which can harm the reproductive system) or to the nervous system, or capable of disrupting endocrine (hormone) systems.

This study was undertaken to investigate the presence of hazardous chemicals in wastewaters discharged from two textile-manufacturing facilities in China, to provide an indication of the types of chemicals currently being used and released by parts of the textile-manufacturing sector in China. The investigation of these two facilities forms part of a broader investigation of the chemical composition of discharged wastewaters from nine textile-manufacturing facilities within China, the further results of which will be published at a later date.

These facilities should therefore be seen as specific case examples of contemporary effluent discharges. However, while providing a useful indication of what might be occurring at other textile facilities, it is not intended that these two facilities, nor the additional seven that form part of the broader study, should be taken as representative of this sector in China as a whole.

This study was carried out to provide data on hazardous chemical pollutants being directly discharged from individual industrial facilities, and specifically textile manufacturers, to the river systems of the Yangtze River Delta and Pearl River Delta. One of the facilities, the Youngor Textile Complex, is located on, and discharges wastewaters to, the Yangtze River Delta. The second, the Well Dyeing facility, is located on, and discharges wastewaters to, the Pearl River Delta. More information on the two facilities investigated is provided in the Section 4.

The Yangtze River, also known as the Chang Jiang (‘Long River’), is the longest river in China, with the Pearl River being the third longest. The two river delta areas are highly industrialised, having undergone rapid development in recent decades, with both now housing a wide range of industrial activities. The Pearl River Delta accounted for about 10% of the total Gross Domestic Product (GDP) of China in 2009, while the Yangtze River Delta accounted for over 20% (Guangdong Province 2010, Zhejiang Province 2010).

For both deltas, previous studies have demonstrated contamination of the river systems with a range of hazardous chemicals, including persistent organic chemicals and heavy metals (for example, Fu et al. 2008, Müller et al. 2008, Wang et al. 2008, Fu et al. 2003, Chau 2006). Many of the chemicals identified in these studies are amongst the very large number of chemicals that are likely to be used and released by industry within these river basins. Others, including several persistent organic pollutants, are no longer manufactured or used in the region, but their continued presence highlights the long-term consequences of the use and release of persistent chemicals in general.

Previous investigations related to hazardous chemical inputs to the Yangtze and Pearl River basins have tended to focus on the presence and levels of certain pollutants found within the river systems. While providing useful data, such an approach has limitations due to its inability to identify individual point sources. A previous report published by Greenpeace in 2009 took an alternative approach by demonstrating the direct discharge of hazardous chemicals within industrial wastewaters from industries located within the Pearl River Delta, including from a textile manufacturing facility (Brigden et al. 2009). The current study extends the scope of this work, providing additional case studies, both within the Pearl River Delta and also within the Yangtze River Delta.

2. Sampling programme

Samples for this study were collected in the vicinity of two textile-manufacturing facilities; the Youngor Textile Complex in Ningbo within the Yangtze River Delta, and the Well Dyeing facility in Zhongshan City within the Pearl River Delta. The two sites were visited in June 2010, and samples of discharged wastewater, other water discharges, and river sediments were collected. At each site, samples of water were collected from each of the outfalls that could be identified and accessed close to the perimeter of each site. In addition, samples of sediment were collected from rivers and canals into which wastewaters were being discharged, adjacent to the outfalls.

Youngor Textile Complex was revisited in March 2011 and additional samples of discharged wastewater were collected from the wastewater treatment plant discharge pipe on three separate occasions over a period of 15 hours in order to allow more detailed and up to date quantitative analysis for a range of target chemicals. Similar re-sampling was, unfortunately, not possible at the Well Dyeing facility.

Details of all samples collected are presented in Section 4 (Results and discussion) below.

For those samples collected in June 2010, all were collected from the locations set out in Tables 1 and 5, and stored in pre-cleaned glass bottles that had been rinsed thoroughly with nitric acid and analytical grade pentane in order to remove all heavy metal and organic residues. Wastewater samples were collected in 0.5 litre screw-cap bottle for use in the quantitative analysis of metals and qualitative analysis of solvent extractable (semi-volatile) organic compounds. A duplicate sample was collected in a separate 125 ml amber bottle with a ground-glass stopper (filled to leave no headspace) to be analysed for volatile organic chemicals. Sediment samples were collected in 100ml screw-cap bottles. All samples were immediately chilled and kept cool and dark during transit to the Greenpeace Research Laboratories at the University of Exeter in the UK for analysis.

For samples collected from the Youngor Textile Complex in March 2011, three separate sets of bottles were used, as prepared and supplied according to the standard procedures employed by the independent laboratory in the Netherlands commissioned by Greenpeace to carry out the more detailed quantitative analyses. These sets of bottles were filled at three discrete times with effluent from one of the pipelines previously identified and sampled in June 2010 (at 1100 on 8 March, 2100 on 8 March and 0200 on 9 March 2011).

All samples were immediately chilled and kept cool and dark during transit to OMEGAM Laboratoria, at which they were received on 11 March 2011.

3. Methodology

For those samples analysed at the Greenpeace Research Laboratories, a number of different analyses were carried out on the wastewater and sediment samples. Heavy metal concentrations were determined for all samples by ICP atomic emission spectrometry (AES), following acid digestion and using appropriate certified reference materials in addition to intra-laboratory standards. All water samples contained suspended solids and therefore both the total concentrations in the whole (unfiltered) sample and the concentrations of dissolved forms in a filtered sample were determined separately.

Extractable organic compounds were isolated from each sample and identified as far as possible using gas chromatography and mass spectrometry (GC/MS), following accelerated solvent extraction into a mixture of pentane and acetone for solid samples or solid phase extraction with ethyl acetate, pentane and toluene for wastewater samples. Volatile organic chemicals (VOCs) were identified and quantified in wastewater samples as received (with no pre-treatment) using GC/MS with HeadSpace sample introduction technique. Detailed description of the sample preparation and analytical procedures are presented in the Appendix 1, including a list of VOCs quantified in the water samples, with limits of detection and quantification (Table 7).

For those samples collected in March 2011 and analysed at OMEGAM Laboratoria, a range of substance-specific or group-specific quantitative analytical methods were applied, as summarised below:-

- Metals (cadmium, lead, mercury and chromium (VI)) – ICP-mass spectrometry (ICP-MS), conforming to NEN-EN-ISO 17294-2 (cadmium & lead) or NEN-EN 1483 (mercury);
- Phenols, chlorinated phenols and alkylphenols, chlorinated benzenes, amines, brominated flame retardants, phthalate esters, organotin compounds – gas chromatography/mass spectrometry (GC/MS), with chlorophenol analysis conforming to NEN-EN 12673;
- Perfluorinated chemicals – GC/MS using triplequad detector; and
- Chlorinated aliphatic solvents and non-chlorinated volatiles (BTEX) – GC/MS with ‘purge and trap’ sample preparation, conforming to NEN-EN-ISO15680.

The analyses for metals, chlorinated and non-chlorinated volatiles, chlorinated phenols, amines and organotins were all conducted using accredited methods and certified accordingly. In addition, the effluents collected at these times were also subjected by the same independent laboratory to a qualitative 'screening' analysis for both volatile and non-volatile organic compounds using GC/MS techniques with the detector operated in scan mode. These analyses were similar to the screening methods applied in our laboratory in the case of the samples collected in June 2010 (see above), but employed different libraries of spectra and criteria for 'fingerprinting' chemicals than were applied in our laboratory.

A complete list of the substances included in the quantitative analyses conducted by the independent laboratory is provided in Appendix 2. Further details regarding the methods employed can be provided on request.

4. Results and discussion

The results are presented below in two sections, the first for samples associated with the Youngor Textile Complex, and the second section for samples associated with the Well Dyeing textile manufacturing facility. A summary of the details for samples collected from the two textile manufacturing sites are presented in Tables 1, 3 and 5, along with maps showing the locations from which samples were collected (Figures 1 and 3). Background information on certain key pollutants detected during this study is presented in Boxes A-F through the discussion text.

It should be noted that all metals quantified in this study are naturally found at some level in uncontaminated environmental samples, such as sediments and surface waters, though generally at low concentrations. Inputs from point sources such as industrial discharges can, however, result in levels that far exceed natural background concentrations. The following sections focus on those metals found in the various samples at concentrations that are above typical background levels for uncontaminated waters and, therefore, indicative of significant inputs from industrial or other anthropogenic sources. For those samples that contained concentrations of the quantified metals that were either below limits of detection for the methods used or within the ranges expected for uncontaminated surface waters, the data are not discussed.

4.1 Youngor Textile Complex (Yangtze River Delta)

The Youngor Textile Complex houses a number of individual facilities, including Youngor Sunrise Textile Dyeing & Finishing Co., Ltd., Ningbo Youngor Pants Co., Ltd., Ningbo Youngor Fashion Co., Ltd. and Ningbo Youngor Worsted Spinning, Weaving & Dyeing Co., Ltd., as well as a wastewater treatment plant (WWTP). This large industrial complex is situated in Ningbo, on the banks of the Fenghua River, occupying approximately 3km of the river bank. The Fenghua River, a tributary to the Yangtze River Delta, is tidal at this location. No other industrial facilities are located on the banks for the river in the vicinity of the Youngor Textile Complex.

The site was visited on two occasions during which various samples collected, including samples of water discharged from pipes in the vicinity of the complex and sediment from the adjacent river. Details of the samples that were collected along with the results from the analyses of these samples are presented in Tables 1 and 2 in Section 4.1.1 (samples collected in June 2010) and in Tables 3 and 4 in Section 4.1.2 (samples collected in March 2011).

4.1.1. Samples collected in June 2010

Four pipes were observed to discharge water into the Fenghua River in the vicinity of the Youngor Textile Complex. In June 2010, five samples of discharged water were collected from these pipes (pipe 1 was sampled on two consecutive days), along with four samples of river sediment from locations adjacent to these pipes. In addition, a sample of river sediment (CN10041) was collected from the Fenghua River, approximately 300m upstream of all discharge pipes.

Wastewater samples from the most southerly pipe (pipe 1), which is connected to the wastewater treatment plant (WWTP) of this complex, were collected on two separate occasions. One sample (CN10042) was collected on the evening (pm) of the 21st June 2010, when the flow of wastewater from the pipe was low. A second sample (CN10050) was collected on the following morning (am), at which time the rate of discharge was observed to be substantially greater. Local have reported that wastewaters of different colours are discharged via this pipe at different times. A sample of sediment (CN10043) was collected from the Fenghua River adjacent to pipe 1.

Samples were also collected from the three other discharge pipes, along with samples of sediment from the river adjacent to these three pipes. After the collection of samples from pipe 2, pipe 3 and pipe 4, it could not be confirmed that these pipes (pipe 2, pipe 3 or pipe 4) were discharge pipes exclusively for wastewaters generated within the Youngor Textile Complex.

Details of these samples are provided in Table 1, and their relative locations are shown in Figure 1. The data from the analyses of all samples associated with the Youngor Textile Complex that were collected in June 2010 are presented in Table 2.

Table 1. Description of samples associated with the Youngor Textile Complex, Yangtze River Delta, China, June 2010

Sample	Type	Description
CN10041	Sediment	Fenghua River, approx. 300m upstream of all discharge pipes
CN10042	Wastewater	Discharge pipe 1, from the Youngor WWTP; 21-06-10 (pm)
CN10050	Wastewater	Discharge pipe 1, from the Youngor WWTP; 22-06-10 (am)
CN10043	Sediment	Fenghua River, adjacent to pipe 1 (see CN10042 & 50)
CN10046	Wastewater	Discharge pipe 2
CN10047	Sediment	Fenghua River, adjacent to pipe 2 (see CN10046)
CN10044	Wastewater	Discharge pipe 3
CN10045	Sediment	Fenghua River, adjacent to pipe 3 (see CN10044)
CN10048	Wastewater	Discharge pipe 4
CN10049	Sediment	Fenghua River, adjacent to pipe 4 (see CN10048)

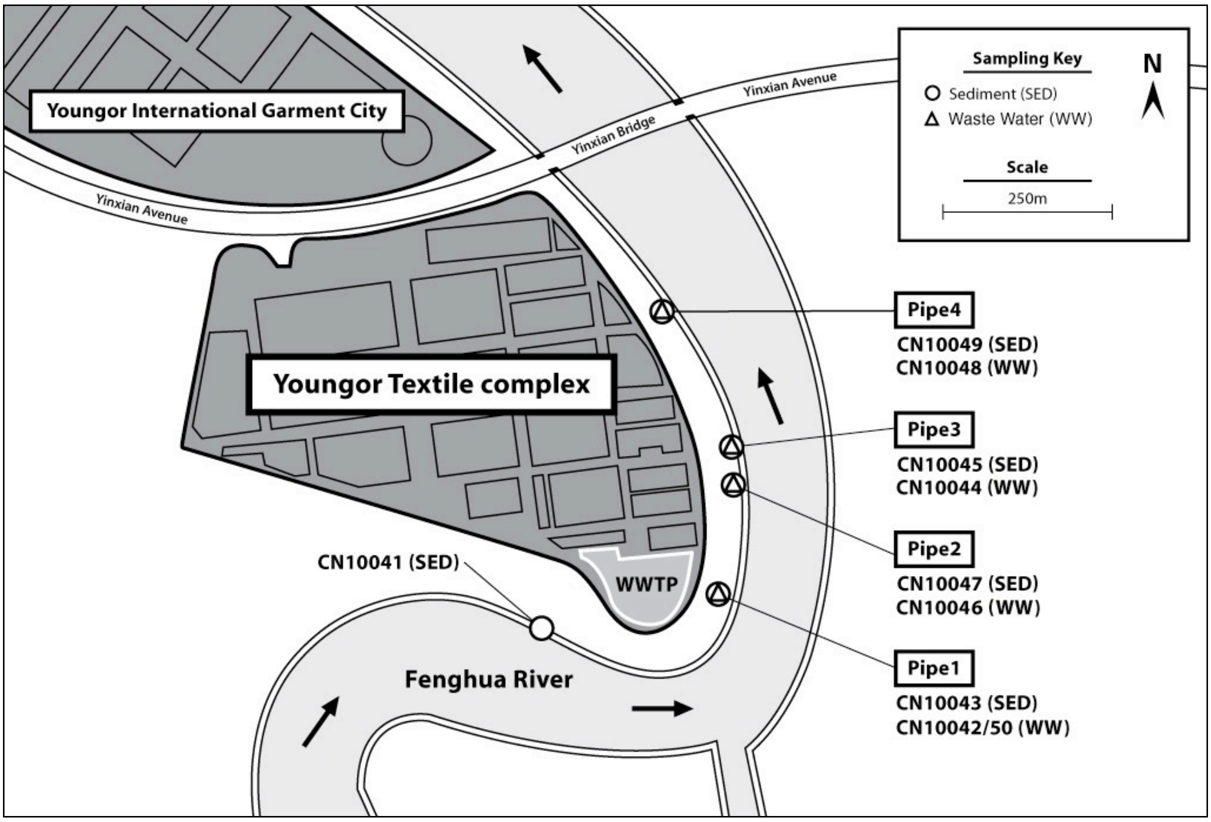


Figure 1. Sketch map of the Youngor Textile Complex showing the locations from which samples were collected.

WW - wastewater, SED - sediment

Location	River	Pipe 1					Pipe 2			Pipe 3			Pipe 4		
Sample	CN10041	CN10042 (pm)		CN10050 (am)		CN10043	CN10046		CN10047	CN10044		CN10045	CN10048		CN10049
Type	river sediment	wastewater		wastewater		river sediment	wastewater		river sediment	wastewater		river sediment	wastewater		river sediment
		total	diss	total	diss		total	diss		total	diss		total	diss	
METAL	(mg/kg)	(µg/l)	(µg/l)	(µg/l)	(µg/l)	(mg/kg)	(µg/l)	(µg/l)	(mg/kg)	(µg/l)	(µg/l)	(mg/kg)	(µg/l)	(µg/l)	(mg/kg)
Antimony	<20	<100	<100	<100	<100	<20	<100	<100	<20	<100	<100	<20	<100	<100	<20
Arsenic	<20	<50	<50	<50	<50	<20	<50	<50	<20	<50	<50	<20	<50	<50	<20
Barium	167	25	22	38	17	154	71	71	108	106	52	127	44	44	120
Cadmium	<1	<5	<5	<5	<5	<1	<5	<5	<1	<5	<5	<1	<5	<5	<1
Chromium	281	<20	<20	54	<20	792	<20	<20	209	53	<20	355	<20	<20	202
Chromium (VI)	-	-	<50	-	<50	-	-	<50	-	-	<50	-	-	<50	-
Cobalt	19	<20	<20	<20	<20	21	<20	<20	13	<20	<20	14	<20	<20	13
Copper	169	<20	<20	28	<20	253	<20	<20	103	21	<20	167	<20	<20	95
Lead	69	<50	<50	<50	<50	24	<50	<50	17	<50	<50	24	<50	<50	31
Manganese	644	143	106	229	143	742	29	<10	515	361	66	570	83	49	691
Mercury	<0.5	<2	<2	<2	<2	<0.5	<2	<2	<0.5	<2	<2	0.8	<2	<2	0.6
Nickel	112	<20	<20	<20	<20	191	<20	<20	74	<20	<20	91	<20	<20	73
Selenium	<30	<200	<200	<200	<200	<30	<200	<200	<30	<200	<200	<30	<200	<200	<30
Vanadium	62	<20	<20	<20	<20	54	<20	<20	37	54	<20	40	<20	<20	38
Zinc	276	33	<10	52	<10	284	28	<10	202	107	<10	277	29	<10	222
No. organic compounds isolated	29	53		9		46	5		27	1		30	30		25
No. identified (% of total)	19(66 %)	12 (23%)		4 (44 %)		21(46 %)	1 (20 %)		17(63 %)	0 (0 %)		19(63 %)	7 (23 %)		16(64 %)
Nonylphenol													6		
Phthalate (DEHP)							1								
Tributylphosphate (TBP)		1													
(Iso-)quinolines/quinolinones		5		1											
Amino-anthraquinone		1		1											
N-ethyl-toluenesulfonamide		3		2											
Decynediol derivative		1													
Chlorinated benzenes		1											(1)		
Di-tolyl ether									6			6			6
Fatty acid derivative						1						1			1
Alkyl benzene/aliphatic hydrocarbon	19					20			11			12			9

Table 2. Organic compounds identified, and concentrations of metals, in samples of discharged wastewaters and river sediment associated with the Youngor Textile Complex, China, 2010. For organic compounds, (..) signifies those identified at trace levels using a selective SIM method. Concentrations of metals in wastewater and canal water samples are reported in µg/l, both as total concentrations in the whole (unfiltered) sample and separately as (dis)solved concentrations in filtered samples. Concentrations of metals in sediments are reported as mg/kg dry weight.

Of all wastewater samples collected from the site on this occasion, the greatest number and widest range of organic chemicals were identified in one of the wastewater samples (CN10042) collected from pipe 1, which discharges wastewater from the wastewater treatment plant (WWTP) of this complex. 53 substances were isolated from this sample, though it was possible to only identify 12 of these. The substances identified included a trialkyl phosphate (tributyl phosphate, TBP), as well as quinoline and quinolinone derivatives, an anthraquinone (AQ) derivative, toluenesulfonamides, dichlorofluorobenzene, as well as a decynediol derivative. Far fewer organic chemicals were isolated from the second wastewater sample collected from this pipe the following morning (CN10050), when the rate of discharge was visibly greater. Nonetheless, of the four compounds that were identified, all were also present in the sample collected the previous evening (CN10042); namely amino-anthraquinone, a quinoline derivative, and two toluenesulfonamides. This situation highlights the potential for considerable variability in the composition of wastewaters discharged from single point sources over time.

Tributyl phosphate (TBP) is widely used in various industrial processes, including by the textile industry due to properties of being a strong wetting agent and strong polar solvent. Trialkyl phosphates, including TBP, have been frequently found in surface water environments (Andersen et al. 2004, Alink et al. 2005, Reemtsma et al. 2008, Regnery & Puttmann 2010). More information on trialkyl phosphates is presented in Box A.

Five **quinoline-related compounds** were identified, including dimethyl-quinolines, methyl-isoquinoline and methyl-quinolinone. Some of these dye related chemicals have been previously reported in wastewater from dyes and intermediary products industries (Yuan et al. 2006, Oliveira et al. 2007). Methyl-quinolinones are reported to be biodegradation metabolites of methyl quinolines (Wang et al. 2010). Little information is available on the toxicity of these substances, though bioassay studies have provided evidence that dimethylquinolines are moderately toxic in the aquatic environment (Birkholz et al. 1990).

The AQ derivative (**amino-anthraquinone**) is a common intermediate in the synthesis of a range of AQ dyes, many of which can degrade to release amino-anthraquinone (Novotny et al. 2006). Many AQ derivatives, including amino-anthraquinone, are known to be toxic to animals and/or plants (Sendelbach 1989). More information on AQ and its derivatives is given in Box B.

N-ethyl-toluenesulfonamides (NETSAs) are a group of three closely related chemicals. Commercial formulations of NETSAs, also known as Ketjenflex® 8 and Santicizer® 8, have a variety of industrial uses including as a plasticiser in printing inks, coatings & adhesives, nylons and plastics (Edgar et al. 2001, Alliance Chemicals 2009, Milne 2005). NETSAs have previously been detected in landfill leachates (Lei & Aoyama 2010, Agenson et al. 2003). There is limited information on the toxicity of this group of chemicals, though some transient effects on mammals following long-term exposure have been reported (VKM 2008), as well as an indication of toxicity using bioassay testing (Lei & Aoyama 2010).

The **decynediol derivative** (2,4,7,9-Tetramethyl-5-decyne-4,7-diol, also known as TMDD) is produced and used in large quantities, primarily as an industrial defoaming surfactant under the trade name Surfynol 104 (US EPA 2002, US EPA 2011). Other uses include the production of polyethylene glycol ether surfactants, in dye and printing inks formulations, and pesticide formulations (Musselman & Chander 2002, USDA 2009, HSDB 2002). TMDD has rarely been reported as an environmental contaminant, though one study reported its presence in river water in Germany, which was attributed to TMDD inputs via sewage effluents (Dsikowitzky et al. 2004a&b). TMDD has been found to be toxic to aquatic organisms in laboratory experiments on fish (fathead minnows and carp), aquatic invertebrates (*Daphnia magna*), and green algae (*Selenastrum capricornutum*) (US EPA 2002).

Dichlorofluorobenzene is used as an intermediate in organic syntheses, including the manufacture of certain antibiotics (Sekar & Prasad 2009). There is no known connection to the textile sector, and very little information is available on the toxicity of this chemical (Cole Parmer 2004).

For the water from pipe 2, only one compound was identified; the phthalate ester di(2-ethylhexyl) phthalate (DEHP). This toxic chemical is among the most ubiquitous man-made chemicals distributed within the environment, largely as a result of large-scale losses from products in which it had been incorporated, especially as a plasticiser in the plastic PVC (CSTEE 2001, Fromme et al. 2002). This is the most likely source of DEHP in this sample.

No organic chemicals were detected in the water sample discharged via pipe 3 (CN10044).

Two chemical groups were identified in the sample of water discharged via pipe 4 (CN10048); 6 isomers of nonylphenol, and 1,4-dichlorobenzene. The sources of these chemicals to this sample are not known.

Box A. Trialkyl phosphate esters

Tributyl phosphates (TBPs) may exist in two isomeric forms: tributyl phosphate (TBP) and tri-iso-butyl phosphate (TiBP). TBP is used as a solvent for cellulose esters, lacquers, and natural gums, as a primary plasticiser in the manufacture of plastics, textile and vinyl resins, as a metal extractant, as a base stock in the formulation of fire-resistant aircraft hydraulic fluids, as a flame retardant, and as an antifoaming agent. (Dodi & Verda 2001; Lamouroux et al. 2000, WHO 1991). TiBP is mainly used as an antifoaming agent in various aqueous systems where it has the ability to both destroy the foam and act as a foam inhibitor. It is also used in the production of solutions of synthetic resins and natural rubber, and as a flame-retarding plasticiser in both cellulose based plastics and synthetic resins. Other uses of TiBP include as a pasting agent for pigment pastes, as an important component in the manufacture of hydraulic fluids for aircraft, and as a very strong wetting agent and very strong polar solvent in the textile industry and in the field of adhesives.

TBP is continuously lost to the air and aquatic environment during many of its various uses. The biodegradation of TBP is moderate or slow depending on the ratio of TBP to active biomass. Degradation involves stepwise enzymatic hydrolysis to orthophosphate and butanol, which subsequently undergoes further degradation. Both TBP and TiBP can be photochemically degraded in aquatic environments (Regnery & Puttmann 2010). TiBP has been found frequently in air (Isetun et al. 2004), in surface water, especially near sewage treatment plants discharges (Andersen et al. 2004, Regnery & Puttmann 2010), in sediments (García-López et al. 2009), in fish and in liver samples from seabirds (Evenset et al. 2009). TiBP was recently reported in wastewaters discharged from textile manufacturing facilities, and receiving surface water in Thailand (Brigden et al. 2010b).

TBP toxicity has primarily been investigated for aquatic organisms and rodents (WHO 1991, Verbruggen et al. 2005). It has also been shown that TBP is toxic to selected protozoa species (Bowen et al. 2006). The inhibitory concentrations (EC0, EC50, EC100) of TBP for the multiplication of unicellular algae, protozoa, and bacteria have been estimated to lie within the range of 3.2-100 mg/litre (WHO 1991). The acute toxicity to fish (LC50) ranges from 4.2 to 11.8 mg/litre. Maximum permissible concentrations of 66 µg/l and 11 µg/l been derived for TBP and TiBP, respectively, in freshwaters to protect all species from adverse effects (Verbruggen et al. 2005). TBP increases the drying rate of plant leaves, which results in rapid and complete inhibition of leaf respiration. TBP is reported to be slightly teratogenic at high dose levels. Administration of TBP to male mice was associated with an increased incidence of benign liver tumours (hepatocellular adenomas) and an increase in liver weights for male and female mice (Auletta et al. 1998). Only limited data exist on the toxic effect of TBP on humans. In an *in vitro* study, TBP has been reported to have a slight inhibitory effect on human plasma cholinesterase (WHO 1991). Also, both TBP and TiBP are classified as skin irritants (Rudnick 2006).

Tris(2-butoxyethyl) phosphate (TBEP) has many uses, including as a plasticiser, an antifoam agent and an additive in resins and floor polish. Commercial TBEP formulation may contain traces of TBP and 2-butoxyethanol (WHO 2000). TBEP has been frequently found in different environmental media, particularly in surface waters and sediments, as well as in effluents from municipal wastewater treatment plants (Bohlen et al., 1989, ECETOC 1992, WHO 2000, Martínez-Carballo et al. 2007, Reemtsma et al. 2008, Regnery & Puttmann 2010). TBEP has also been detected in fish collected close to sewage treatment plants (Sundkvist et al. 2010). Once released to the aquatic environment, TBEP is likely to adsorb onto sediment particles, and tends to be readily biodegradable in surface water environments (Mead & Handley 1998, WHO 2000). Little or no information is available on breakdown products, though one potential breakdown product is 2-butoxyethanol, a toxic, though not persistent substance (ATSDR 1998, IPCS 1998). TBEP has been detected in human adipose tissue (Lebel & Williams 1986). For the general population, exposure is primarily via food and water due to the use of TBEP as a plasticiser in packaging and distribution materials (WHO 2000).

Animal experiments have shown that the liver is the main target organ for TBEP toxicity, though other effects have been reported including impacts on the nervous system. TBEP is moderately toxic to aquatic organisms, with LC50 values in fish ranging between 16 and 24 mg/litre (WHO 2000). A maximum permissible concentration of 13 µg/l been derived for TBEP in freshwaters to protect all species from adverse effects (Verbruggen et al. 2005).

Triethyl phosphate (TEP) is primarily used as an industrial catalyst, as a plasticiser and flame retardant, and to a lesser extent as a solvent and an intermediate in the manufacture of pesticides and other chemicals. TEP is water soluble, and it is reported to be released in effluents from some TEP manufacturing facilities, and has also been detected in river water in Europe (OECD 1998, Alink et al. 2005).

TEP is not readily biodegradable, but can be biodegraded in wastewater treatment plants using with industrial inoculums. TEP has a low bioaccumulation potential (OECD 1998), and is less toxic to aquatic organisms compared to other trialkyl phosphates such as TiBP and TBEP (Verbruggen et al. 2005). TEP shows moderate acute toxicity in mammals, exhibiting certain neurotoxic properties, though only low chronic toxicity (OECD 1998). Little is known about health effects on humans.

Box B. Quinone derivatives and di-ketone derivatives

Anthraquinone (AQ) and its derivatives are a group of chemicals closely related to the carcinogenic PAH anthracene, which have both natural and man-made sources. Synthetic AQs are widely used in dyeing operations (second in bulk only to azo dyes, Baughman & Weber 1994), most commonly for cotton, cellulose-based fibres and some synthetic fabrics (Novotny et al. 2006). They have also been used as colourants in hair dyes (Nohynek et al. 2004) and as a thermal stabiliser for rigid PVC plastic (Sabaa et al. 2006). Other derivatives of anthraquinone have uses as photo initiators (light-sensitive compounds used to induce polymerisation or to cure materials), as used in producing decorative coatings (Corrales et al. 2003). Some AQ compounds are known to occur naturally in higher plants and fungi (Tikkanen et al. 1983). AQs, including anthraquinone itself, can also formed as by-products of fossil fuel combustion (Tukaj & Aksmann 2007, Wei et al. 2010). Because of the nature of fibre-dyeing processes, the textile sector is thought to be a very significant point source of discharges of AQ compounds to water.

Many AQ derivatives are known to be toxic to animals and/or plants (see e.g. Sendelbach 1989 for a review of early evidence); indeed, their ability to cause oxidative damage to DNA in dividing cells has led to their use in very controlled doses as anti-tumour drugs, among other medical applications (Wei et al. 2010). Aminoanthroquinone, a common intermediate in the synthesis of a range of AQ dyes, has been shown to be carcinogenic in laboratory studies, as well as damaging to the kidneys (Krishna Murthy et al. 1979, Ramanathan et al. 1981). Anthraquinone itself can readily undergo photo-oxidation reactions in sunlight to form a range of more harmful AQ derivatives which are toxic to aquatic life, including planktonic algae at the base of the food chain. Photo-oxidation of aminoanthaquinone greatly enhances its toxicity to the water flea (*Daphnia magna*) (Wang et al. 2009). Under anaerobic conditions, such as may prevail in organic-rich sediments, some AQ dyes may be resistant to degradation as a result of inhibition of bacterial activity (Lee & Pavlostathis 2004), while others break down relatively quickly forming the more persistent derivative anthrone (Baughman & Weber 1994).

Benzoquinones. Derivatives of ortho- and para- benzoquinones are used as intermediates in processes involving photochemical reactions (van der Graaf et al. 1991). Very limited information is available on the toxicity and fate of these chemicals. There is some evidence for the toxicity of DBQ to mammalian cells from *in vitro* tests, in that congeners of p-benzoquinone, including 2,6-di-tert-butyl-p-benzoquinone (DBQ), have been found to express cytotoxicity in primary rat hepatocyte and PC12 cell cultures tests (Siraki et al. 2004). DBQ has been found among the semi-volatile chemicals emitted during heating of nitrogen-containing plastic (Watanabe et al. 2007).

Naphthalic anhydride is an important intermediate in the production of various dyestuffs, especially yellow/orange spectrum fluorescent dyes (Peters & Bide 1985, Xuhong et al. 1989) and optical brightening agents (Grabtchev & Philipova 1995) for synthetic polymer fibres. It has also been used extensively as a herbicide 'safener' (reducing the impacts on commercial crops of thiocarbamate and chloroacetamide herbicides) and as a fungicide in its own right (Abu-Qare & Duncan 2002). Despite its intended purpose in herbicide formulations, naphthalic anhydride can be toxic to plants at high doses (Boldt & Barrett 1991), though it is expected to degrade fairly rapidly to naphthalic acid in soils and water (Abu-Qare & Duncan 2002). The toxicity of naphthalic anhydride to animals has not been fully investigated to date, though it is capable of causing irritation to the eyes, skin, respiratory and digestive tracts in mammals following acute exposure (ACROS Organics 2009).

Methyl 2-benzoylbenzoate, a derivative of benzophenone, is known to be used as a photoinitiator in UV-curable inks (Müller et al. 2008). This chemical has been reported in sewage water treatment plant effluent in Sweden (Lilja et al. 2010). Little information is available on its toxicity. Acute toxicity to fish (48h LC_{50}, Oryzias latipes) has been reported at 20ppm (TCI, 2010) and, assuming the reliability of this figure, the compound would be classified by the EPA as slightly toxic (US EPA 2010). No information is available on subacute and long term effects of this chemical, or on the toxicity of this chemical to other species including humans. This chemical is not expected to bioaccumulate (TCI 2010).

Nonylphenol (NP) is a well known persistent environmental contaminant with hormone-disrupting properties for many aquatic organisms. NP has a range of industrial uses, and can also be formed by the breakdown of nonylphenol ethoxylates (NPEs), industrial detergent used in many industrial sectors, including in the manufacture of textiles. More information on these substances is presented in Box C. 1,4-dichlorobenzene is another well known environmental contaminant, largely due to its widespread use as a deodoriser and disinfectant in sanitary products. This substance was only identified using a sensitive SIM (selective ion monitoring) method, indicating its presence in this sample at only trace levels.

Neither the wastewater samples collected from pipe 1, nor the water samples collected from pipe 2, pipe 3 and pipe 4, were significantly contaminated with any of the quantified metals. Their concentrations were either below limits of detection for the methods used or within the ranges expected for uncontaminated surface waters. In some instances, the concentration in the whole (unfiltered) sample were somewhat higher than the respective dissolved concentration, indicating that a proportion of the metal present was bound to suspended particulates.

Box C. Alkylphenols and their ethoxylates

Nonylphenol (NP) and octylphenol (OP) are groups of related (isomeric) chemicals that are manufactured for a range of specialised industrial uses as well as to produce alkylphenol ethoxylates (APEs), a group of non-ionic surfactants. The most widely used APEs are nonylphenol ethoxylates (NPEs) and, to a lesser extent, octylphenol ethoxylates (OPEs). Once released to the environment, NPEs and OPEs can degrade back to NP and OP respectively, which are persistent, bioaccumulative and toxic to aquatic life. NPEs have been used as surfactants, emulsifiers, dispersants and/or wetting agents in a variety of industrial and consumer applications including the manufacture of textiles, the largest share in industrial and institutional cleaning products (detergents), with smaller amounts used as emulsifiers, textile and leather finishers and as components of pesticides and other agricultural products and water-based paints (OSPAR 2004, Guenther et al. 2002). OPEs are reported to have had a similar range of uses to NPEs, although fewer reliable data are available for this group. Within China, the production of APEs (primarily NPEs) was estimated to be about 50,000 tons in 1998 (Huang 1998), and the use of APEs (primarily NPEs) is also thought to have increased substantially in recent years (Feng 2005). Exact levels of current production and consumption are not known.

Both APEs and APs (especially NP and its derivatives) are widely distributed in fresh and marine waters and, in particular, sediments, in which these persistent compounds accumulate (Fu et al. 2008, Shue et al. 2010, David et al. 2009). In China, NP have been identified in river water and sediment in many areas including the Pearl River & Yangtze River basins, as well as in drinking water derived from contaminated river waters (Fu et al. 2008, Shao et al. 2005, Xu et al. 2006, Yu et al. 2009). Because of their releases to water, APEs and APs are also common components of sewage effluents and sludge (Micic & Hofmann 2009, Ying et al. 2009, Yu et al. 2009), including that applied to land. NP has been detected in rain and snow in Europe (Fries & Püttmann 2004, Peters et al. 2008), while residues of both NP and OP have been reported as contaminants in house dust (Butte & Heinzow 2002, Rudel et al. 2003) and indoor air (Rudel et al. 2003, Saito et al. 2004). Research into levels in wildlife remains limited, although there have been reports of significant levels in both invertebrates and fish in the vicinity of sites of manufacture and/or use of APEs and close to sewer outfalls (Lye et al. 1999, Rice et al. 2003, Mayer et al. 2008). Both NP and OP are known to accumulate in the tissues of fish and other organisms, and to biomagnify through the food chain (OSPAR 2004). Basheer et al. (2004) identified APs as common contaminants of seafood from Singapore. More recently, the presence of NP and OP have been reported in wild fish collected from locations along the Yangtze River (Brigden, et al. 2010b). NP and OP have also been recently reported in human tissues (Lopez-Espinosa et al. 2009).

The most widely recognised hazard associated with NP and OP is their estrogenic activity, ie their ability to mimic natural estrogen hormones. This can lead to altered sexual development in some organisms, most notably the feminisation of fish (Jobling et al. 1995, 1996). Atienzar et al. (2002) described direct effects of NP on DNA structure and function in barnacle larvae, a mechanism that may be responsible for the hormone disruption effects seen in whole organisms. In rodents, exposure to OP caused adverse effects on male and female reproductive systems, including lower sperm production and increased sperm abnormalities (Blake et al. 2004). Chitra et al. (2002) and Adeoya-Osiguwa et al. (2003) describe effects on mammalian sperm function, while DNA damage in human lymphocytes has also been documented (Harreus et al. 2002), although some have challenged the significance of these findings. Impacts on immune system cells *in vitro* have also been described (Iwata et al. 2004).

The manufacture, use and release of NP and OP, and their ethoxylates, are not currently regulated in China. However, NP and NPEs have very recently been included on the 'List of toxic chemicals severely restricted on the import and export in China' that requires prior permission for the import or exports of NP or NPEs (MEP 2011). Outside China, regulations addressing the manufacture, use and release of NP, OP and their ethoxylates do exist in some regions. More than 10 years ago, the Ministerial Meeting under the OSPAR Convention agreed on the target of cessation of discharges, emissions and losses of hazardous substances to the marine environment of the north-east Atlantic by 2020, and included NP/NPEs on the first list of chemicals for priority action towards this target (OSPAR 1998). Subsequently, t-OP was also listed under this category in 2000 (OSPAR 2006). Since then, NP has been included as a 'priority hazardous substance' under the European Union (EU) Water Framework Directive, such that action to prevent releases to water will be required throughout Europe within 20 years of adoption of the regulation (EU 2001). In 2008, the Water Framework Directive was amended, laying down environmental quality standards (EQS) for a number of priority substances (EU 2008). Octylphenols have been designated as 'priority substances' under the Water Framework Directive. Even before their listing under this Directive, however, the widely recognised environmental hazards presented by NP and OP and their ethoxylates have led to some long-standing restrictions on their use in many countries. Among these, a Recommendation agreed by the Paris Commission (now part of the OSPAR Commission) in 1992, required the phase-out of NPEs from domestic cleaning agents by 1995 and industrial cleaning agents by the year 2000 (PARCOM 1992). However, the precise extent to which this measure was effective is unclear. Furthermore, within the EU, products containing greater than 0.1% NP or NPEs may no longer be placed on the market within Europe after January 2005, with some minor exceptions principally for 'closed-loop' industrial systems (EU 2003). Relatively little information exists regarding the ongoing uses of OP and its derivatives in consumer products within Europe, though it remains widely used in the manufacture of tyres, insulating varnishes, and in certain polymers, inks and paints, as well as in some pesticide formulations (EA 2005).

Other alkylphenols: Methyl phenols (also known as cresols), are high production volume chemicals which have numerous uses including as preservatives and solvents, as well as in the production of numerous other chemicals, including dyestuffs (ATSDR 2008a). These chemicals can also be formed during combustion, and can therefore occur naturally at low levels (WHO 1995). Methyl phenols have previously been detected in receiving wastewaters at the Shaoxing wastewater treatment plant (SWWTP) in Zhejiang Province of China which mainly contain industrial wastewaters from printing and dyeing industries (Wang et al. 2008), though were not present in treated water suggesting that cresols may be successfully removed from wastewaters through conventional treatment. Some toxicity of cresols to aquatic protozoa has been reported (Duchowicz et al. 2008). 4,4'-Methylenebisphenol, also known as bisphenol F (BPF), has many uses including in resins and as an antioxidant (Pospíšil et al. 1971, Wang et al. 2008). BPF has been previously been detected in wastewater from the textile industry (Castillo & Barceló 2001), as has been commonly detected in the freshwater environment despite very few toxicity data being available for this chemical (Stachel et al. 2003, Murray et al. 2010). Information on bis(1-phenylethyl)phenol is likewise very limited. It has been previously detected in wastewater from a paper-mill industrial area suggesting use in thermal paper (Terasaki et al. 2007). One bioassay study suggested that this chemical has estrogenic activity.

River sediments

Following their discharge within wastewaters, many metals and organic chemical pollutants can accumulate in sediments within the receiving water body, and therefore analysis of river sediments can provide an indication of the nature and extent of chemicals released to the river over time, although with less certainty about the specific point sources of the substances identified compared to analysis of wastewaters themselves.

The sediment sample collected from the Fenghua River upstream of all pipes (CN10041) contained only aliphatic hydrocarbons and alkyl benzenes, the presence of which are likely to indicate contamination of the Fenghua River sediment by oil or other petrochemicals possibly arising from a wide range of urban or industrial sources (Overton 1994).

Very similar profiles of organic chemicals were found in the four samples of river sediment collected adjacent to the four discharge pipes; once again, aliphatic hydrocarbons and alkyl benzenes were common in these samples. In addition, however, two other types of organic chemicals were identified in the majority of these samples; 6 isomers of di-tolyl ether, present in all but CN10043 (collected close to pipe 1), and an ester of di-tert butyl benzenepropanoic acid (a long chain fatty acid derivative) found in all but the CN10047 (collected close to pipe 2). For these substances it is unclear whether there is any likely connection with textile processing operations at this location. Indeed, neither ditolyl ether nor the di-tert butyl benzenepropanoic acid ester were identified in the river sediment collected upstream from Youngor Chemicals (CN10041), nor in any of the water samples collected from this site. It is possible that both may be present in the sediment as a result of historic or only periodic discharges from the Youngor complex, though sources unrelated to the Youngor complex cannot be ruled out.

Ditolyl ether (otherwise known as 1,1'-oxybis(methyl)benzene) is known to be used in the preparation of dyestuffs, and was formerly used as a dielectric impregnating and insulating agent in electronics (marketed as Baylectrol 4900). It also occurs as an impurity in preparations used as levelling agents in the dyeing of nylon. Toxicological data are scarce, though there is evidence that it can cause structural changes in the liver, lungs and gastrointestinal tract of mice and is strongly irritating to the skin in rabbits (NICNAS 1994).

The concentrations of metals were similar in all five river sediment samples collected adjacent to the Youngor Textile Complex site including the sample collected from the river upstream of all pipes (CN10041), with the exception of chromium, and to a lesser extent, nickel in CN10043 collected close to pipe 1. Although the concentrations of chromium, nickel and zinc in all samples were somewhat higher than typical levels for uncontaminated river sediments, this may simply be indicative of general contamination of the Fenghua River in this area (or again may reflect historic or periodic discharges), since none of these metals were conspicuous in any of the wastewaters at the time of sampling.

4.1.2.Samples collected in March 2011

The Youngor Textile Complex site was revisited on 8 and 9 March 2011, and samples of discharged wastewater were collected from pipe 1, the pipe associated with the wastewater treatment plant, on three separate occasions within a total period of around 15 hours. The colour of the wastewater was observed to vary considerably over time during this period. Details of the samples collected on this occasion are provided in Table 3; note that, for clarity, a single sample number (CN11001-3) has been assigned for each of the three sampling times, although in practice the wide range of chemical-specific analyses conducted necessitated the filling of multiple sample bottles at each of these times. Data from these analyses are presented in summary form in Table 4 and discussed below. Complete results for all analyses conducted are provided in Appendix 2.

Table 3. Description of samples associated with the Youngor Textile Complex, Yangtze River Delta, China, March 2011.

Sample	Type	Description
CN11001	Wastewater	Discharge pipe 1, from the Youngor WWTP; 08-03-2011 (11 am)
CN11002	Wastewater	Discharge pipe 1, from the Youngor WWTP; 08-03-2011 (9 pm)
CN11003	Wastewater	Discharge pipe 1, from the Youngor WWTP; 08-03-2011 (9 pm)

Concentrations of the metals cadmium, lead and mercury in these samples were within or below the ranges reported for the samples collected from the same discharge pipe in June 2010 (CN10042 and CN10050) and are not considered to be indicative of significant levels of contamination. Specific analysis of chromium (VI), the most toxic and carcinogenic form of chromium, also revealed concentrations in this effluent which were not distinguishable from background levels which may be expected in uncontaminated waters. Among the organic compounds identified and quantified in these follow-up analyses were traces of:

- toluene (0.9 and 0.8 µg/l, or parts per billion [ppb], in CN11002 and CN11003 respectively);
- the chlorinated volatile compounds dichloroethane (0.9 µg/l in CN11003), trichloromethane/chloroform (all three samples at 0.9-4.8 µg/l) and tetrachloroethene (0.4 µg/l in CN11002 and CN11003);
- phenol and cresol (especially in CN11003, at 2.8 and 18 µg/l respectively);
- di-, tri- and pentachlorophenols (in the range of 0.03-0.06 µg/l);
- naphthalene (from 0.18-0.80 µg/l in all three samples); and
- certain amines, including aniline, 2-chloroaniline, methylaniline, ethylaniline and diethylaniline (in all three samples at concentrations ranging from 0.1-2.1 µg/l), as well as the carcinogenic amine, o-anisidine (at 0.07-0.08 µg/l).

It is worth noting that o-anisidine is a carcinogenic amine covered by EU Directive 2002/61/EC which prohibits the use "in textile and leather articles which may come into direct and prolonged contact with the human skin or oral cavity" dyes that can degrade to form a concentration of greater than 30 ppm of carcinogenic amines (such as o-anisidine) in the finished article under specific test conditions. Although the concentrations of this chemical present in the effluent are low and cannot be used to estimate concentrations in finished products manufactured on site, its presence suggests that further investigation in relation to the nature and quantities of harmful dyestuffs in use may be justified.

The presence of this diverse array of chemicals at concentrations in the low or sub parts per billion range indicate that this effluent discharge is acting as a point source (in some cases a periodic point source) of a range of hazardous substances to the local aquatic environment. On the basis of information available, it is not possible to determine the specific sources of these various substances in the wastewater within the facility, though they could include the deliberate use of these chemicals in processing and finishing operations on the site or the washing out of residues of such chemicals or their degradation products from yarn, fabric or textile products brought on to the site for processing from manufacturers located elsewhere. Further detailed investigations of activities taking place within the facility would be necessary in order to determine likely sources.

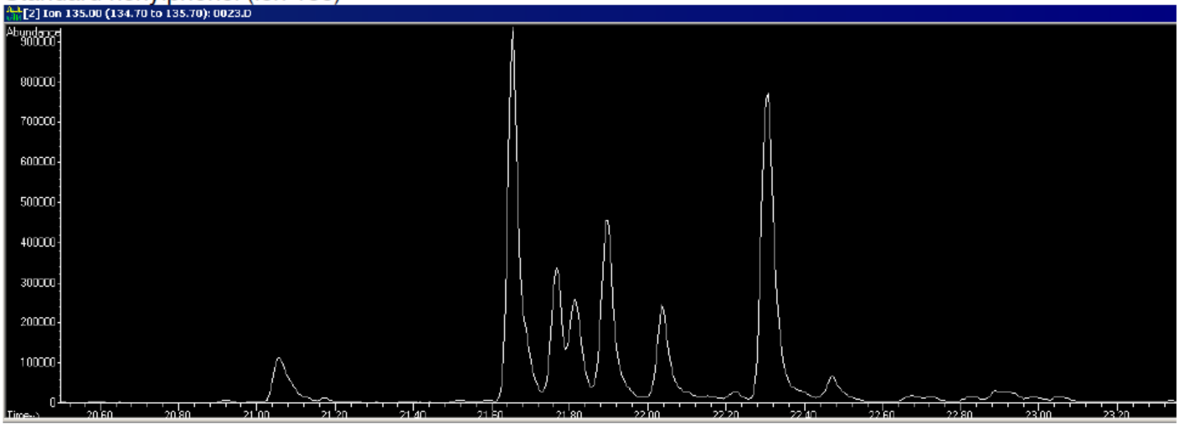
Perhaps of greatest significance was the presence of the persistent hormone-disrupting man-made chemical nonylphenol at 14 µg/l in the effluent collected at 1100 on 8 March (CN11001). This substance was not present at above limits of quantification for the method applied (10 µg/l) in the samples collected at the other two times. Although present at concentrations close to the limits of detection in CN11001, the match between total ion chromatographs and mass spectra of the compound isolated and a standard of nonylphenol (analysed using the same method) provide positive confirmation of its presence (Figure 2). Furthermore, the limits of quantification in this case were relatively high compared to those achieved in other studies and compared to the concentrations that might routinely be expected for surface water samples. The concentration of 14 µg/l is above the range typically reported for nonylphenol in river and lake water samples from a number of countries (generally <1 µg/l), including Spain (Céspedes et al. 2008), the Netherlands (Jonkers et al. 2005), Germany (Fries & Puttmann 2003), Korea (Li et al. 2004) and the United States (Rice et al. 2003), though higher concentrations were more routinely reported for samples collected in the 1990s and early 2000s before tight marketing and use restrictions came in to effect in Europe and certain other regions (see e.g. reviews by Ying et al. 2002 and Li et al. 2007). Relatively high (>1 µg/l) values have been reported more recently for some locations in East Asia, including in rivers in Taiwan (0.7-3.2 µg/l, Cheng et al. 2006) and mainland China (0.1-7.3 µg/l, Shao et al. 2005) in 2000, in the waters of Shihwa Lake in

Korea in 2002 (up to 1.5 µg/l in lake water and over 15 µg/l in some small industrialised creeks flowing in to the lake, Li et al. 2004), on the Mai Po marshes in Hong Kong in 2004 (0.029-2.59 µg/l, Li et al. 2007) and in various subtropical lakes in mainland China (1.9-32.8 µg/l, Wu et al. 2007). In such cases, the presence of elevated levels of nonylphenol has generally been attributed to the proximity of the water bodies to intensive industrial and/or livestock production zones.

Table 4. Organic chemicals and metals (in µg/l) in samples of wastewater discharged from pipe 1 of the Youngor Textile Complex wastewater treatment plant, 8 – 9 March 2011.

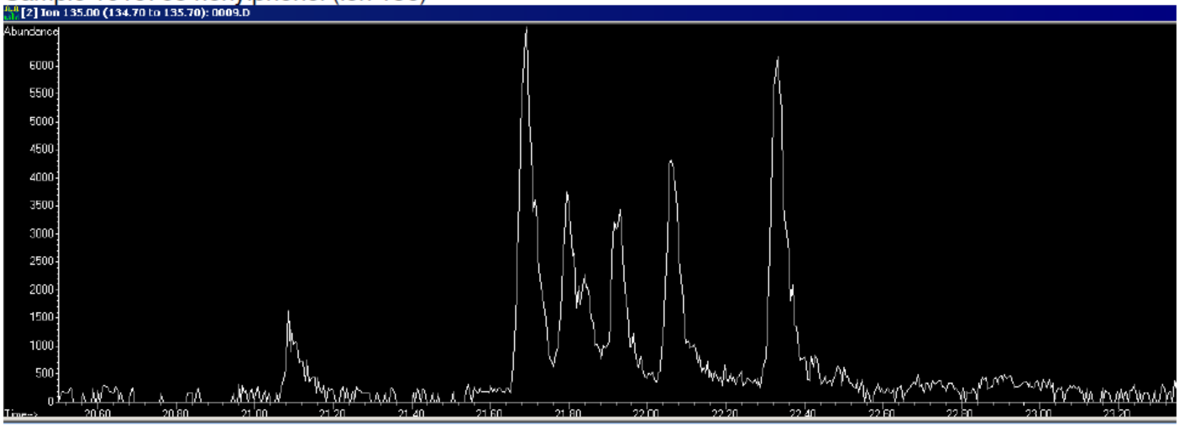
Substance or group	**CN11001 8 March, 11:00**	**CN11002 8 March, 21:00**	**CN11003 9 March, 02:00**
Metals			
Cadmium	<1.0	<1.0	<1.0
Mercury	0.14	0.05	0.07
Lead	32	<5	<5
Chromium (VI)	9	12	11
Non-chlorinated VOCs			
Benzene	<0.2	0.3	<0.2
Toluene	<0.2	0.9	0.8
Ethylbenzene + xylene	<0.4	<0.4	<0.4
Chlorinated VOCs			
1,2-Dichloroethane	<0.5	<0.5	0.9
Trichloromethane	0.9	1.5	4.8
Tetrachloroethane	<0.1	0.4	0.4
Others (11)	<0.1 - <0.5	<0.1 - <0.5	<0.1 - <0.5
Chlorinated benzenes			
1,4-Dichlorobenzene	<0.02	0.04	0.03
Others	<0.02	<0.02	<0.02
Phenols			
Phenol	<1.0	<1.0	2.8
m & p-Cresol	<1.0	1.1	18
Others (15)	<0.5 - <1.0	<0.5 - <1.0	<0.5 - <1.0
Chlorinated phenols			
2,4- & 2,5-Dichlorophenol	0.06	0.04	0.04
2,4,6-Trichlorophenol	0.03	0.03	0.03
Pentachlorophenol	0.04	0.03	0.03
Others (15)	<0.02 - <0.5	<0.02 - <0.5	<0.02 - <0.5
Alkylphenols			
Nonylphenol (NP)	14	<10	<10
NP mono- and di-ethoxylates	<10	<10	<10 - <50
PAHs			
Napthalene	0.18	0.80	0.80
Others (15)	<0.10 - <0.20	<0.10 - <0.20	<0.10 - <0.20
Perfluorinated chemicals (PFCs)			
Perfluorooctane sulfonate (PFOS)	0.0031	0.0087	0.0083
Perfluoroctanoic acid (PFOA)	0.140	0.130	0.130
Other perfluoro carboxylic acids	0.0310	0.0143	0.0131
Others	0.0021	0.0020	0.0015
Phthalate esters			
DnBP	<1.0	<1.0	1.3
DiBP	<1.0	1.1	1.5
DEHP	3.1	5.8	7.2
Others (8)	<1.0	<1.0	<1.0
Amines			
2-Chloroaniline	0.10	1.1	1.0
Aniline	1.9	1.8	2.1
n-Methyl- + n-ethyl- + n,n'-diethy-aniline	0.77	1.49	1.70
o-Anisidine	0.07	0.08	0.08
Others (43)	<0.05 - <1	<0.05 - <1	<0.05 – <1
Additional compounds			
BDEs (9)	<0.0005	<0.0005 - <0.04	<0.0005
Organotins (7)	<0.01 - <0.02	<0.01 - <0.02	<0.01 - <0.02
Tris(2-ethylhexyl)ester, phosphoric acid	Qualitative only	Qualitative only	Qualitative only

Standard nonylphenol (ion 135)



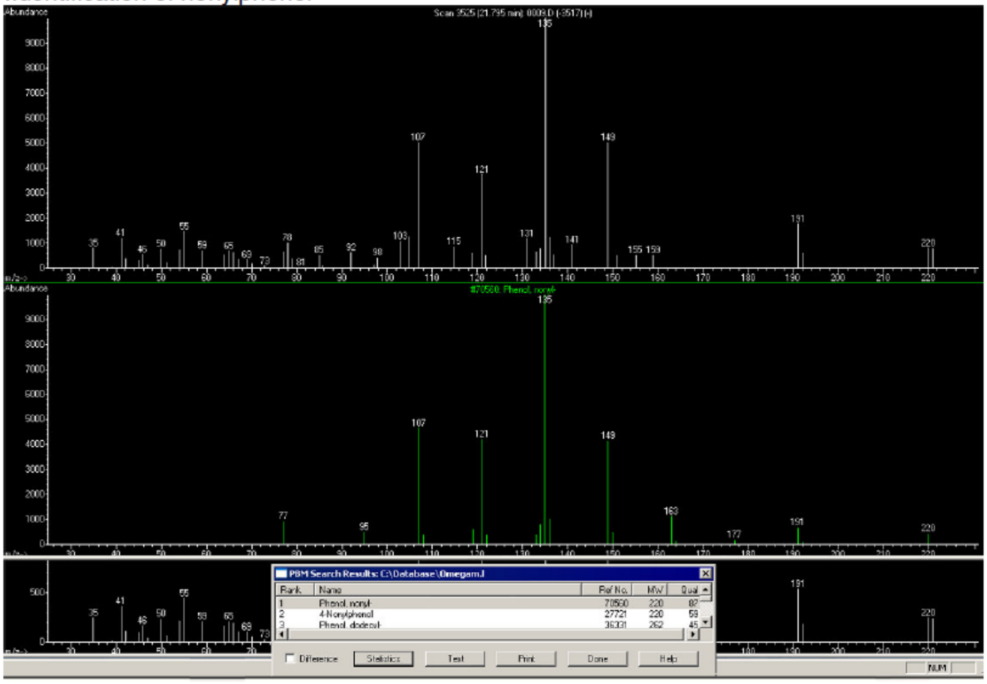
[2] Ion 135.00 (134.70 to 135.70): 0023.D

Sample 1015768 nonylphenol (ion 135)



[2] Ion 135.00 (134.70 to 135.70): 0009.D

.Identification of nonylphenol



Scan 3525 (21.795 min): 0009.D (-3517) (+)

#70560: Phenol, nonyl-

PBM Search Results: C:\Database\Omegam.l

Rank	Name	Ref No.	MW	Qual
1	Phenol, nonyl-	70560	220	87
2	4-Nonylphenol	27721	220	59
3	Phenol, dodecyl-	36331	262	45

Figure 2: Total ion chromatograph (TIC) and mass spectrum confirming the positive identification of nonylphenol (isomeric) in sample CN11001, collected from Pipe 1 of the Youngor Textile Complex at 1100 on 8th March 2011.

Relatively few recent studies report concentrations of nonylphenol in effluents collected directly from wastewater treatment plants at the point of discharge, though those data which are available suggest very wide variations in concentration (see reviews by Ying et al. 2002 and Soares et al. 2008), probably due to a combination of differences in the nature of inflowing wastes and in the efficiency with which treatment plants operate to partition nonylphenol residues to the sludges. However, the 14 µg/l recorded for one of the three additional samples in the current study is within the broad range which has historically been reported for nonylphenol residues in effluents from urban wastewater treatment plants in Europe before the EU prohibition on most uses of nonylphenol and its derivatives came in to effect e.g. 15 µg/l for sewage treatment plant effluent in Switzerland (Ahel et al. 1994), 0.25-2.3 µg/l in Germany (Spengler et al. 2001), 5.5-6.6 µg/l in Spain (Farre et al. 2002) and as high as 32-63 µg/l in the UK (Sheahan et al. 2002). In comparison, relatively low concentrations have been reported in the few studies available to date for sewage treatment plant effluents in China; for example, Lian et al. (2009) reported concentrations ranging from below limits of detection to approximately 0.44 µg/l for four major sewage treatment plants serving Beijing.

As noted in Box C, nonylphenol is generally found in environmental samples as a mixture of related (isomeric) chemicals with the same molecular weight but differing in their precise chemical structure, accounting for the multiple peaks apparent from Figure 2 that were identified as nonylphenol in both the standard and sample CN11001. Its presence may result from one of a variety of uses of the parent compound or, perhaps more commonly, the partial degradation of nonylphenol ethoxylate compounds which still have widespread use in some parts of the world (including China) as powerful detergent chemicals. The presence of nonylphenol in one of the three effluent samples collected in March 2011 indicates that the Youngor Textile Complex is, at least periodically, acting as a localised point source of this chemical group to surface waters. As for the other organic compounds identified, it is not possible from the investigations conducted to date to determine the likely process origins of nonylphenol within the factory site, though again there are the possibilities that these levels arise from deliberate use on site, from the washing out of residues from yarn, fabric or textiles manufactured elsewhere and brought in to the plant or a combination of the two. Furthermore, as we have analysed effluent discharges from only one facility in the area, it is not possible to speculate on the relative significance of this point source in terms of its contribution to overall inputs and levels of nonylphenol in the local and regional environment.

Also of interest was the confirmed presence of several perfluorinated chemicals in the effluent samples collected at all 3 sampling times, especially perfluoroctanoic acid (PFOA) at concentrations between 0.13 and 0.14 µg/l (130-140 ng/l). A number of other perfluorocarboxylic acids were also found, though at concentrations around an order of magnitude lower (0.013-0.031 µg/l, 13-31 ng/l), while perfluoroctane sulphonate (PFOS) was present at lower levels again (0.0031-0.0087 µg/l, 3.1-8.7 ng/l). Although these concentrations appear low, those for PFOA are above the range commonly reported for such chemicals in surface waters in several parts of the world and within the range reported for effluents from wastewater treatment plants, especially those which are known to receive industrial effluents for treatment.

For example, Loos et al. (2009) summarise data for water samples from 100 rivers in Europe, with average concentrations of PFOA and PFOS (commonly the two most abundant PFCs in surface water) of 12 and 39 ng/l, and medians of 3 and 6 ng/l respectively. Similarly, Ahrens et al. (2009, 2010) report levels of PFOA and PFOS at different points along the Elbe River of 2.8-9.6 and 0.5-2.9 ng/l respectively. Some water courses in Europe do appear to be more contaminated, yielding highs of 88 ng/l PFOA in the Belgian Scheldt, 100 ng/l in the UK's River Wyre and 116 ng/l in the Rhone in France, and similar or even higher concentrations of PFOS in the Scheldt (Belgium and Netherlands), Seine (France) and Severn (UK) (Loos et al. 2009). McLachlan et al. (2007) reported 200 ng/l PFOA in Northern Italy's River Po.

Although fewer data are available for locations in Asia, studies are beginning to build a picture of typical contaminant levels in China. Jin et al. (2009) found both PFOS and PFOA at various locations along the Yangtze River in 2004, with medians of 5.4 ng/l PFOA and 4.2 ng/l PFOS, though two locations immediately downstream from centres of industry yielded concentrations of 110 and 297 ng/l respectively. Similarly, So et al. (2007) reported levels of PFOA of 0.85-13 ng/l for stations on the Pearl River and 2-260 ng/l on the Yangtze, with PFOS showing the opposite trend (higher ranges of values on the Pearl River than on the Yangtze), possibly reflecting differences in patterns of industrial use and release of PFC formulations. Very recently, Yang et al. (2011) have reported the presence of PFOA and PFOS, among other PFCs, in waters from both the Liao River and Lake Taihu, with concentrations in the tens to hundreds of ng/l. In some parts of China, the presence of PFCs such as these has already become a problem for the clean-up of drinking water supplies (Jin et al. 2009).

In another recent study, this time in Korea, Kim et al. (2011) note that, while elevated concentrations of PFOS can be found immediately downstream from industrial zones dominated by metal-working facilities (34-550 ng/l), the highest concentrations of PFOA (26-730 ng/l) occur downstream from areas with high densities of textile manufacturing and finishing operations. The importance of textile production as point sources of PFOA, as well as PFOS and other PFCs in some instances, has also been noted from studies of effluent discharges from wastewater treatment plants in other countries, including Italy (Bossi et al. 2008) and Germany (Ahrens et al. 2009). Furthermore, in the US, concentrations of PFOA (2.5-97 ng/l) and PFOS (1.1-130 ng/l) were found to be far higher in effluents from treatment plants receiving a high proportion of mixed industrial waste than those receiving primarily urban wastewater (Schulz et al. 2006, Sinclair & Kannan 2006), a pattern also reported more recently for treatment plants in Singapore (PFOA 15.8-1057.1 ng/l, PFOS 7.3-461.7 ng/l) sampled during 2006-7 (Yu et al. 2009).

PFCs have relatively low solubility in both water and oils (which partly accounts for the commonly low concentrations found in surface waters), unique properties that have underpinned their development and widespread use as water, grease and stain-repellent finishes on textile and paper products, as well as for specialised solvents and surfactants used in industry, and as components of cosmetics and plastic products (OECD 2002, Hekster et al. 2003). Their resistance to breakdown even at high temperatures has also led to their use in fire fighting foams and in lubricants for high temperature applications (OSPAR 2006). Further background information on perfluorinated chemicals is provided in Box D. Once again, while there may be a number of possible sources of such chemicals on a site such as the Youngor Textiles Complex, identifying the specific origins of the residues found in these effluent samples is not possible based on available information.

As noted in Section 3 (Methodology), samples CN11001-3 were also subjected to additional, non-quantitative screening analyses by the independent laboratory in order to try to identify other organic chemicals present in the effluent being discharged. Although the analytical methods employed by the independent laboratory were similar to those employed in our own laboratory for samples collected in June 2010, the qualitative data generated were analysed in a different way, using a more restricted library of mass spectra to 'fingerprint' the chemicals present. For this reason, although many other chemicals were also seen to be present (in the form of 'peaks' in the total ion chromatograms), only one could be positively identified by matching of the mass spectrum to an acceptable quality. This chemical, the tris(2-ethylhexyl)ester of phosphoric acid, was also more tentatively identified in the effluent samples collected in June 2010 from the same pipeline, which were analysed in our laboratory (not included in Table 2 as the match quality against the library spectrum was insufficient for reliable identification in that case).

Box D: Perfluorinated chemicals (PFCs)

PFCs are man-made chemicals in which all the carbon-hydrogen bonds present in the organic chemical backbone have been replaced by strong carbon-fluorine bonds, making them highly resistant to chemical, biological and thermal degradation (So et al. 2004). Many PFCs also have relatively low solubility in both water and oils, unique properties that have underpinned their development and widespread use as water, grease and stain-repellent finishes on textile and paper products, as well as for specialised solvents and surfactants used in industry and as components of cosmetics and plastic products (OECD 2002, Hekster et al. 2003). Their resistance to breakdown even at high temperatures has also led to their use in firefighting foams and in lubricants for high temperature applications (OSPAR 2006). However, the properties of this group of chemicals also result in one of their major environmental down-sides of PFCs, namely their long persistence in the environment once they are released, whether from manufacturing or disposal operations or during the useful lifetime of a product (Key et al. 1997).

The PFCs that have been manufactured over the past 60 years fall into four broad categories; Perfluorinated alkyl sulphonates (PFASs), perfluorocarboxylic acids (PFCAs), fluoropolymers (the best known being PTFE, marketed as Teflon and widely used for 'non-stick'cookware) and fluorotelomer alcohols (FTOH) (Dinglasan et al. 2004). Until 2000, the most widely used PFASs globally were those based on perfluorooctane sulfonate (PFOS). At that time, the annual production of all PFOS-related chemicals in the USA alone was 3,000 tonnes (Stock et al. 2004). Since 2000, global production of PFOS and equivalent chemicals has fallen sharply, and is currently estimated to be below 1,000 tonnes per annum (Paul et al. 2009). In contrast, production within China has increased in recent years. It has been reported that large-scale production of PFOS in China began in 2003, with total production before 2004 of 50 tonnes, increasing to over 200 tonnes per annum in 2006, of which approximately half was exported to the EU, Japan and Brazil (Bao et al. 2010).

Perfluorooctanoic acid (PFOA) is the most well-known of the PFCAs, being used as a polymerisation aid in the manufacture of the fluorinated polymer PTFE. It has been reported that PFOA is not currently manufactured in China, but is imported to produce PFC-related formulations (FECO/MEP 2009). Other than through deliberate production, PFOA (along with other shorter and longer-chain PFCAs) can also be generated as an unintended by-product in the manufacture of perfluorinated telomer alcohols, or FTOHs (Poulsen & Jensen 2005).

PFASs (especially PFOS) and PFCAs (especially PFOA) have been reported as contaminants in almost all environmental media, including freshwater, groundwater and seawater sediments and soils. Within China, PFCs including PFOS and PFOA have been reported in various environmental media including waters from many river systems (So et al. 2007, Jin et al. 2009, Lien 2007 cited in Kunacheva et al. 2009). A range of PFCs - particularly PFOA, but also PFOS and, to a lesser extent, PFOSA and some other PFCAs (including perfluorohexanoic acid (PFHxA), perfluoroheptanoic acid (PFHpA) and perfluorodecanoic acid (PFDA)) - have been detected in river water from locations along the course of the Yangtze River, including at Chongqing, Nanjing and Shanghai (So et al. 2007). Highest concentrations were generally found in samples from near Shanghai. Others have reported the presence of PFOS and PFOA in Yangtze River water along the course of the river between Chongqing and Wuhan. In addition, PFOA, PFOS and other PFCs have been reported in the sediments of various rivers in China, including sediment collected from the Huangpu River, a tributary to the lower Yangtze River, which contained PFOA at levels higher than those generally reported in rivers worldwide (Bao et al. 2010). Yangtze River Estuary sediments have yielded some of the highest PFOS concentrations ever recorded (Pan & You 2010). Furthermore, the presence of PFOS and PFOA has also been reported in tap water in many cities in China, including Chongqing, Wuhan and Nanjing. Some of the highest concentrations of PFOS and PFOA were found in tap waters from Shanghai, Wuhan and Nanjing on the Yangtze River, as well as in Guangzhou and Shenzhen on the Pearl River Delta (Jin et al. 2009, Mak et al. 2009).

Unlike many persistent organic pollutants, PFOS accumulates in the bodies of animals by binding to proteins in the blood, thereby building up to particularly high levels in liver tissue (Giesy & Kannan 2001, Martin et al. 2003a, b). Numerous studies have reported the presence of PFCs in tissues of aquatic invertebrates, amphibians, fish, birds and mammals (from mice to far larger mammals including whales and polar bears) (Giesy & Kannan 2001, Houde et al. 2006). PFOS and PFOA have also been reported in red and giant pandas from zoos and wildlife parks within China (Dai et al. 2006). PFOS is generally the predominant PFC reported in the tissues analysed (Giesy & Kannan 2001, Kannan et al. 2005, Houde et al. 2006). PFOA and perfluorononanoic acid (PFNA) – or, occasionally perfluoro-n-undecanoic acid (PFUnDA) - have generally been the most abundant PFCAs reported in animal tissues, particularly in top predators (Martin et al. 2004, Smithwick et al. 2005). In China, PFCs have been found in the blood of fish, including common carp and leather catfish from Gaobeidan Lake near Beijing (Li et al. 2008), in wild sturgeon, carp and catfish from the Yangtze (Peng et al. 2010, Brigden et. al. 2010b) and in seafood from Zhoushan and Guangzhou (Gulkowska et al. 2006).

PFOS and other PFCs have been found in human blood, cord blood and breast milk in many countries around the world, even in remote areas such as the Canadian Arctic, with PFOS generally the most abundant (Kannan et al. 2004, Toms et al. 2009, Rylander et al. 2010, Dallaire et al. 2009, Monroy et al. 2008, Tao et al. 2008). The same is true in China, with perflurohexansulfonate (PFHxS) being the next most abundant (Yeung et al. 2006, 2008, Liu et al. 2009), though other PFCs have also been detected. In the USA, average concentrations of PFOS, PFOA and PFHxS in blood samples have reduced in recent years, which may be as a result of the discontinuation of industrial production of PFOS and related chemicals in the US in 2002 (Calafat et al. 2007). Conversely, in Shenyang, China, levels of PFOS and PFOA in human blood increased between 1987 and 2002 (Jin et al. 2007). Research shows that food intake is the major route for exposure to PFCs in the general population (Fromme et al. 2009, Vestergren and Cousins 2009, Zhang et al. 2010a), though additional exposure may occur in populations living near PFC production facilities. In China, PFOS and PFCAs are found in various foods (meat and eggs), though seafood may account for the majority of human exposure there (Zhang et al. 2010).

Studies of laboratory animals indicate that PFCs can cause adverse impacts during development and during adulthood. PFOS and PFOA have both been reported to have adverse effects on the liver in rodents and monkeys (Kawashima et al. 1995, Adinehzadeh et al. 1999, Berthiaume & Wallace 2002, Lau et al. 2007). PFCs have also been shown to act as hormone (endocrine) disruptors (Jensen and Leffers 2008). Some examples of the effects PFCs have on endocrine systems include decreasing testosterone levels and increasing estradiol levels in adult rats, which can result in changes in the cells of the testis (Jensen and Leffers 2008), as well as the hormone imbalance and growth defects in offspring of rodents exposed to PFOS when pregnant (Lau et al. 2007, Yu et al. 2009). Other studies indicate impacts on the developing immune system of mice following prenatal exposure to PFOS (Keil et al. 2008) and in adult rodents (Yang et al. 2002, Lau et al. 2007, DeWitt et al. 2008, Peden-Adams et al. 2008).

PFCs may also impact on hormone systems in humans. In the USA, higher PFOA and PFOS concentrations in blood have been associated with thyroid disease in the general adult population (Melzer et al. 2010). High combined levels of PFOA and PFOS in men’s blood in Denmark were associated with having fewer normal sperm (Joensen et al. 2009), while Danish women with higher blood levels of PFOA and PFOS took longer to become pregnant than those with lower levels (Fei et al. 2009). Elevated levels of PFC exposure may also affect foetal growth and development, though there are inconsistencies between different studies (Fei et al. 2008 & 2009, Olsen et al. 2009). Likewise, evidence for adverse health effects as a result of occupational exposure to PFOA and PFOS in the workplace remains inconclusive (Lau et al. 2007, Alexander & Olsen 2007, EFSA 2008).

Marketing and use of PFOS is prohibited within the EU and in Canada for certain uses, although many exemptions exist (EU 2006, CEPA 2008). The above regulations do not apply to PFCAs and other PFCs. In China, there are currently no regulations of the manufacture and use of PFOS, or other PFCs. However, PFOS has recently been included among the persistent organic pollutants (POPS) regulated under the Stockholm Convention, a global treaty to protect human health and the environment. Under the terms of the Convention, contracting parties must take measures to restrict the production and use of PFOS, although a wide range of uses are currently exempt, including uses in the semiconductor and photographic industries, metal plating operations, aviation hydraulics, fire-fighting foams, and certain pesticides (UNEP 2009).

4.2 Well Dyeing (Pearl River Delta)

The Well Dyeing facility is located in the Gao Ping Industry District, Sanjiao, Zhongshan City, in Guangdong Province, within the Pearl River Delta. This facility is one of many dyeing facilities located in the Gao Ping Industry District. The Well Dyeing facility is a large complex that includes various production plants and a wastewater treatment plant (WWTP), as well as a power generation plant, workers' dormitories and administration buildings. This facility manufactures a wide variety of fabrics including knitted fabrics, velour, fleece and spandex. Processes carried out include the pre-production treatment of fibres, bleaching, dyeing and textile finishing (ZRIEP 2008, Well Dyeing 2010).

The Shiji River borders the west side of the Well Dyeing site, and the Gaosha River borders the east side of this site. These two small rivers are both tributaries to the far larger Huangshali Channel, a branch of the Pearl River Delta that ultimately flows into the South China Sea (see Figure 3). The river system is tidal at this location, though the Shiji River is connected to the Huangshali Channel by a sluice gate that controls the flow of water. Other facilities, not connected with Well Dyeing, are located in the vicinity, some of which also appear to discharge wastewaters to the Shiji River.

Greenpeace investigations revealed that the discharge from pipe 1 (which was observed as leading from the discharge point right up to the perimeter wall of this facility, and on the other side of this wall, to within 2 metres of the WWTP) was sampled at a time when there was no rain (nor standing surface water within or outside the Well Dyeing site) for several hours preceding the sampling. This pipe (pipe 1), located closest to the main Huangshali Channel, discharges wastewater sporadically into the Shiji River which is connected to the Huangshali Channel, and was only observed discharging during the night. Pipe 2 discharges wastewater from the Well Dyeing facility (including domestic sewage, rainwater and industrial wastewater). Pipe 2 was observed on a number of occasions to continually discharge wastewater to the Shiji River with a substantial flow.

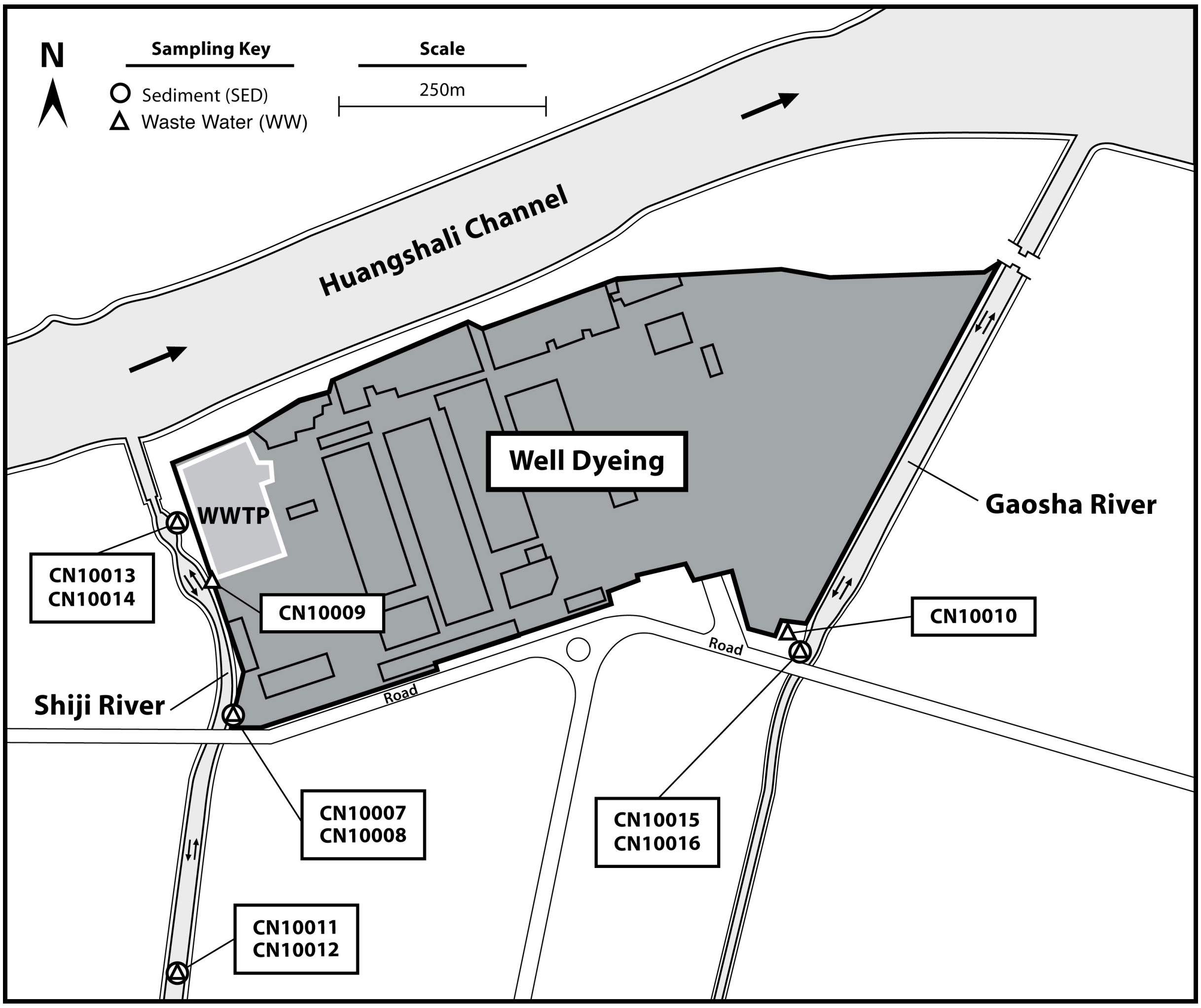


Figure 3. Sketch map of the Well Dyeing complex showing the locations from which samples were collected. Gao Ping Industry District, Zhongshan City, in Guangdong Province, China 2010. WW - wastewater, SED – sediment

Pipe 3, the outfall of which is semi-submerged in the Shiji River and which is located in the southwestern corner of the Well Dyeing complex adjacent to a road bridge over the Shiji River, discharged wastewater from the Well Dying site.

It was not possible to confirm whether these pipes (pipe 1, pipe 2 or pipe 3) are connected to the WWTP within the Well Dyeing facility, which is located close to the junction of the Shiji River and the Huangshali Channel

Samples of discharged wastewater were collected from each of these pipes, along with samples of river sediment from locations adjacent to pipes 1 and 3. In addition, a sample of river water (CN10011) and river sediment (CN10012) were collected from the Shiji River approximately 200 m from pipe 3, in the opposite direction to the Huangshali Channel. Details of these samples are provided in Table 5, and their locations are shown in Figure 3.

Table 5. Description of samples associated with the Well Dyeing complex, Gao Ping Industry District, Zhongshan City, in Guangdong Province, China, 2010

Sample	Type	Description
CN10011	River water	Shiji River, approx. 100 m from discharge pipe 1
CN10012	Sediment	Shiji River, approx. 100 m from discharge pipe 1 (as CN10011)
CN10007	Wastewater	Pipe 3; Semi-submerged discharge pipe into the Shiji River, by road bridge
CN10008	Sediment	Shiji River, adjacent to pipe 3 (see CN10007)
CN10009	Wastewater	Pipe 2; discharge pipe into the Shiji River, from the Well Dyeing facility
CN10013	Wastewater	Pipe 1; discharge pipe into the Shiji River, intermittent flow
CN10014	Sediment	Shiji River, adjacent to pipe 1 (see CN10013)
CN10010	Wastewater	Wastewater holding tank outside factory perimeter, intermittently discharged into the Gaosha River. Origin of water is unknown
CN10015	Wastewater	Pipe 4; Submerged pipe adjacent to the wastewater tank (CN10010), discharging into the Gaosha River. Origin of pipe is unknown
CN10016	Sediment	Gaosha River, adjacent to pipe 4 (see CN10015)

On the opposite side of the Well Dyeing complex, an open tank containing wastewater is situated just outside the perimeter wall of the complex and adjacent to the Gaosha River. There have been reports that water from the tank is intermittently discharged to the river, though this was not taking place at the time of sampling. Due to the proximity of the holding tank to the complex and the reported discharge to the river, a sample of wastewater (CN10010) was therefore collected from this tank as part of the current study. However, the origin of wastewater within the tank is not known.

Located close to this tank, an additional small pipe (pipe 4) was observed to be discharging black coloured wastewater beneath the surface of the Gaosha River. The origin of wastewater discharged via pipe 4 is not known. A sample of wastewater/river water (CN10015) was collected from the point of discharge, along with a sample of sediment (CN10016) from the Gaosha River adjacent to this outfall.

The data from the analyses of all samples associated with the Well Dyeing facility are presented in Table 6.

Location	**Shiji River**											**Gaosha River**				
	River			Pipe 3			Pipe 2		Pipe 1			Tank		Pipe 4		
Sample	**CN10011**		**CN10012**	**CN10007**		**CN10008**	**CN10009**		**CN10013**		**CN10014**	**CN10010**		**CN10015**		**CN10016**
Type	River water		River sediment	Wastewater		River sediment	Wastewater		Wastewater		River sediment	Wastewater		Wastewater		River sediment
	Total	Diss.		Total	Diss.		Total	Diss.	Total	Diss.		Total	Diss.	Total	Diss.	
Metals	(µg/l)	(µg/l)	(mg/kg)	(µg/l)	(µg/l)	(mg/kg)	(µg/l)	(µg/l)	(µg/l)	(µg/l)	(mg/kg)	(µg/l)	(µg/l)	(µg/l)	(µg/l)	(mg/kg)
Antimony	<100	<100	<20	<100	<100	<20	<100	<100	529	<100	<20	<100	<100	<100	<100	<20
Arsenic	<50	<50	27	<50	<50	<20	<50	<50	<50	<50	27	<50	<50	<50	<50	<20
Barium	48	32	177	34	34	167	79	48	4290	12	222	68	71	50	45	132
Cadmium	<5	<5	2	<5	<5	<1	<5	<5	33	<5	4	<5	<5	<5	<5	2
Chromium	<20	<20	101	<20	<20	71	<20	<20	2820	42	47	<20	<20	277	183	89
Chromium (VI)	-	<50	-	-	<50	-	-	<50	-	<50	-	-	<50	-	<50	-
Cobalt	<20	<20	19	<20	<20	25	<20	<20	99	<20	13	<20	<20	<20	<20	18
Copper	<20	<20	156	<20	<20	59	25	<20	13400	24	47	<20	<20	<20	<20	67
Lead	<50	<50	48	<50	<50	28	<50	<50	894	<50	53	<50	<50	<50	<50	30
Manganese	90	<10	835	73	72	1110	319	<10	9000	26	915	425	426	133	129	577
Mercury	<2	<2	<0.5	<2	<2	<0.5	<2	<2	17	<2	<0.5	<2	<2	<2	<2	<0.5
Nickel	<20	<20	50	<20	<20	43	<20	<20	2800	37	28	<20	<20	<20	<20	32
Selenium	<200	<200	<30	<200	<200	<30	<200	<200	<200	<200	<30	<200	<200	<200	<200	<30
Vanadium	<20	<20	77	<20	<20	106	<20	<20	324	<20	52	<20	<20	<20	<20	70
Zinc	23	<10	277	11	<10	123	68	<10	20100	24	211	61	18	35	13	153
Organic compounds isolated	11		12	44		17	22		172		11	66		93		18
No. identified (% of total)	4 (36)		5 (42%)	14 (32%)		11 (65%)	7 (32%)		30 (18%)		10 (91%)	22 (33%)		32 (34%)		14 (78%)
Tri-alkyl phosphates; (TEP)	1			1			1		1			1				
Others; (TBP/TiBP/TBEP)				1			1		1			2		1		
Phenols																
Nonylphenol									15							
Octylphenol									1			1		1		
Octylphenol ethoxylates														1		
Others												1		3		
Quinoline and derivatives				1					1			1		2		
Glycol ethers									2			2		1		
Phthalates (DEHP & DEP)				1										1		
Dichloroaniline (DCA)									1							
Di- and tri-chlorobenzenes				(4)												
Quinone / ketone derivatives																
Benzoquinone derivatives														2		
Anthraquinone and derivatives												2				
Naphthalic anhydride												1				
Benzophenone derivative									1							
Fatty acid and derivatives									1			1		1		
Other oxygenated aliphatics	1										1			1		
Atrazine	(1)			1			(1)					(1)				
PAHs												2		2		2
Oxygenated PAHs												4				
Benzothiazole derivatives												1		1		
Alkyl benzenes												1		1		3
Aliphatic hydrocarbon	1		5	5		11	4		5		9	1		13		9
Steroids / sterols									1					1		

Table 6. Organic chemicals identified, and concentrations of metals, in samples of discharged wastewaters, river water and sediment associated with the Well Dyeing complex, Gao Ping Industry District, China, 2010. For organic compounds, (..) signifies those identified at trace levels using a selective SIM method. Concentrations of metals in wastewater and river water samples are reported in µg/l, both as total concentrations in the whole (unfiltered) sample and separately as (dis)solved concentrations in filtered samples. Concentrations of metals in sediments are reported as mg/kg dry weight.

A diverse range of hazardous substances were identified in the samples of discharged wastewater collected from the vicinity of the Well Dyeing facility, including known outfalls from this facility. Certain groups of organic chemicals were identified in either all, or in the majority of, the wastewater samples.

Analysis of the wastewater sample collected from Pipe 2 to the Shiji River (CN10009) identified two trialkyl phosphate esters. These two substances were triethyl phosphate (TEP) and tributyl phosphate (TBP). TBP was also identified in a sample of river water (CN10011) collected from the Shiji River approximately 100 m to the south of discharge Pipe 3.

Tributylphosphate (TBP) is widely used in various industrial processes, including by the textile industry due to properties of being a strong wetting agent and strong polar solvent. Triethyl phosphate (TEP) has uses as a plasticiser and flame retardant. These chemicals have been frequently found in surface water environments, especially near sewage treatment plants discharges (Andersen et al. 2004, Alink et al. 2005, Reemtsma et al. 2008, Regnery & Puttmann 2010). More information on these tri-alkyl phosphates is presented in Box A.

Other organic substances identified in this sample (atrazine and aliphatic hydrocarbons) were commonly found in samples from this site, and their presence is likely to be unrelated to the manufacture of textiles at this site. These substances are discussed towards the end of Section 4.2.

A wider range of organic chemicals were identified in the wastewater sample (CN10013) collected from the other pipe located in the vicinity of the WWTP (pipe 1), which discharges sporadically to the Shiji River, and was only observed discharging during the night. These included the trialkyl phosphates identified in the sample from pipe 2 (TBP and TEP), as well as alkylphenols and related alkylphenol ethoxylates, a quinoline derivative, dichloroaniline (DCA), glycol ethers and a benzophenone derivative. Due to the sporadic nature of releases via pipe 1, had the collection of wastewater samples for this site been attempted at other times it is possible that no sample from pipe 1 would have been available, and therefore the discharge of substances identified in CN10013 via this outfall would remain unknown.

Two types of alkylphenol were identified in the wastewater sample from pipe 1 (CN10013); nonylphenols (15 individual isomers) and the closely related chemical, octylphenol. Nonylphenol (NP) and octylphenol (OP) are well known persistent and bioaccumulative environmental contaminants, with hormone-disrupting properties for many aquatic organisms. These chemicals are formed by the partial breakdown of nonylphenol ethoxylates (NPEs) and octylphenol ethoxylates (OPEs) respectively, substances which are used as detergents in numerous industrial processes, including during the manufacture of textiles. Nonyl and octylphenol do also have other industrial uses in their own right. The use and release of NP and OP, and their ethoxylates, are regulated in certain regions outside of China (EU 2003, EU 2008, OSPAR 1998). The import of NP and NPEs into China has very recently been regulated (MEP 2011), though their use and release are not currently controlled. More information on these substances is presented in Box C.

Quinoline is used in the manufacture of dyes (amongst other industrial uses), including water-soluble dyes such as Quinoline Yellow (US EPA 2001). The related chemicals identified, methylquinolines, are also used as precursors to certain dyes (Gu et al. 2010, Shindy et al. 2002). Quinoline and its derivatives are known environmental contaminants, having been found in wastewaters discharged from dyes and intermediary products industry (Yuan et al. 2006, Oliveira et al. 2007), as well as at sites contaminated with tar oils (Reineke et al. 2007, Thomsen et al. 1999). Quinoline is a toxic chemical with reported effects including liver damage in mammals (US EPA 2001). Methylquinolines have been reported to have moderate aquatic toxicity in bioassay tests (Birkholz et al. 1990).

Dichloroanilines (DCAs), also known as dichlorobenzenamines, are aromatic amines, chemicals with many industrial uses which include the manufacturing of dyes (Stellman et al. 1998, Sokolowska et al. 2001). Anilines are an important class of environmental water pollutants due to their wide usage and high solubility in water. The release of anilines into the environment within industrial effluents has been previously reported, including within effluents from the textile sector (Pinheiro et al. 2004, Carvalho et al. 2010). DCAs are toxic to wide range of aquatic organisms (Dom et al. 2010), and mammals (Valentovic et al. 1995 & 1997). DCAs were not identified in any other wastewater samples associated with this site. More information on chlorinated anilines is presented in Box E.

One of the two glycol ethers identified was dipropylene glycol monomethyl ether (DPGME), a substances primarily used in solvents for paints and inks (OECD 2001, VWA 2005). Such glycol ethers have previously been reported as widespread environmental contaminants in other regions, including in river water & derived drinking water (Hendriks et al. 1994, Paune et al. 1998). However, these chemicals are not persistent in the environment following release, and are not expected to bioaccumulate (US EPA 2006). Furthermore, studies indicate that DPGME has low acute toxicity to animals (OECD 2001), though there are large gaps in the knowledge of the biological effects of many glycol ethers.

The benzophenone derivative, methyl 2-benzoylbenzoate, has uses as a photoinitiator in UV-curable inks, though little information is available on the toxicity of this chemical (Müller et al., 2008), see Box D.

In addition to the organic chemicals identified, the wastewater sample from pipe 1 (CN10013) also contained concentrations of dissolved chromium (42 µg/l), copper (24 µg/l) and nickel (37 µg/l) at levels that were slightly elevated (2-4 times higher) above levels typically found in uncontaminated surface waters. Background surface waters concentrations of dissolved chromium and copper are both typically below 10 µg/l, and often far lower, while those of nickel are generally below 20 µg/l (ATSDR 2004, ATSDR 2005, ATSDR 2008b, Comber et al. 2008). The levels of dissolved chromium, copper and nickel in these samples were considerably lower than their maximum allowable concentrations under the Guangdong effluent standard (Guangdong Province 2001) and national effluent standards relevant to the textile industry (MEP 1992), see Box F for details.

Box E. Chlorinated anilines

Dichlorobenzenamines, also called as dichloroanilines (DCAs), belong to a family of aromatic amines or anilines. They are used in the manufacturing of rubbers and plastics, dyes, pesticides, herbicides and pharmaceuticals (Stellman et al. 1998, WHO 2003, Sokolowska et al. 2001). Anilines can be released into the environment directly within industrial effluents from the textile (Pinheiro et al. 2004) or leather industry, or indirectly as breakdown products of herbicides and pesticides such as diuron, linuron and propanil (Stasinakis et al. 2009, Badawi et al. 2009, Mestankova et al. 2010, Carvalho et al. 2010). Anilines are an important class of environmental water pollutants due to their wide usage and high solubility in water.

Aniline and its chlorinated derivatives, including mono-, di- and trichlorinated isomers, are toxic to wide range of aquatic organisms (Dom et al. 2010). 3,4-DCA is the most studied dichloroaniline in terms of aquatic toxicity. In one of the earlier studies (Adema & Vink 1981), the aquatic toxicity of 3,4-dichloroaniline was determined for many marine and fresh water animals and several unicellular algae. Sub-lethal effects were detectable after exposure to very low concentrations of DCAs in those test organisms. Reproduction of *Ophryotrocha diadema* and of *Daphnia magna* were the most sensitive criteria. Significant effects were obtained with both test species at concentration of 0.010 mg/l. Similar results were obtained in later studies of 3,4-DCA aquatic toxicity (Crossland 1990, Guilhermino et al. 1994). 3,4-DCA has been also found to be toxic to fish (Monteiro et al. 2006, Schäfers & Nagel 1993, Ito et al. 2010). Two other dichloroaniline isomers (3,5-DCA and 2,6-DCA) have been used as model toxic compounds in experiments with algae *Chlorella vulgaris* for the identification and assessment of novel toxic compounds (Xia et al. 2009).

Toxicity of DCA to mammals is also well known. Similar to other aromatic amines, DCA can induce an increase of blood methemoglobin content (Guilhermino et al. 1998), anaemia and kidney and liver injury, with greater *in vitro* effects on kidney cells than on liver cells (Lo et al, 1990, Valentovic et al. 1995 & 1997). The reproductive toxicity of 3,4-DCA has been confirmed in a study on the activities of testicular enzymes of male rats treated with this chemical (Zhang & Lin 2009).

There is limited information on the human toxicity of DCA. An in vitro investigation has shown that 3,4-DCA may interfere with the development of blood cells, by affecting erythroid colony formation (Malerba et al. 2002). Two DCA isomers (3,4-DCA and 3,5-DCA) have been detected in human urine in the general population in Bavaria, Germany, as metabolites of pesticides that were taken up with diet (Angerer et al. 2007).

Far higher total concentrations (dissolved forms plus those bound to suspended particulates) were found for most metals in the whole (unfiltered) sample from pipe 1 (CN10013). These metals were present almost exclusively (99% or more) in particulate-bound forms. This sample (CN10013) visually contained a very high level of suspended solids, which may be due to resuspension of sediment from within the discharge pipe into the wastewater as a result of the intermittent nature of the discharge via this pipe.

Surface water sediments that are not contaminated by anthropogenic inputs will naturally contain traces of the metals quantified in this study, typically at low concentrations. The presence of uncontaminated sediments in the wastewater sample (CN10013) can account for the total concentrations of most metals in this sample.

However, the total concentrations of chromium, copper, nickel and zinc in this sample are considerably higher than what could be accounted for in this way. As discussed above, the concentrations of dissolved forms of chromium, copper and nickel were also present at somewhat elevated levels. These data suggest that wastewater intermittently discharged from the Well Dyeing facility via pipe 1 is a source of chromium, copper and nickel, and possibly zinc to the receiving river system. These metals are known to have uses in the textile-manufacturing sector, and they can have toxic effects, particularly at high concentrations. More information on these metals is provided in Box F.

Box F. Metals

Chromium (Cr) is primarily used in the metallurgical industry (in stainless steel and other alloys), as well as in various industrial processes including leather tanning and certain textile processes (ATSDR 2008b, IPPC 2003). Hexavalent chromium compounds are used in metal finishing (chrome plating), and also in certain textile manufacturing processes, in wood preservatives and as corrosion inhibitors (ATSDR 2008b, IPPC 2003). Chromium normally exists in the environment in trivalent Cr(III) forms, which generally have very low solubility in water and tend to rapidly precipitate or adsorb onto suspended particles and bottom sediments. Hexavalent Cr(VI) forms can exist, though far less frequently, and these compounds are usually converted rapidly to trivalent Cr(III) compounds by reducing compounds. Hexavalent forms tend to be readily soluble in water and therefore can be highly mobile in aquatic environments (ATSDR 2008b, DeLaune et al. 1998, Lin 2002). Uncontaminated surface water typically contains less than 10 µg/l of chromium, and concentrations in uncontaminated freshwater sediments are typically below 100 mg/kg (ATSDR 2008b, Salomons & Forstner 1984). Chromium (III) is an essential nutrient for animals and plants, though large doses may be harmful. In contrast, hexavalent chromium is highly toxic even at low concentrations, including for many aquatic organisms (Baral et al. 2006). Hexavalent chromium compounds are also corrosive, and in humans allergic skin reactions readily occur following exposure, independent of dose (ATSDR 2008b). Furthermore, hexavalent chromium is a known human carcinogen under some circumstances (IARC 1990a). The Chinese national wastewater discharge standard and the equivalent Guangdong Province standard set the same maximum permissible concentrations of 1500 µg/l (1.5 mg/L) total chromium, and of 500 µg/l (0.5 mg/L) hexavalent chromium (Guangdong Province 2001, MEP 1998).

Copper (Cu) is a widely used metal, primarily as a pure metal or as part of mixtures (alloys) with other metals, though there are also many other uses of copper compounds, including within metal finishing processes and textile manufacture, including dyeing processes (IPPC 2003). The manufacture of plumbing materials is one of the main uses of main uses of copper metal and alloys, in part due to the malleability and thermal conductivity of copper (ATSDR 2004). Levels of copper in the environment are typically quite low, commonly less than 50 mg/kg in uncontaminated freshwater sediments (ATSDR 2004). Background concentration of dissolved copper in uncontaminated surface waters can vary significantly, but levels are typically below 10 µg/l, and often far lower (ATSDR 2004, Comber et al. 2008). Copper is an important element for humans and animals in low doses. However, expose to high levels of bio-available copper can lead to bioaccumulation and toxic effects (ATSDR 2004). Releases of copper to aquatic systems are of particular concern as many aquatic organisms are extremely sensitive to copper, particularly in soluble forms which are generally far more bio-available and toxic to a wide range of aquatic plants and animals (ATSDR 2004, Adams & Chapman 2006), with some effects occurring even at very low concentrations (Sandahl et al. 2007). The Chinese national wastewater discharge standard and the equivalent Guangdong Province standard set the same maximum permissible concentrations of copper of between 500 and 2000 µg/l (0.5 - 2.0 mg/L) depending on how the receiving water body is used (Guangdong Province 2001, MEP 1992, MEP 1998).

Nickel (Ni) as a metal and its alloys, as well as nickel compounds, have many industrial uses, including in metal plating, the manufacture of plumbing and electronic devices, in catalysts, batteries, pigments and ceramics (ATSDR 2005, DHHS 2005). Nickel is also used in certain textile dyes (eg phthalocyanine dyes), but to a lesser extent than other metals such as copper and chromium (IPPC 2003). Levels of nickel in the environment are typically low, with uncontaminated freshwater sediments generally containing below 60 mg/kg nickel. Concentrations in uncontaminated surface waters are typically below 20 µg/l (Alloway 1990, ATSDR 2005, Salomons & Forstner 1984). Although nickel bound to sediments and soils is generally persistent, water-soluble nickel compounds can be quite mobile. Very small amounts of nickel are essential for normal growth and reproduction in most animals and plants, and this is most likely also true for humans (ATSDR 2005). However, toxic and carcinogenic effects can result from exposure to higher concentrations for a wide range of life forms, including gastrointestinal and cardiac effects (ATSDR 2005, Cempel & Nikel 2006). In humans, a significant proportion of the population (2-5%) are also nickel sensitive, and effects can occur in sensitised individuals at far lower concentrations (ATSDR 2005). For some aquatic organisms, impacts can occur at very low nickel concentrations (Deleebeeck et al. 2008). Furthermore, some nickel compounds have been classified as carcinogenic to humans, and there is also evidence of carcinogenicity in animals (DHHS 2005, IARC 1990b). The Chinese national wastewater discharge standard and the equivalent Guangdong Province standard set the same maximum permissible concentrations of nickel of 1000 µg/l (1.0 mg/L) (Guangdong Province 2001, MEP 1998).

The third outfall to the Shiji River (pipe 3), located in the southwestern corner of the Well Dyeing complex, is semi-submerged in the river water, and therefore it is possible that this sample contained a mixture of wastewater and river water. A number of organic chemicals were identified in the sample collected from the end of this pipe (CN10007). Some of these were also identified in wastewater samples collected from pipe 1 and/or pipe 2, including two trialkyl phosphates (TBP and TEP) and methylquinoline, as discussed above. However, given the nature of this sample, the possibility that these chemicals originated from river water within this sample following their discharge to the river via pipe 1 and pipe 2 cannot be excluded.

Other chemicals identified in this wastewater sample (CN10007) were not present in other samples from this site, including four chlorobenzenes and a phthalate ester (diethyl phthalate, DEP). The di- and tri-chlorobenzenes were identified using a SIM method, indicating their presence in the sample at only trace levels. One of the dichlorobenzenes (1,4-dichlorobenzene) is a well-known environmental contaminant, largely due to its common use as a deodoriser and disinfectant in sanitary products, which may be the source of dichlorobenzenes in this sample. Tri-chlorobenzenes have many uses, including as dye carriers in the textile industry (WHO 2004).

It cannot be discounted that river water in CN10007 was the source of the chlorobenzenes and the phthalate in this sample, and that their presence is not due to releases from the Well Dyeing facility. However, phthalates and chlorinated benzenes are known to have uses in textile manufacturing.

Many of the substances described above were also identified in the wastewater samples collected from the eastern side of the facility adjacent to the Gaosha River, including the sample collected from the water hold tank that is sporadically discharged to the Gaosha River (CN10010), and that collected from pipe 4 (CN10015). As mentioned above, the origins of wastewater in the holding tank and wastewater discharged via pipe 4 are not known.

Substances identified in wastewater from the holding tank (CN10010) included trialkyl phosphate esters, alkylphenols, as well as methylquinoline and glycol ethers, substances described above.

One of the trialkyl phosphate esters (TEP) was also identified in all discharges to the Shiji River, as described above, though it should be emphasised that the origin of the water in the holding tank is not known. In addition, tri-iso-butyl phosphate (TiBP) and tris(2-butoxyethyl) phosphate (TBEP) were identified in CN10010. TiBP, a closely related chemical to TBP, has similar uses to TBP, including within textile manufacture. Uses of TBEP include as an antifoam agent and as a plasticiser. Trialkyl phosphate esters have been frequently reported in surface water environments (Reemtsma et al. 2008, Regnery & Puttmann 2010), and TiBP was recently reported in discharged wastewaters from textile manufacturing facilities in Thailand (Brigden et al. 2010b). More information on these tri-alkyl phosphates is presented in Box A.

This sample (CN10010) also contained octylphenol, a toxic alkylphenol that was also found in the wastewater from pipe 1, as described above, as well as octylphenol ethoxylate (OPE). The origin of water in the holding tank (CN10010) is not known. OPEs are used as detergents in numerous industrial processes, including during the manufacture of textile, and known to breakdown into octylphenol. Additional information on these substances is provided in Box C. Another phenol, bis(1-phenylethyl)phenol was also identified in this sample (CN10010), little information is available on this substance (see Box C).

The two glycol ethers identified in CN10010 included DPGME, a chemical also found in wastewater sample CN10013 (pipe 1), and tetraethylene glycol dimethyl ether (TEGDME), a substance with uses as a solvent in the production of certain dyes and inks (OECD 2001, VWA 2005). The limited data available indicates that TEGDME exhibits low to moderate acute toxicity (Ash 2004), though large gaps exist in the knowledge of the biological effects of this substance.

Other substances were identified only in this sample (CN10010), including quinone and ketone derivatives. The two quinones (anthraquinone (AQ) and an AQ derivative), and the ketone (naphthalic anhydride) have connections to textile manufacturing. Anthraquinone (AQ) is the parent substance for a large class of synthetic dyes, and the AQ derivative (amino-anthraquinone) is a common intermediate in the synthesis of a range of AQ dyes, many of which can degrade to release amino-anthraquinone (Novotny et al. 2006). The textile sector is thought to be a significant point source of discharges of AQ and derivative compounds to water. Many AQ derivatives, including amino-anthraquinone, are known to be toxic to animals and/or plants (Sendelbach 1989). More information on AQ and its derivatives is given in Box B.

Naphthalic anhydride, a di-ketone, is an important intermediate in the production of various dyestuffs, and optical brightening agents (Peters & Bide 1985, Xuhong et al. 1989, Grabtchev & Philipova 1995), though uses in herbicide formulations are also substantial (Abu-Qare & Duncan 2002). Following release, it is likely to degrade fairly rapidly to naphthalic acid (Abu-Qare & Duncan 2002). The toxicity of naphthalic anhydride to animals has not been fully investigated. More information is provided in Box B.

Substances identified in the wastewater sample collected by the outfall of the submerged pipe 4 (CN10015) included the trialkyl phosphate TBEP, octylphenol and octylphenol ethoxylates, quinolines and a glycol ether (DPGME). These substances were also identified in the sample from the holding tank (CN10010), as discussed above. It is possible that the presence of these substances in CN10015 originated from river water within this sample, following their discharge from the wastewater holding tank (CN10010) into the river.

Other substances were only identified in this sample (CN10015), and no other samples, including two quinones, two phenols and a phthalate. The two quinones (both benzoquinone derivatives) were 2,6-di-tert-butyl benzoquinone (DBQ), and a derivative of DBQ. Benzoquinone derivatives, including DBQ, can be used as photo initiators in various industrial processes (van der Graaf et al. 1991), and DBQ has been previously identified in wastewater discharged from a textile manufacturing facility (Brigden et al. 2009). More information is provided in Box B. The phenols, methyl phenol and 4,4'-methylenebis phenol (also known as bisphenol F), have uses within textile manufacture, though their source to this sample is not certain. More information on these is provided in Box C.

The phthalate, di(2-ethylhexyl) phthalate (DEHP), has uses in textile manufacture, though it also has numerous other uses, primarily as a plasticiser (or softener) in plastics. DEHP is one of the most widely used phthalates, and this toxic chemical is widely distributed in the environment, largely as a result of losses from products in which it had been incorporated (CSTEE 2001, Fromme et al. 2002). This generic environmental contamination may be the source of DEHP in this sample, which may contain a mixture of river water and wastewaste discharged via pipe 4.

This sample (CN10015) also contained chromium at a level above what is typically found in uncontaminated surface waters (total 277 µg/l, dissolved 183 µg/l). Background surface waters concentrations of dissolved chromium are typically below 10 µg/l, and often far lower (ATSDR 2008b). As for certain organic substances, it cannot be discounted that river water in this sample collected from a submerged pipe was the source of the chromium. The most toxic form of chromium (hexavalent chromium, Cr(VI)) was not detected in this, or any other sample.

In addition to the chemicals described above, certain substances were commonly identified in samples from this site in a way that indicates their presence is not related to manufacturing activities within the Well Dyeing complex.

For example, atrazine was detected at trace levels in three wastewater samples and also in the river water sample (CN10011) collected from the Shiji River. Atrazine is one of the most widely used herbicides for the control of weeds and grasses (US EPA 2009), and its presence at this site may be due to this use, and not due to use in textile manufacture within the facility. As a result of its environmental persistence and toxicity, atrazine is recognised as a substance requiring regulation in certain places outside China, including being listed as a priority substance under the Water Framework Directive of the European Union (EC 2001).

Furthermore, aliphatic hydrocarbons were detected in all wastewater and sediment samples associated with the Well Dyeing facility, together with alkyl benzenes, polycyclic aromatic hydrocarbon (PAHs), and a benzothiazole derivative in the wastewater samples collected from pipe 4 (CN10015) and from the adjacent wastewater holding tank (CN10010). The presence of these chemicals suggests contamination of the wastewater and river sediment samples by oil or related chemicals (Overton 1994). Oxygenated PAHs were also identified in the wastewater from the tank (CN10010), substances which can originate either from combustion sources (Sidhu et al. 2005), or from the degradation of PAHs (Wischmann & Steinhart 1997, Zhong et al. 2011). Oxygenated PAHs may be more toxic than the parent PAH compounds (Lundstedt et al. 2007, Walgraeve et al. 2010).

Other than these oil-related chemicals, the samples of sediment collected from the Shiji and Gaosha Rivers, adjacent to the outfall pipes, did not contain any of the organic chemicals identified in the various wastewater samples, nor any additional significant substances.

Metals within surface waters can accumulate in bottom sediments overtime, and therefore ongoing inputs of metals to a river system can lead to increasing levels of metals in bottom sediments. This accumulation can result in long-term impacts in sensitive aquatic species and, potentially, predators further up the food web. With certain exceptions, the concentrations of most of the quantified metals in the various river sediment samples were within the typical ranges reported for these metals in uncontaminated river sediments.

Copper, lead and zinc were, however, present in the samples of river sediment collected from both the Shiji and Gaosha Rivers at levels up to 2-3 times their typical background concentration ranges for these metals in river sediments. These levels indicate the accumulation of these metals as a result of ongoing inputs to the river system, which can include the discharge of wastewaters containing high concentrations of these metals. However, the levels of these metals were similar for all river sediment samples collected from various locations on both rivers. Furthermore, the sample of sediment collected from the Shiji River furthest from the discharge pipes (CN1012) contained the highest concentrations. In addition, although wastewater discharged via pipe 1 (CN10013) contained elevated levels of metals at the time of sampling, particularly chromium, copper and nickel, the sediment sample collected from the Shiji River adjacent to this pipe (CN10014) did not contain high levels of these metals. Rather, their concentrations in this sample were the lowest amongst all sediments collected from the Shiji River, other than for zinc.

These data suggest that metals discharged in wastewaters from Well Dyeing are not substantially contributing to the elevated sedimentary levels of certain metals in the Shiji and Gaosha Rivers. It is likely that the elevated sedimentary levels originate from other (so far unidentified) sources on the main Huangshali Channel, to which both these rivers are connected, due to the tidal flow of the river system in this area.

5. Conclusions

This study has demonstrated that a diverse range of industrial chemicals are discharged within wastewaters from the two (unrelated) textile manufacturing facilities investigated, with some similarities in the chemicals, or chemical groups, present in the wastewaters from the two facilities. Some of the chemicals identified are known to have hazardous properties. For others, little is known in terms of their toxicology or potential impacts following release to the environment. In addition, the majority of organic chemicals isolated from wastewater samples could not be reliably identified, and therefore their properties and potential impacts, whether alone or as components of complex mixture of contaminants, simply cannot be assessed. This study has also highlighted instances where the composition and volume of discharged wastewaters from an outfall can vary significantly at different times, or where active discharge only occurs sporadically. These situations raise difficulties and concerns regarding the monitoring of discharges from these and potentially other industrial facilities.

Many of the substances identified in the wastewater samples from outfalls discharging to the Pearl River and Yangtze River Deltas are soluble in water, enabling them to remain relatively mobile within the river systems to which they are released. Some are known to be highly persistent within aquatic environments and/or able to bioaccumulate within organisms. Ongoing releases of such substances are likely to lead to ever increasing levels in the receiving environment, which in some cases will not significantly decrease for long periods of time, even after controls on their release have been introduced.

Legislation that address the discharge of hazardous chemicals within wastewaters from the two facilities investigated, including the Guangdong Province effluent standard and the Chinese national wastewater discharge standard, do set controls for certain chemicals pollutants, including some identified within this study. These regulations set maximum permissible levels for listed substances rather than prohibiting their discharge. However, these regulations do not cover the discharge of the majority of substances identified in the wastewater samples that were analysed for this study.

Textile manufacturing is a global sector. Considerable differences currently exist between regulations applicable in different countries on the use and release of chemicals during manufacturing, particularly for hazardous chemicals. Some of the hazardous substances identified in this study, which are not subject to discharge regulation within China, are nonetheless specifically listed as priority substances in one or more regulations or conventions developed to protect the environment and/or human health in other regions. Amongst these, the alkylphenols (octyl and nonylphenol) are listed as priority substances under the European Union (EU) Water Framework Directive, a regulation designed to improve the quality of water within the EU (EU 2008). These have also been identified as substances for priority action under the OSPAR convention, under which signatory countries have agreed a target of cessation of discharges, emissions and losses of all hazardous substances to the marine environment of the North-East Atlantic by 2020 (OSPAR 1998). Despite the lack of regulation addressing their discharge within China, the import of nonylphenol and nonylphenol ethoxylates into China has very recently been regulated, though not prohibited (MEP 2011).

Clearly, this study did not attempt to investigate all sources of hazardous chemicals to the Pearl River Delta and Yangtze River Delta. However, the releases of hazardous chemicals within wastewaters from these two facilities may provide an illustration of what might also be occurring at other textile facilities, both within China and elsewhere. For example, a similar study recently demonstrated the discharge of hazardous chemicals in wastewaters discharged from textile manufacturing facilities in Thailand, including some of the same substances identified in this study (Brigden et al. 2010a).

There is an urgent need for more in-depth investigation into the discharge of hazardous chemicals from all sources to these river basins, as well as in other parts of China.

For many hazardous substances, however, the use of ‘end-of-pipe’ measures, including through the use of conventional wastewater treatment plants, cannot effectively address their presence in effluents or other waste streams. For example, many persistent organic pollutants will either pass through the treatment process unchanged, be converted into other hazardous substances, or accumulate in other wastes generated during the treatment process (such as sludges) which then become hazardous wastes in themselves.

What is required are solutions which seek alternatives to the use of such hazardous substances in manufacturing processes, progressively replacing them with less hazardous, and preferably non-hazardous, alternatives in order to bring about rapid reductions and ultimate cessation in their discharges, emissions and losses. Through systematic rethinking and redesigning products and processes, these changes can contribute to the building of a more sustainable industry and to eliminating both the waste of resources and the pervasive threats to the environment and human health which the ongoing use and release of hazardous chemicals entails.

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Appendix 1. Analytical methodology

Analysis for Volatile Organic Compounds (VOCs)

Method

VOCs were analysed using an Agilent 6890 gas chromatograph with an Rtx-624 column (30m, 0.25mm ID, 1.4µm film thickness) connected to an Agilent 7694 Headspace Sampler and linked to an Agilent 5973N MSD operated in EI mode. The GC oven temperature programme included an initial temperature of 35^{0}C (held for 4min), rising to 55^{0}C at 5^{0}C/min, and then to 210^{0}C at 15^{0}C/min. The carrier gas was helium, supplied at 1ml/min. From each sample, three 10ml portions were sub-sampled into 20ml headspace vials. One sub-sample was analysed with the GC-MS in total ion monitoring (SCAN) mode to identify as many of the volatile organic compounds present as possible. Identification of compounds was carried out by matching spectra against the Wiley7N Library, employing expert judgment in order to avoid misidentifications. The two remaining sub-samples were then used for duplicate quantitative analysis for those halogenated (chlorinated, brominated and mixed) VOCs which had been detected in the samples through screening. Quantification was performed in Selective Ion Monitoring (SIM) mode using a 5 point external calibration method. Halogenated VOCs quantified in the water samples with limits of detection and quantification are presented in Table 7.

Quality control

Standard deviation (SD), relative standard deviation (RSD) and limits of detection (LOD) were calculated using data from the analysis of seven replicates of a standard mixture of commonly occurring VOCs, containing 1ppb of each analyte. Limits of quantification (LOQ) were determined as the lowest concentration in the linear regression used for quantification. A number of blanks of laboratory air capped at the time that sub-sampling had taken place were also analysed, alongside samples of the ultra pure reagent water which was used for the preparation of standard calibration solutions. The initial calibration curve for each compound of interest was verified immediately prior to sample analysis by analysing a calibration standard at a concentration near the midpoint concentration for the calibration range of the GC-MS.

Compound	LOQ, µg/l	LOD, µg/l	SD, µg/l	RSD, %	r^2
Chloroform	0.3	0.3	0.05	4.5	0.999
Bromoform	0.5	0.4	0.06	6.6	0.999
Ethene, dichloro-	0.4	0.4	0.06	5.6	1.000
Ethene, 1,2-dichloro-, cis- *	1.0	1.0	0.20	10.1	0.999
Ethene, trichloro-	0.4	0.4	0.06	7.1	0.999
Ethene, tetrachloro-	0.3	0.3	0.05	5.1	0.999
Ethane, 1,1,1-trichloro-	0.3	0.3	0.05	4.3	0.999
Ethane, 1,2-dichloro-*	1.0	1.0	0.21	9.4	1.000
Ethane, 1,1,2,2-tetrachloro-	0.2	0.2	0.04	6.1	1.000
Methane, bromodichloro-*	0.5	0.5	0.09	3.7	0.997
Methane, dibromochloro-	0.5	0.2	0.03	3.9	0.999
Methane, dichloro-	0.2	0.2	0.03	4.3	0.999
Methane, tetrachloro-	0.3	0.3	0.05	3.9	0.999

Table 7. Standard deviation (SD), relative standard deviation (RSD) and limit of detection (LOD) were calculated using concentration of the seven replicas of the standard mixture containing 1ppb of each analyte. Limit of quantification (LOQ) is the lowest concentration in the linear regression (r^2 – corresponding correlation coefficient) used for quantification.
* - 2ppb of analyte was used.

Analysis for extractable organic compounds

Preparation

20 µg of deuterated naphthalene was added as an Internal Standard (IS) to each portion of sample that was subject to extraction. For sediment samples, approximately 10 g of each sample (wet weight) was extracted employing Accelerated Solvent Extraction (ASE) technique using Dionex ASE-350 with a mixture of pentane and acetone 3:1,

at a temperature of 100°C. Water samples (500ml) were prepared using solid phase extraction technique with Dionex AutoTrace workstation, eluting solvents were ethyl acetate followed by a mixture of pentane and toluene 95:5. Obtained extracts were concentrated to a volume of 3ml with a stream of clean nitrogen and cleaned up prior to analysis.

Clean-up procedures were the same for the crude extracts from both aqueous and sediment samples. Each extract was shaken with 3ml isopropyl alcohol and 3ml TBA-reagent (mixture of 3% tetrabutylammonium hydrogen sulphate and 20% sodium sulphite in deionised water) and left to stand until the aqueous and organic phases had separated. The pentane phase was collected and eluted through a Florisil column, using a 95:5 pentane:toluene mixed eluent, and the cleaned extract concentrated to a final volume of 1ml as before. 20 µg of bromonaphthalene was added to each extract as a second IS prior to GC-MS analysis.

Analysis

For the total organic compounds screening, samples were analysed using an Agilent 6890 Series II GC with Restek Rtx-XLB column (30m, 0.25mm ID, 0.25 µm film thickness) linked to an Agilent 5973 Inert MSD operated in EI mode and interfaced with an Agilent Enhanced Chem Station data system. The GC oven temperature programme employed was as follows: an initial temperature of 35°C, held for 2 minutes, raised to 260°C at 10°C/min, then to 320°C at 6°C/min (held for 8min). The carrier gas was helium, supplied at 1ml/min. Identification of compounds was carried out by matching spectra against both the Wiley 7N and Pesticides Libraries, using expert judgment as necessary in order to avoid misidentifications. Additionally, both the spectra and retention times of compounds isolated from the samples were matched against those obtained during GC-MS analysis of standard mixtures containing a range of chlorinated benzenes, phenols and pesticides, polychlorinated biphenyls (PCBs), phthalates, polycyclic aromatic hydrocarbons (PAHs) and aliphatic hydrocarbons.

Quality control

A number of extraction and solvent blanks were also analysed to ensure the detection of any possible contamination resulting from sample handling in the laboratory.

Analysis for metals

Preparation

A representative portion of each sediment sample was air dried to constant weight, homogenised, sieved through a 2mm mesh and then ground to a powder using a pestle and mortar. Approximately 0.5g of each sample was digested with 2 ml concentrated hydrochloric acid and 8 ml concentrated nitric acid, firstly overnight at room temperature, then using microwave-assisted digestion with a CEM MARS Xpress system, with a temperature ramp to 180°C over 15 minutes followed by holding at 180°C for a further 15 minutes. Following cooling, digests were filtered and made up to 50ml with deionised water.

For water samples, to obtain total concentrations, a representative portion of each whole sample was acidified by the addition of concentrated nitric acid to give a final concentration of 10% v/v. In addition, a portion of each whole sample was filtered and then acidified in the same way. 50 ml of each acidified sample was digested firstly overnight at room temperature, then using microwave-assisted digestion with a CEM MARS Xpress system, with a temperature ramp to 180°C over 15 minutes followed by holding at 180°C for a further 15 minutes. Cooled digests were filtered and made up to 50 ml with deionised water.

Analysis

Prepared sample digests were analysed by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) using a Varian MPX Simultaneous Spectrometer. Multi-element standards at concentrations of 0.5, 1.0, 2.5 and 10 mg/l respectively, and matrix matched to the samples, were used for instrument calibration. Any sample exceeding the calibration range was diluted accordingly, in duplicate, and re-analysed. Analysis of the mercury content in the samples was carried out separately. Mercury (Hg) was determined using cold vapour generation ICP-AES. Ionic mercury, Hg (II), was reduced to elemental mercury, Hg (0), through reaction of the sample with sodium borohydride (0.6% w/v), sodium hydroxide (0.5% w/v) and hydrochloric acid (10 molar). The elemental mercury vapour was carried in a stream of argon into the spectrometer. Two calibration standards were prepared, at 10 µg/l and 100 µg/l, matrix matched to the samples.

Quality control

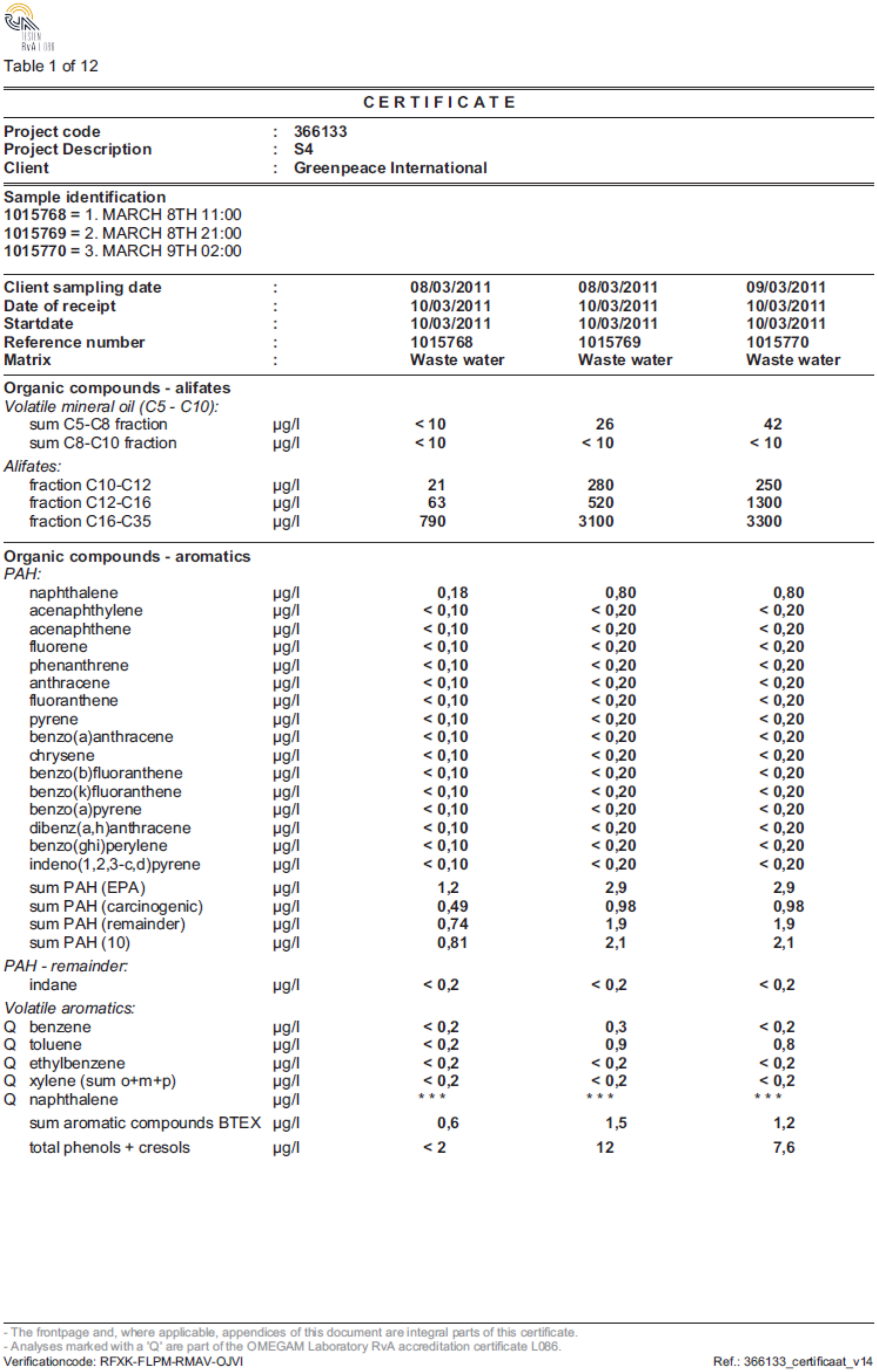
For sediment samples, three samples were prepared in duplicate and analysed to verify method reproducibility, along with an identically prepared blank. To check the method efficiency, certified reference material (CRM) samples were prepared in an identical manner; GBW07311, stream sediment certified by the China National Analysis Centre for Iron and Steel, Beijing, China, and LGC6187, leachable metals in river sediment certified by the Laboratory of the Government Chemist, UK.

For water samples, four samples were prepared in duplicate and analysed to verify method reproducibility, along with a blank sample (10% v/v nitric acid in deionised water), and a mixed metal quality control solution of 4 mg/ l, other than mercury at 80 µg/l. All control samples were prepared in an identical manor to the samples.

Calibration of the ICP-AES was validated by the use of quality control standards at 8 mg/l and 0.8 mg/l (sediment samples) or 4 mg/l and 0.4 mg/l (water samples) prepared in an identical manner but from different reagent stocks to the instrument calibration standards. For cold vapour generation mercury analysis, the calibration was validated using two quality control standards (10 µg/l and 80 µg/l), prepared internally from different reagent stock.

Further details of the methods employed can be provided on request.

Appendix 2. Details of analyses conducted by the independent laboratory



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CERTIFICATE

Project code : **366133**
Project Description : **S4**
Client : **Greenpeace International**

Sample identification
1015768 = 1. MARCH 8TH 11:00
1015769 = 2. MARCH 8TH 21:00
1015770 = 3. MARCH 9TH 02:00

Client sampling date	:	**08/03/2011**	**08/03/2011**	**09/03/2011**
Date of receipt	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Startdate	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Reference number	:	**1015768**	**1015769**	**1015770**
Matrix	:	**Waste water**	**Waste water**	**Waste water**
Organic compounds - alifates				
Volatile mineral oil (C5 - C10):				
sum C5-C8 fraction	µg/l	**< 10**	**26**	**42**
sum C8-C10 fraction	µg/l	**< 10**	**< 10**	**< 10**
Alifates:				
fraction C10-C12	µg/l	**21**	**280**	**250**
fraction C12-C16	µg/l	**63**	**520**	**1300**
fraction C16-C35	µg/l	**790**	**3100**	**3300**
Organic compounds - aromatics				
PAH:				
naphthalene	µg/l	**0,18**	**0,80**	**0,80**
acenaphthylene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
acenaphthene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
fluorene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
phenanthrene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
anthracene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
fluoranthene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
pyrene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
benzo(a)anthracene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
chrysene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
benzo(b)fluoranthene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
benzo(k)fluoranthene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
benzo(a)pyrene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
dibenz(a,h)anthracene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
benzo(ghi)perylene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
indeno(1,2,3-c,d)pyrene	µg/l	**< 0,10**	**< 0,20**	**< 0,20**
sum PAH (EPA)	µg/l	**1,2**	**2,9**	**2,9**
sum PAH (carcinogenic)	µg/l	**0,49**	**0,98**	**0,98**
sum PAH (remainder)	µg/l	**0,74**	**1,9**	**1,9**
sum PAH (10)	µg/l	**0,81**	**2,1**	**2,1**
PAH - remainder:				
indane	µg/l	**< 0,2**	**< 0,2**	**< 0,2**
Volatile aromatics:				
Q benzene	µg/l	**< 0,2**	**0,3**	**< 0,2**
Q toluene	µg/l	**< 0,2**	**0,9**	**0,8**
Q ethylbenzene	µg/l	**< 0,2**	**< 0,2**	**< 0,2**
Q xylene (sum o+m+p)	µg/l	**< 0,2**	**< 0,2**	**< 0,2**
Q naphthalene	µg/l	* * *	* * *	* * *
sum aromatic compounds BTEX	µg/l	**0,6**	**1,5**	**1,2**
total phenols + cresols	µg/l	**< 2**	**12**	**7,6**

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- Analyses marked with a 'Q' are part of the OMEGAM Laboratory RvA accreditation certificate L086.

Verificationcode: RFXK-FLPM-RMAV-OJVI

Ref.: 366133_certificaat_v14

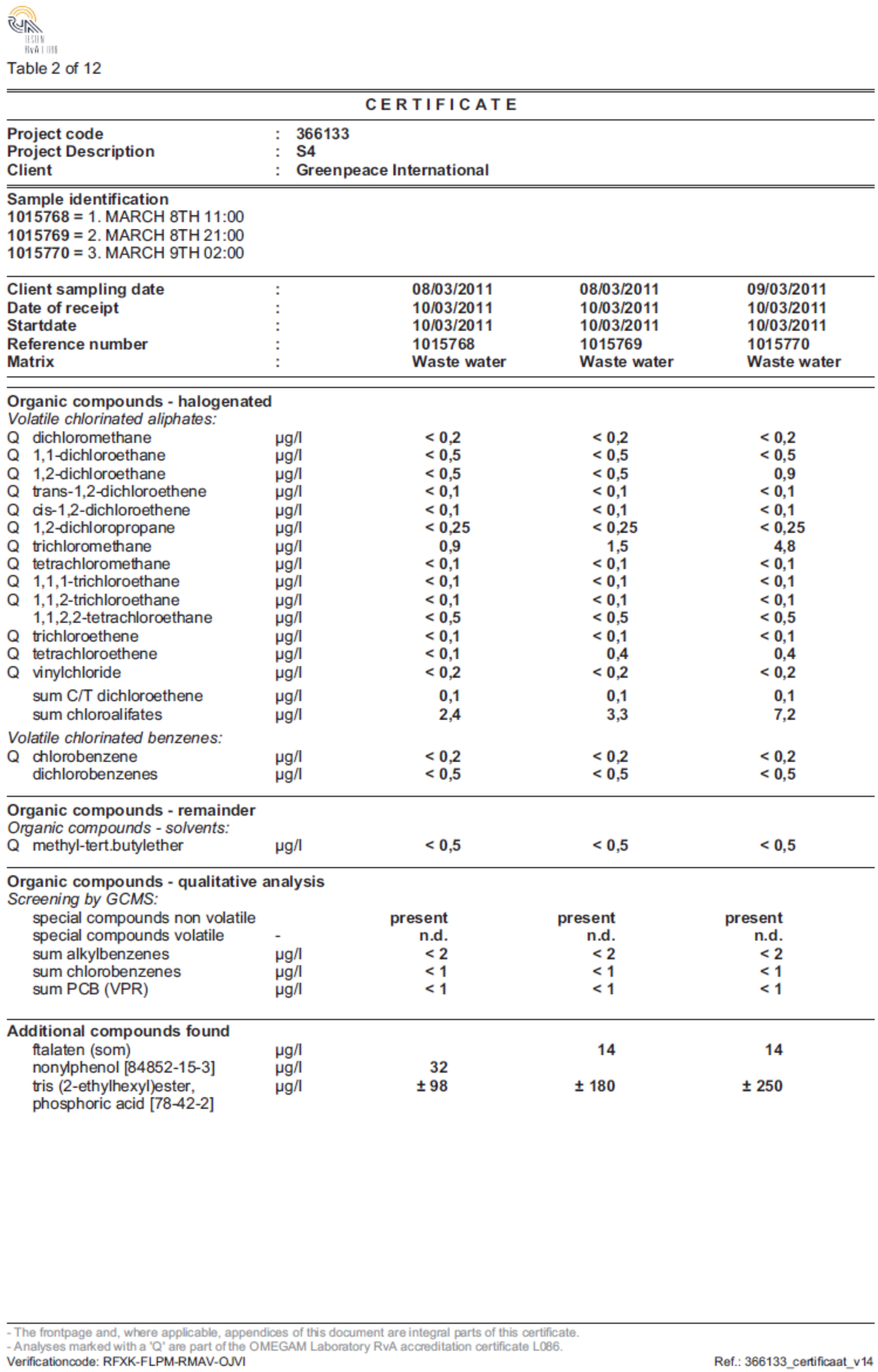

Table 2 of 12

CERTIFICATE

Project code : **366133**
Project Description : **S4**
Client : **Greenpeace International**

Sample identification
1015768 = 1. MARCH 8TH 11:00
1015769 = 2. MARCH 8TH 21:00
1015770 = 3. MARCH 9TH 02:00

Client sampling date	:	**08/03/2011**	**08/03/2011**	**09/03/2011**
Date of receipt	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Startdate	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Reference number	:	**1015768**	**1015769**	**1015770**
Matrix	:	**Waste water**	**Waste water**	**Waste water**

	Organic compounds - halogenated				
	Volatile chlorinated aliphates:				
Q	dichloromethane	µg/l	**< 0,2**	**< 0,2**	**< 0,2**
Q	1,1-dichloroethane	µg/l	**< 0,5**	**< 0,5**	**< 0,5**
Q	1,2-dichloroethane	µg/l	**< 0,5**	**< 0,5**	**0,9**
Q	trans-1,2-dichloroethene	µg/l	**< 0,1**	**< 0,1**	**< 0,1**
Q	cis-1,2-dichloroethene	µg/l	**< 0,1**	**< 0,1**	**< 0,1**
Q	1,2-dichloropropane	µg/l	**< 0,25**	**< 0,25**	**< 0,25**
Q	trichloromethane	µg/l	**0,9**	**1,5**	**4,8**
Q	tetrachloromethane	µg/l	**< 0,1**	**< 0,1**	**< 0,1**
Q	1,1,1-trichloroethane	µg/l	**< 0,1**	**< 0,1**	**< 0,1**
Q	1,1,2-trichloroethane	µg/l	**< 0,1**	**< 0,1**	**< 0,1**
	1,1,2,2-tetrachloroethane	µg/l	**< 0,5**	**< 0,5**	**< 0,5**
Q	trichloroethene	µg/l	**< 0,1**	**< 0,1**	**< 0,1**
Q	tetrachloroethene	µg/l	**< 0,1**	**0,4**	**0,4**
Q	vinylchloride	µg/l	**< 0,2**	**< 0,2**	**< 0,2**
	sum C/T dichloroethene	µg/l	**0,1**	**0,1**	**0,1**
	sum chloroalifates	µg/l	**2,4**	**3,3**	**7,2**
	Volatile chlorinated benzenes:				
Q	chlorobenzene	µg/l	**< 0,2**	**< 0,2**	**< 0,2**
	dichlorobenzenes	µg/l	**< 0,5**	**< 0,5**	**< 0,5**
	Organic compounds - remainder				
	Organic compounds - solvents:				
Q	methyl-tert.butylether	µg/l	**< 0,5**	**< 0,5**	**< 0,5**
	Organic compounds - qualitative analysis				
	Screening by GCMS:				
	special compounds non volatile		**present**	**present**	**present**
	special compounds volatile	-	**n.d.**	**n.d.**	**n.d.**
	sum alkylbenzenes	µg/l	**< 2**	**< 2**	**< 2**
	sum chlorobenzenes	µg/l	**< 1**	**< 1**	**< 1**
	sum PCB (VPR)	µg/l	**< 1**	**< 1**	**< 1**
	Additional compounds found				
	ftalaten (som)	µg/l		**14**	**14**
	nonylphenol [84852-15-3]	µg/l	**32**		
	tris (2-ethylhexyl)ester, phosphoric acid [78-42-2]	µg/l	**± 98**	**± 180**	**± 250**

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Verificationcode: RFXK-FLPM-RMAV-OJVI

Ref.: 366133_certificaat_v14

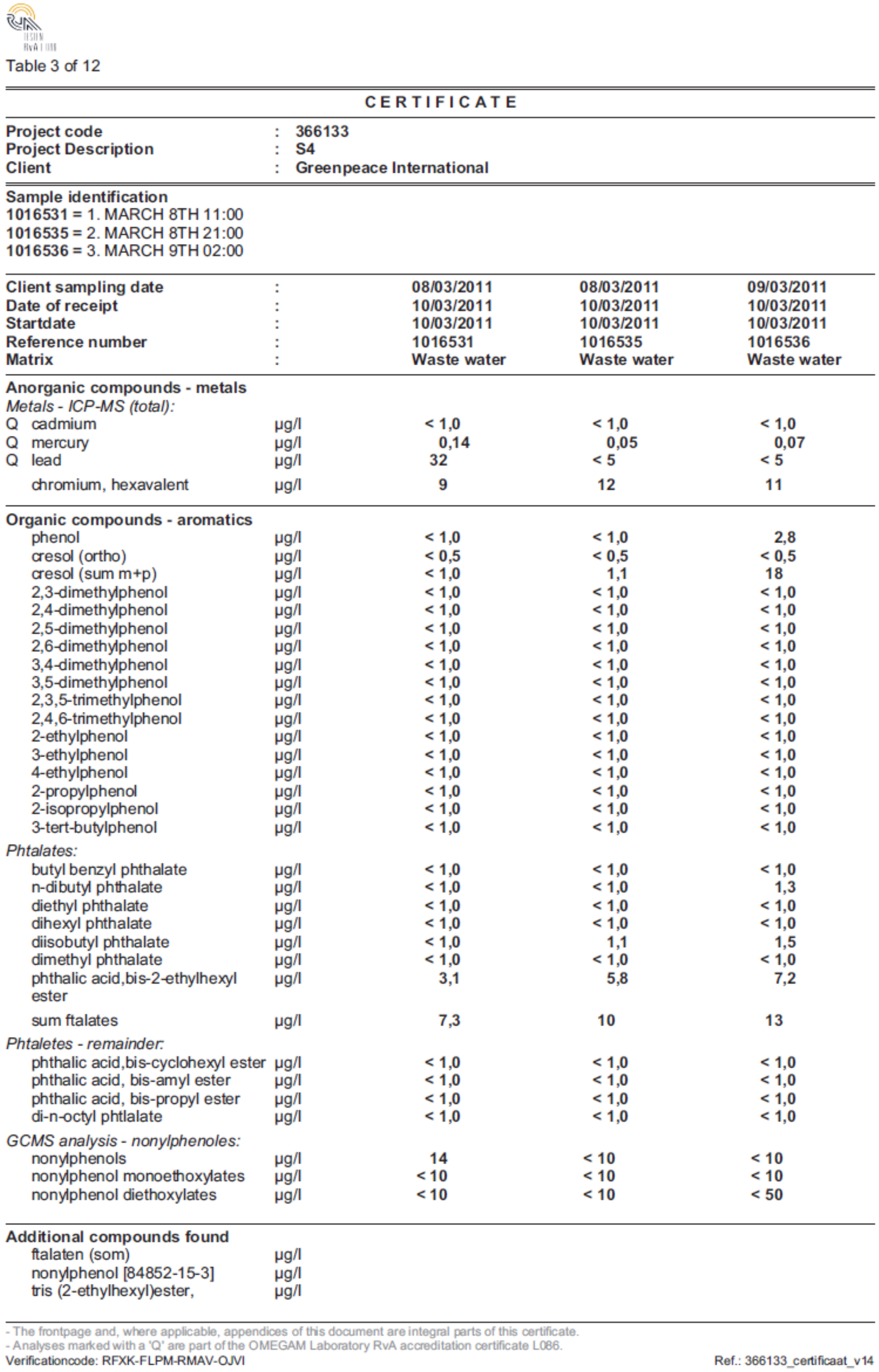

Table 3 of 12

CERTIFICATE

Project code	:	**366133**
Project Description	:	**S4**
Client	:	**Greenpeace International**

Sample identification
1016531 = 1. MARCH 8TH 11:00
1016535 = 2. MARCH 8TH 21:00
1016536 = 3. MARCH 9TH 02:00

Client sampling date	:	**08/03/2011**	**08/03/2011**	**09/03/2011**
Date of receipt	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Startdate	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Reference number	:	**1016531**	**1016535**	**1016536**
Matrix	:	**Waste water**	**Waste water**	**Waste water**

Anorganic compounds - metals				
Metals - ICP-MS (total):				
Q cadmium	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
Q mercury	µg/l	**0,14**	**0,05**	**0,07**
Q lead	µg/l	**32**	**< 5**	**< 5**
chromium, hexavalent	µg/l	**9**	**12**	**11**
Organic compounds - aromatics				
phenol	µg/l	**< 1,0**	**< 1,0**	**2,8**
cresol (ortho)	µg/l	**< 0,5**	**< 0,5**	**< 0,5**
cresol (sum m+p)	µg/l	**< 1,0**	**1,1**	**18**
2,3-dimethylphenol	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
2,4-dimethylphenol	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
2,5-dimethylphenol	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
2,6-dimethylphenol	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
3,4-dimethylphenol	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
3,5-dimethylphenol	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
2,3,5-trimethylphenol	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
2,4,6-trimethylphenol	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
2-ethylphenol	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
3-ethylphenol	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
4-ethylphenol	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
2-propylphenol	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
2-isopropylphenol	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
3-tert-butylphenol	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
Phtalates:				
butyl benzyl phthalate	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
n-dibutyl phthalate	µg/l	**< 1,0**	**< 1,0**	**1,3**
diethyl phthalate	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
dihexyl phthalate	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
diisobutyl phthalate	µg/l	**< 1,0**	**1,1**	**1,5**
dimethyl phthalate	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
phthalic acid,bis-2-ethylhexyl ester	µg/l	**3,1**	**5,8**	**7,2**
sum ftalates	µg/l	**7,3**	**10**	**13**
Phtaletes - remainder:				
phthalic acid,bis-cyclohexyl ester	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
phthalic acid, bis-amyl ester	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
phthalic acid, bis-propyl ester	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
di-n-octyl phtlalate	µg/l	**< 1,0**	**< 1,0**	**< 1,0**
GCMS analysis - nonylphenoles:				
nonylphenols	µg/l	**14**	**< 10**	**< 10**
nonylphenol monoethoxylates	µg/l	**< 10**	**< 10**	**< 10**
nonylphenol diethoxylates	µg/l	**< 10**	**< 10**	**< 50**
Additional compounds found				
ftalaten (som)	µg/l			
nonylphenol [84852-15-3]	µg/l			
tris (2-ethylhexyl)ester,	µg/l			

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- Analyses marked with a 'Q' are part of the OMEGAM Laboratory RvA accreditation certificate L086.

Verificationcode: RFXK-FLPM-RMAV-OJVI

Ref.: 366133_certificaat_v14

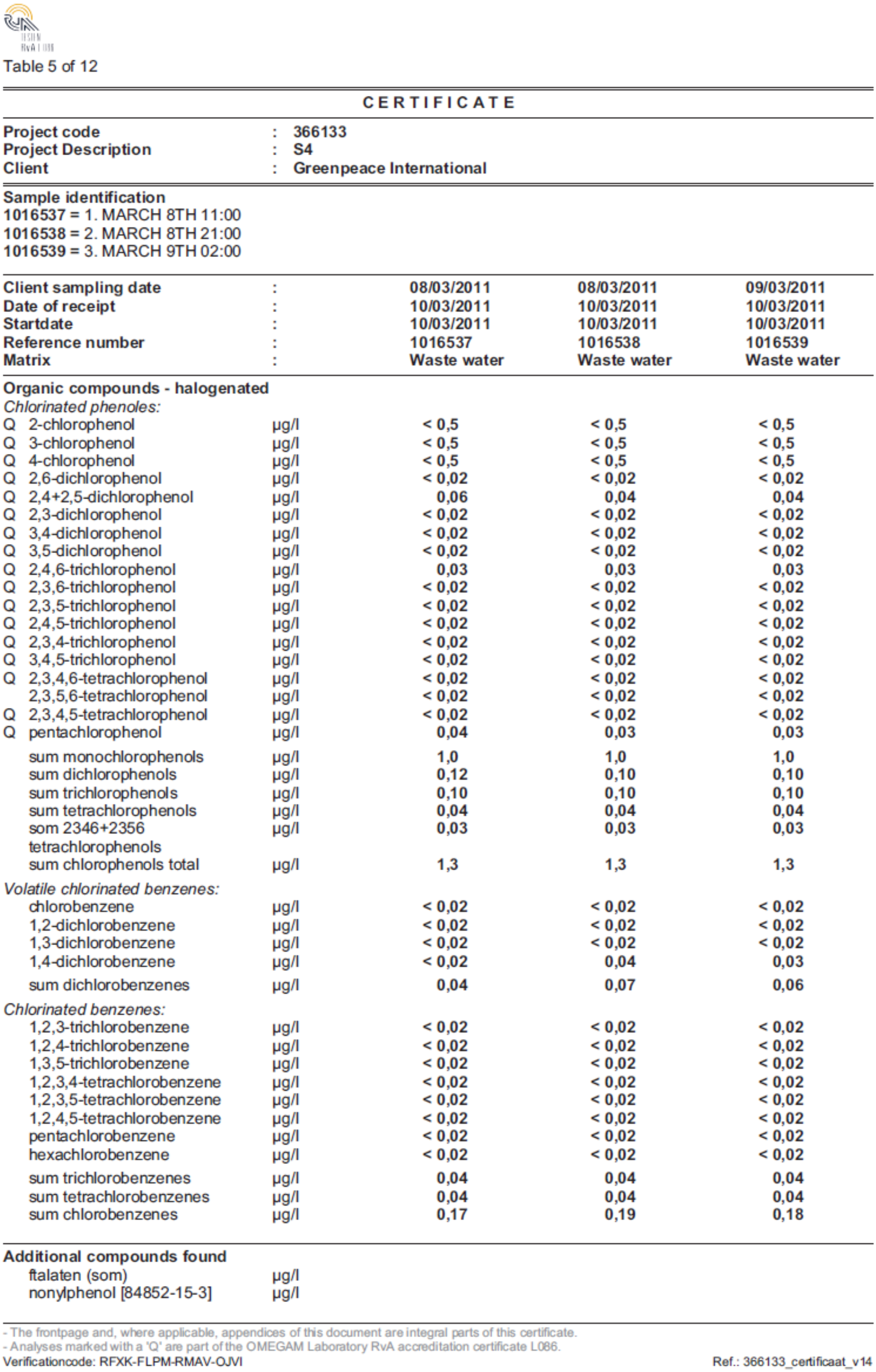
CERTIFICATE

Project code : **366133**
Project Description : **S4**
Client : **Greenpeace International**

Sample identification
1016531 = 1. MARCH 8TH 11:00
1016535 = 2. MARCH 8TH 21:00
1016536 = 3. MARCH 9TH 02:00

Client sampling date	:	**08/03/2011**	**08/03/2011**	**09/03/2011**
Date of receipt	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Startdate	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Reference number	:	**1016531**	**1016535**	**1016536**
Matrix	:	**Waste water**	**Waste water**	**Waste water**

phosphoric acid [78-42-2]



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CERTIFICATE

Project code : **366133**
Project Description : **S4**
Client : **Greenpeace International**

Sample identification
1016537 = 1. MARCH 8TH 11:00
1016538 = 2. MARCH 8TH 21:00
1016539 = 3. MARCH 9TH 02:00

Client sampling date	:	**08/03/2011**	**08/03/2011**	**09/03/2011**
Date of receipt	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Startdate	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Reference number	:	**1016537**	**1016538**	**1016539**
Matrix	:	**Waste water**	**Waste water**	**Waste water**
Organic compounds - halogenated				
Chlorinated phenoles:				
Q 2-chlorophenol	µg/l	**< 0,5**	**< 0,5**	**< 0,5**
Q 3-chlorophenol	µg/l	**< 0,5**	**< 0,5**	**< 0,5**
Q 4-chlorophenol	µg/l	**< 0,5**	**< 0,5**	**< 0,5**
Q 2,6-dichlorophenol	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
Q 2,4+2,5-dichlorophenol	µg/l	**0,06**	**0,04**	**0,04**
Q 2,3-dichlorophenol	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
Q 3,4-dichlorophenol	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
Q 3,5-dichlorophenol	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
Q 2,4,6-trichlorophenol	µg/l	**0,03**	**0,03**	**0,03**
Q 2,3,6-trichlorophenol	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
Q 2,3,5-trichlorophenol	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
Q 2,4,5-trichlorophenol	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
Q 2,3,4-trichlorophenol	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
Q 3,4,5-trichlorophenol	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
Q 2,3,4,6-tetrachlorophenol	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
2,3,5,6-tetrachlorophenol	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
Q 2,3,4,5-tetrachlorophenol	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
Q pentachlorophenol	µg/l	**0,04**	**0,03**	**0,03**
sum monochlorophenols	µg/l	**1,0**	**1,0**	**1,0**
sum dichlorophenols	µg/l	**0,12**	**0,10**	**0,10**
sum trichlorophenols	µg/l	**0,10**	**0,10**	**0,10**
sum tetrachlorophenols	µg/l	**0,04**	**0,04**	**0,04**
som 2346+2356 tetrachlorophenols	µg/l	**0,03**	**0,03**	**0,03**
sum chlorophenols total	µg/l	**1,3**	**1,3**	**1,3**
Volatile chlorinated benzenes:				
chlorobenzene	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
1,2-dichlorobenzene	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
1,3-dichlorobenzene	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
1,4-dichlorobenzene	µg/l	**< 0,02**	**0,04**	**0,03**
sum dichlorobenzenes	µg/l	**0,04**	**0,07**	**0,06**
Chlorinated benzenes:				
1,2,3-trichlorobenzene	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
1,2,4-trichlorobenzene	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
1,3,5-trichlorobenzene	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
1,2,3,4-tetrachlorobenzene	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
1,2,3,5-tetrachlorobenzene	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
1,2,4,5-tetrachlorobenzene	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
pentachlorobenzene	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
hexachlorobenzene	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
sum trichlorobenzenes	µg/l	**0,04**	**0,04**	**0,04**
sum tetrachlorobenzenes	µg/l	**0,04**	**0,04**	**0,04**
sum chlorobenzenes	µg/l	**0,17**	**0,19**	**0,18**
Additional compounds found				
ftalaten (som)	µg/l			
nonylphenol [84852-15-3]	µg/l			

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- Analyses marked with a 'Q' are part of the OMEGAM Laboratory RvA accreditation certificate L086.

Verificationcode: RFXK-FLPM-RMAV-OJVI

Ref.: 366133_certificaat_v14

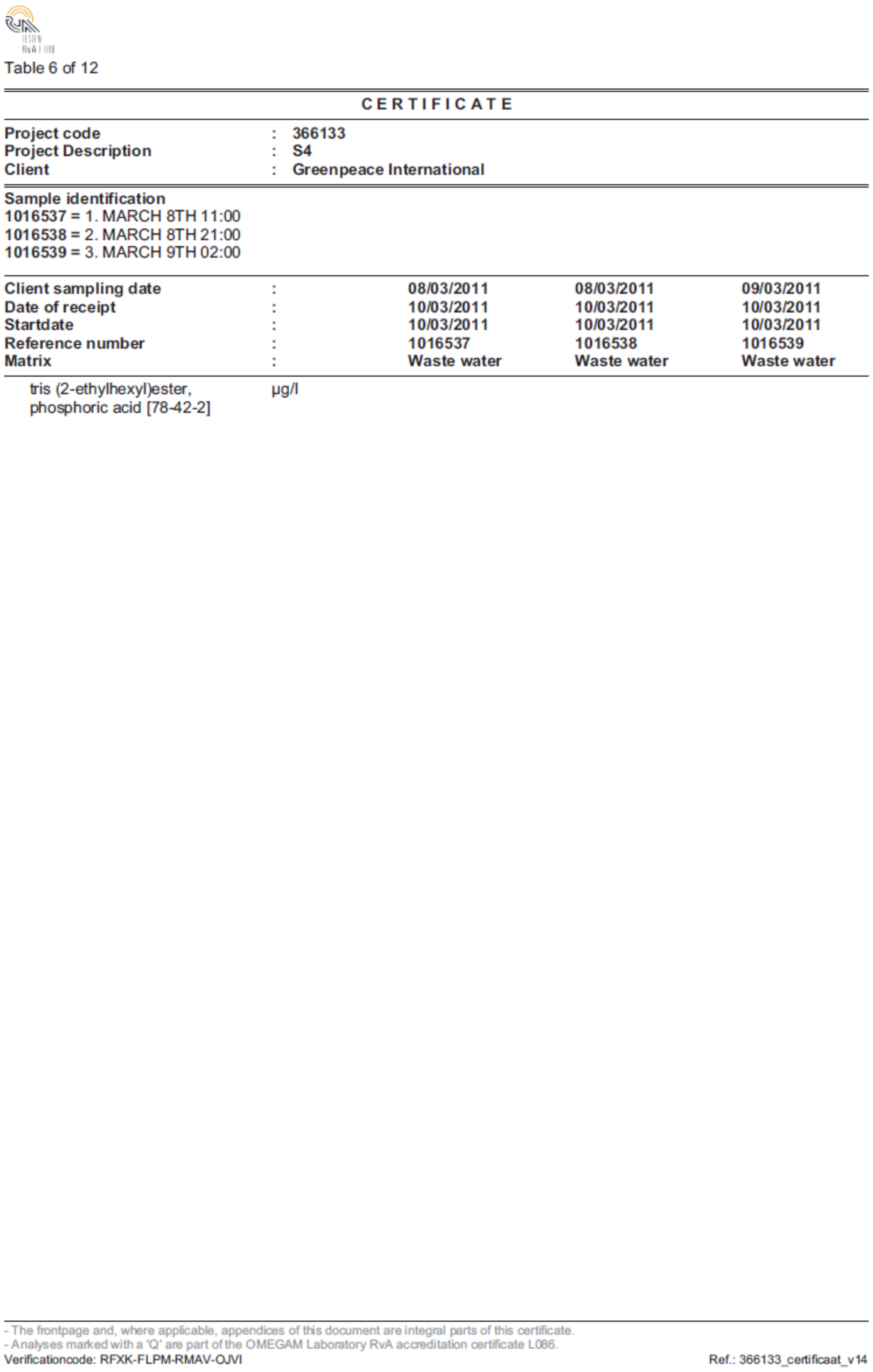

Table 6 of 12

CERTIFICATE

Project code : **366133**
Project Description : **S4**
Client : **Greenpeace International**

Sample identification
1016537 = 1. MARCH 8TH 11:00
1016538 = 2. MARCH 8TH 21:00
1016539 = 3. MARCH 9TH 02:00

Client sampling date	:	**08/03/2011**	**08/03/2011**	**09/03/2011**
Date of receipt	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Startdate	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Reference number	:	**1016537**	**1016538**	**1016539**
Matrix	:	**Waste water**	**Waste water**	**Waste water**
tris (2-ethylhexyl)ester, phosphoric acid [78-42-2]	µg/l			

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Verificationcode: RFXK-FLPM-RMAV-OJVI

Ref.: 366133_certificaat_v14

Table 7 of 12

CERTIFICATE

Project code	:	**366133**
Project Description	:	**S4**
Client	:	**Greenpeace International**

Sample identification
1016540 = 1. MARCH 8TH 11:00
1016543 = 2. MARCH 8TH 21:00
1016544 = 3. MARCH 9TH 02:00

Client sampling date	:	**08/03/2011**	**08/03/2011**	**09/03/2011**
Date of receipt	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Startdate	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Reference number	:	**1016540**	**1016543**	**1016544**
Matrix	:	**Waste water**	**Waste water**	**Waste water**

Organic compounds - aromatics

GCMS analysis - amines:

Q	2-chloroaniline	µg/l	**0,10**	**1,1**	**1,0**
Q	3-chloroaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	4-chloroaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	23-dichlooraniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	24-dichloroaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	25-dichloroaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	26-dichloroaniline	µg/l	**< 0,05**	**< 0,1**	**< 0,1**
Q	34-dichloroaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	35-dichloroaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	234-trichloroaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,20**
Q	245-trichlooraniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	246-trichlooraniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	345-trichlooraniline	µg/l	**< 0,05**	**< 0,2**	**< 0,2**
Q	2356-tetrachloroaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	pentachlooraniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
	3-chloro-4-methoxyaniline	µg/l	**< 0,05**	**< 0,2**	**< 0,2**
Q	3-chloro-4-methylaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	4/5-chloro-2-methylaniline (sum)	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	26-dichloro-4-nitroaniline	µg/l	**< 0,05**	**< 0,1**	**< 0,05**
Q	2-nitroaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	3-nitroaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	4-methyl-2-nitroaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	4-methyl-3-nitroaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	4-methoxy-2-nitroaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	aniline	µg/l	**1,9**	**1,8**	**2,1**
Q	n-methylaniline	µg/l	**0,12**	**0,1**	**0,13**
Q	n.n-dimethylaniline	µg/l	**< 0,1**	**< 2**	**< 2**
Q	n-ethylaniline	µg/l	**0,18**	**0,52**	**0,66**
Q	n.n-diethylaniline	µg/l	**0,47**	**0,87**	**0,91**
Q	23-dimethylaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	24/25-dimethylaniline (sum)	µg/l	**< 0,1**	**< 0,1**	**< 0,1**
Q	26-dimethylaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	34-dimethylaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	35-dimethylaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	26-diethylaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	246-trimethylaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	4-isopropylaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	2-fenylsulfonaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
	n.n-dimethylbenzylamine	µg/l	**< 0,3**	**< 0,3**	**< 0,3**
Q	benzylamine	µg/l	**< 1**	**< 1**	**< 1**
Q	o/p/m-toluidine (som)	µg/l	**< 0,2**	**< 1**	**< 1**
Q	o-anisidine	µg/l	**0,07**	**0,08**	**0,08**
Q	4-bromoaniline	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
	1-naftylamine	µg/l	**< 0,1**	**< 0,2**	**< 0,5**
	2-naftylamine	µg/l	**< 0,1**	**< 0,5**	**< 1**
Q	dibenzylamine	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
Q	tribenzylamine	µg/l	**< 0,05**	**< 0,05**	**< 0,05**
	p-benzidine	µg/l	**< 1**	**< 1**	**< 1**
Q	33-dichlorobenzydine	µg/l	**< 0,05**	**< 0,05**	**< 0,05**

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Verificationcode: RFXK-FLPM-RMAV-OJVI

Ref.: 366133_certificaat_v14

Table 8 of 12

CERTIFICATE

Project code : **366133**
Project Description : **S4**
Client : **Greenpeace International**

Sample identification
1016540 = 1. MARCH 8TH 11:00
1016543 = 2. MARCH 8TH 21:00
1016544 = 3. MARCH 9TH 02:00

Client sampling date	:	**08/03/2011**	**08/03/2011**	**09/03/2011**
Date of receipt	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Startdate	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Reference number	:	**1016540**	**1016543**	**1016544**
Matrix	:	**Waste water**	**Waste water**	**Waste water**
monochloroanilines (sum)	µg/l	**0,17**	**1,2**	**1,1**
Organic compounds - halogenated				
Flame retardants:				
BDE-028	µg/l	**< 0,0005**	**< 0,0005**	**< 0,0005**
BDE-047	µg/l	**< 0,0005**	**< 0,0005**	**< 0,0005**
BDE-049	µg/l	**< 0,0005**	**< 0,0005**	**< 0,0005**
BDE-085	µg/l	**< 0,0005**	**< 0,0005**	**< 0,0005**
BDE-099	µg/l	**< 0,0005**	**< 0,04**	**< 0,0005**
BDE-100	µg/l	**< 0,0005**	**< 0,0005**	**< 0,0005**
BDE-138	µg/l	**< 0,0005**	**< 0,0005**	**< 0,0005**
BDE-153	µg/l	**< 0,0005**	**< 0,0005**	**< 0,0005**
BDE-154	µg/l	**< 0,0005**	**< 0,0005**	**< 0,0005**
Organic compounds - pesticides				
GCMS - organic tin pesticides:				
Q dibutyltin	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
dicyclohexyltin	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
diphenyltin	µg/l	**< 0,02**	**< 0,02**	**< 0,02**
Q tetrabutyltin	µg/l	**< 0,01**	**< 0,01**	**< 0,01**
Q tributyltin	µg/l	**< 0,01**	**< 0,01**	**< 0,01**
Q tricyclohexyltin	µg/l	**< 0,01**	**< 0,01**	**< 0,01**
Q triphenyltin	µg/l	**< 0,01**	**< 0,01**	**< 0,01**
Additional compounds found				
ftalaten (som)	µg/l			
nonylphenol [84852-15-3]	µg/l			
tris (2-ethylhexyl)ester, phosphoric acid [78-42-2]	µg/l			

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Verificationcode: RFXK-FLPM-RMAV-OJVI

Ref.: 366133_certificaat_v14

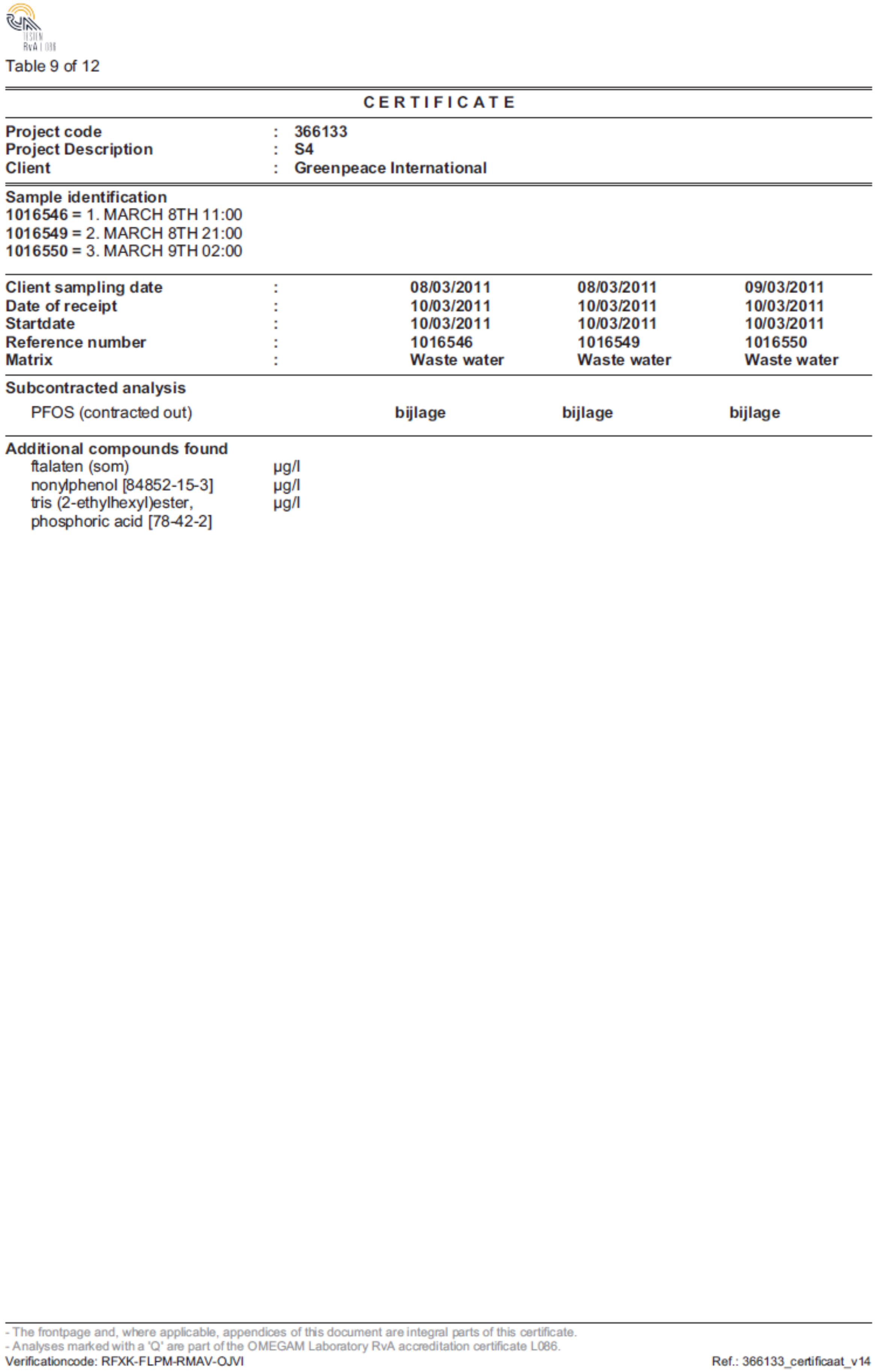

Table 9 of 12

CERTIFICATE

Project code : **366133**
Project Description : **S4**
Client : **Greenpeace International**

Sample identification
1016546 = 1. MARCH 8TH 11:00
1016549 = 2. MARCH 8TH 21:00
1016550 = 3. MARCH 9TH 02:00

Client sampling date	:	**08/03/2011**	**08/03/2011**	**09/03/2011**
Date of receipt	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Startdate	:	**10/03/2011**	**10/03/2011**	**10/03/2011**
Reference number	:	**1016546**	**1016549**	**1016550**
Matrix	:	**Waste water**	**Waste water**	**Waste water**
Subcontracted analysis				
PFOS (contracted out)		**bijlage**	**bijlage**	**bijlage**
Additional compounds found				
ftalaten (som)	µg/l			
nonylphenol [84852-15-3]	µg/l			
tris (2-ethylhexyl)ester, phosphoric acid [78-42-2]	µg/l			

- The frontpage and, where applicable, appendices of this document are integral parts of this certificate.
- Analyses marked with a 'Q' are part of the OMEGAM Laboratory RvA accreditation certificate L086.
Verificationcode: RFXK-FLPM-RMAV-OJVI

Ref.: 366133_certificaat_v14

CERTIFICATE

Project code : **366133**
Project Description : **S4**
Client : **Greenpeace International**

Notes related to analyses

General comments

* * * Betekent dat de verbinding met twee verschillende methoden is geanalyseerd. Ten aanzien van deze verbinding is een voorkeursrapportage ingesteld. Het gerapporteerde resultaat heeft de voorkeur boven het van * * * voorziene resultaat.

Sample identification : **2. MARCH 8TH 21:00**
Reference number : **1015769**

Results Note(s):
acenafteen: - Raised reporting limit because of interference by the matrix.
benzo(ghi)peryleen: - Raised reporting limit because of interference by the matrix.
som PAK (overige): - Raised reporting limit because of interference by the matrix.
fenantreen: - Raised reporting limit because of interference by the matrix.
acenaftyleen: - Raised reporting limit because of interference by the matrix.
pyreen: - Raised reporting limit because of interference by the matrix.
benzo(a)pyreen: - Raised reporting limit because of interference by the matrix.
anthraceen: - Raised reporting limit because of interference by the matrix.
indeno(1,2,3-cd)pyreen: - Raised reporting limit because of interference by the matrix.
benzo(k)fluoranteen: - Raised reporting limit because of interference by the matrix.
dibenz(a,h)anthraceen: - Raised reporting limit because of interference by the matrix.
benzo(a)antraceen: - Raised reporting limit because of interference by the matrix.
som PAK (Carcinogene): - Raised reporting limit because of interference by the matrix.
fluoreen: - Raised reporting limit because of interference by the matrix.
fluoranteen: - Raised reporting limit because of interference by the matrix.
chryseen: - Raised reporting limit because of interference by the matrix.
benzo(b)fluoranteen: - Raised reporting limit because of interference by the matrix.
som PAK (10): - Raised reporting limit because of interference by the matrix.
som PAK (EPA): - Raised reporting limit because of interference by the matrix.

Sample identification : **3. MARCH 9TH 02:00**
Reference number : **1015770**

Results Note(s):
acenafteen: - Raised reporting limit because of interference by the matrix.
benzo(ghi)peryleen: - Raised reporting limit because of interference by the matrix.
som PAK (overige): - Raised reporting limit because of interference by the matrix.
fenantreen: - Raised reporting limit because of interference by the matrix.
acenaftyleen: - Raised reporting limit because of interference by the matrix.
pyreen: - Raised reporting limit because of interference by the matrix.
benzo(a)pyreen: - Raised reporting limit because of interference by the matrix.
anthraceen: - Raised reporting limit because of interference by the matrix.
indeno(1,2,3-cd)pyreen: - Raised reporting limit because of interference by the matrix.
benzo(k)fluoranteen: - Raised reporting limit because of interference by the matrix.
dibenz(a,h)anthraceen: - Raised reporting limit because of interference by the matrix.
benzo(a)antraceen: - Raised reporting limit because of interference by the matrix.
som PAK (Carcinogene): - Raised reporting limit because of interference by the matrix.
fluoreen: - Raised reporting limit because of interference by the matrix.
fluoranteen: - Raised reporting limit because of interference by the matrix.
chryseen: - Raised reporting limit because of interference by the matrix.
benzo(b)fluoranteen: - Raised reporting limit because of interference by the matrix.
som PAK (10): - Raised reporting limit because of interference by the matrix.
som PAK (EPA): - Raised reporting limit because of interference by the matrix.

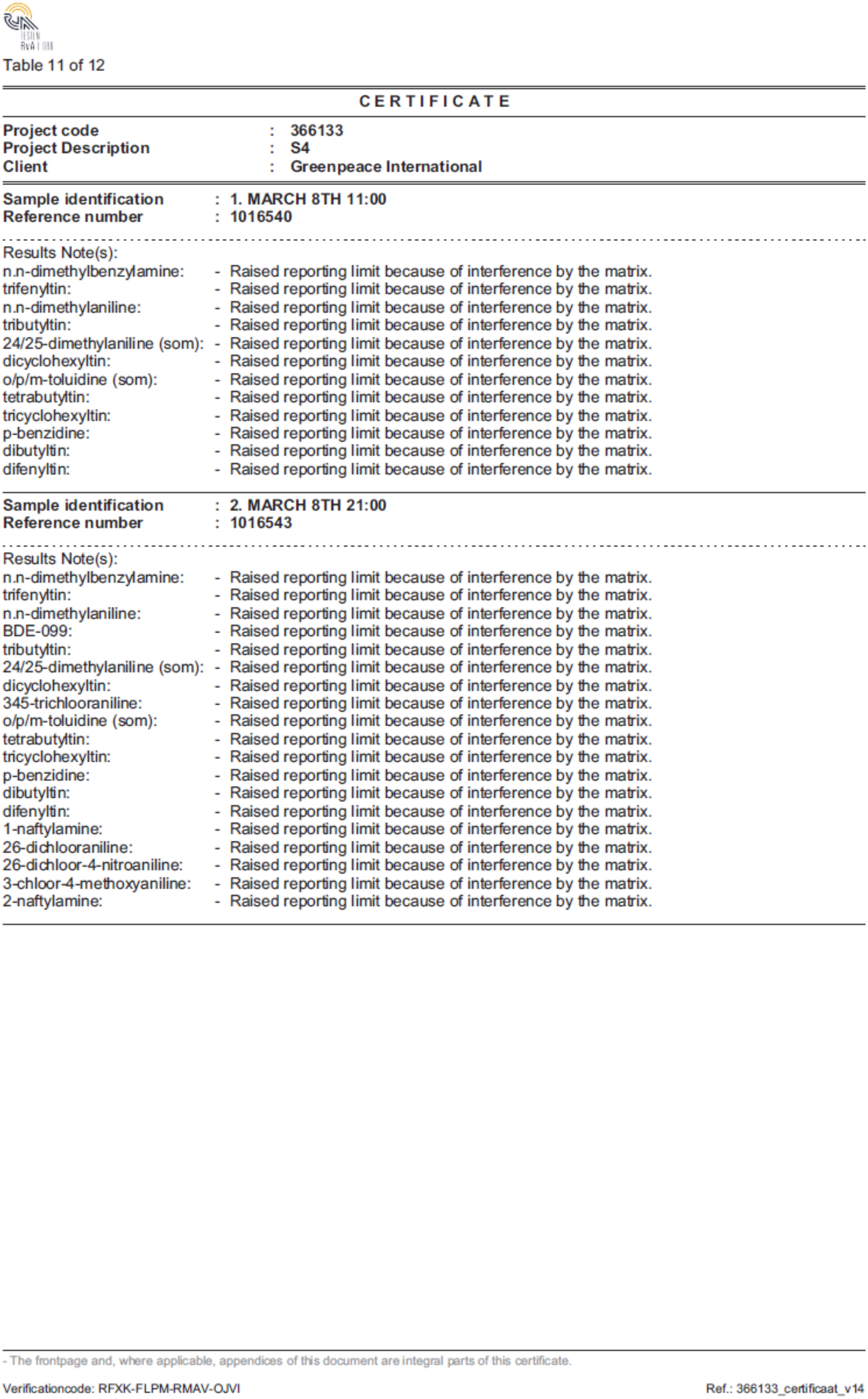

Table 11 of 12

CERTIFICATE

Project code : **366133**
Project Description : **S4**
Client : **Greenpeace International**

Sample identification : **1. MARCH 8TH 11:00**
Reference number : **1016540**

Results Note(s):
n.n-dimethylbenzylamine: - Raised reporting limit because of interference by the matrix.
trifenyltin: - Raised reporting limit because of interference by the matrix.
n.n-dimethylaniline: - Raised reporting limit because of interference by the matrix.
tributyltin: - Raised reporting limit because of interference by the matrix.
24/25-dimethylaniline (som): - Raised reporting limit because of interference by the matrix.
dicyclohexyltin: - Raised reporting limit because of interference by the matrix.
o/p/m-toluidine (som): - Raised reporting limit because of interference by the matrix.
tetrabutyltin: - Raised reporting limit because of interference by the matrix.
tricyclohexyltin: - Raised reporting limit because of interference by the matrix.
p-benzidine: - Raised reporting limit because of interference by the matrix.
dibutyltin: - Raised reporting limit because of interference by the matrix.
difenyltin: - Raised reporting limit because of interference by the matrix.

Sample identification : **2. MARCH 8TH 21:00**
Reference number : **1016543**

Results Note(s):
n.n-dimethylbenzylamine: - Raised reporting limit because of interference by the matrix.
trifenyltin: - Raised reporting limit because of interference by the matrix.
n.n-dimethylaniline: - Raised reporting limit because of interference by the matrix.
BDE-099: - Raised reporting limit because of interference by the matrix.
tributyltin: - Raised reporting limit because of interference by the matrix.
24/25-dimethylaniline (som): - Raised reporting limit because of interference by the matrix.
dicyclohexyltin: - Raised reporting limit because of interference by the matrix.
345-trichlooraniline: - Raised reporting limit because of interference by the matrix.
o/p/m-toluidine (som): - Raised reporting limit because of interference by the matrix.
tetrabutyltin: - Raised reporting limit because of interference by the matrix.
tricyclohexyltin: - Raised reporting limit because of interference by the matrix.
p-benzidine: - Raised reporting limit because of interference by the matrix.
dibutyltin: - Raised reporting limit because of interference by the matrix.
difenyltin: - Raised reporting limit because of interference by the matrix.
1-naftylamine: - Raised reporting limit because of interference by the matrix.
26-dichlooraniline: - Raised reporting limit because of interference by the matrix.
26-dichloor-4-nitroaniline: - Raised reporting limit because of interference by the matrix.
3-chloor-4-methoxyaniline: - Raised reporting limit because of interference by the matrix.
2-naftylamine: - Raised reporting limit because of interference by the matrix.

- The frontpage and, where applicable, appendices of this document are integral parts of this certificate.

Verificationcode: RFXK-FLPM-RMAV-OJVI

Ref.: 366133_certificaat_v14

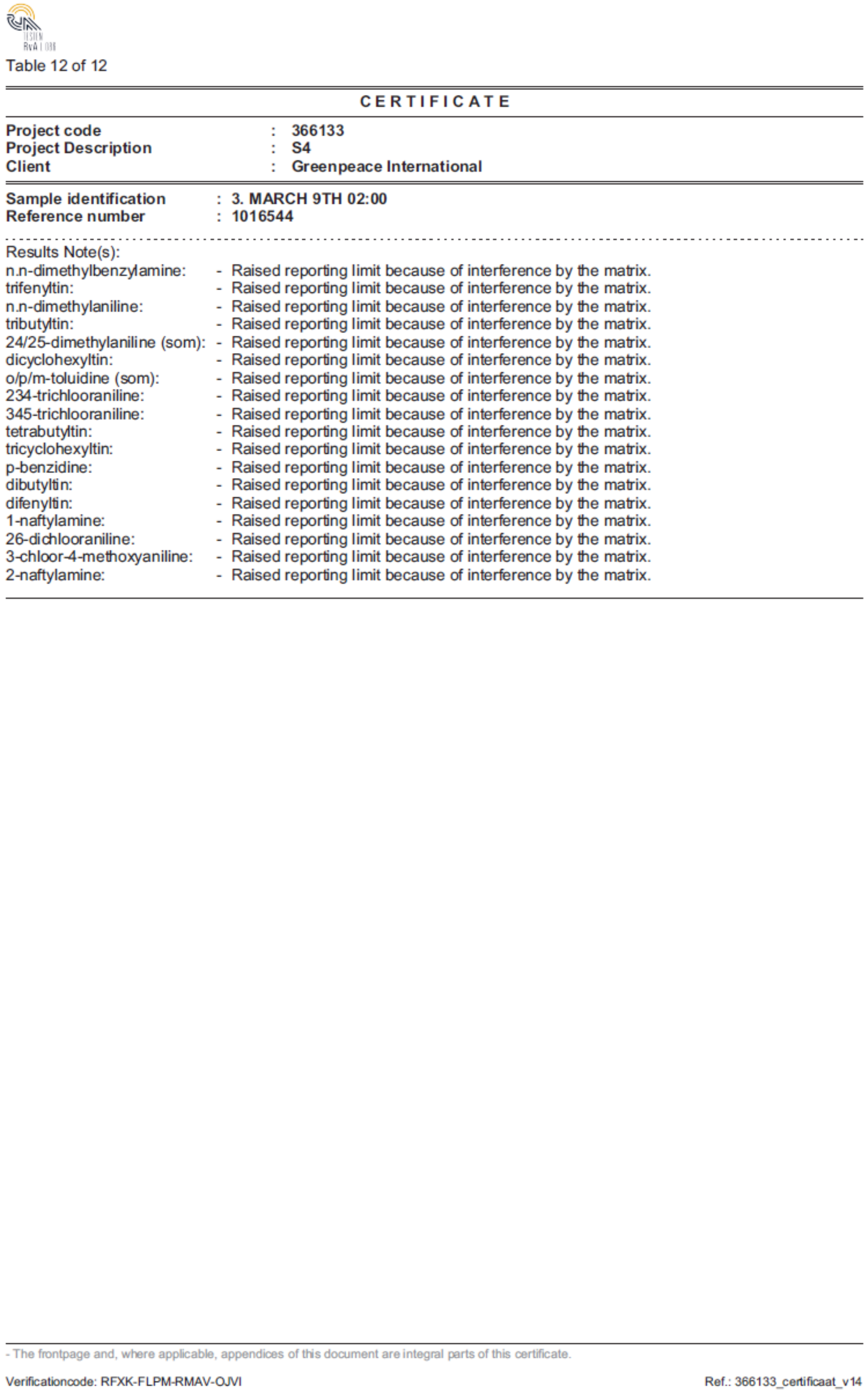

Table 12 of 12

CERTIFICATE

Project code : **366133**
Project Description : **S4**
Client : **Greenpeace International**

Sample identification : **3. MARCH 9TH 02:00**
Reference number : **1016544**

Results Note(s):
n.n-dimethylbenzylamine: - Raised reporting limit because of interference by the matrix.
trifenyltin: - Raised reporting limit because of interference by the matrix.
n.n-dimethylaniline: - Raised reporting limit because of interference by the matrix.
tributyltin: - Raised reporting limit because of interference by the matrix.
24/25-dimethylaniline (som): - Raised reporting limit because of interference by the matrix.
dicyclohexyltin: - Raised reporting limit because of interference by the matrix.
o/p/m-toluidine (som): - Raised reporting limit because of interference by the matrix.
234-trichlooraniline: - Raised reporting limit because of interference by the matrix.
345-trichlooraniline: - Raised reporting limit because of interference by the matrix.
tetrabutyltin: - Raised reporting limit because of interference by the matrix.
tricyclohexyltin: - Raised reporting limit because of interference by the matrix.
p-benzidine: - Raised reporting limit because of interference by the matrix.
dibutyltin: - Raised reporting limit because of interference by the matrix.
difenyltin: - Raised reporting limit because of interference by the matrix.
1-naftylamine: - Raised reporting limit because of interference by the matrix.
26-dichlooraniline: - Raised reporting limit because of interference by the matrix.
3-chloor-4-methoxyaniline: - Raised reporting limit because of interference by the matrix.
2-naftylamine: - Raised reporting limit because of interference by the matrix.

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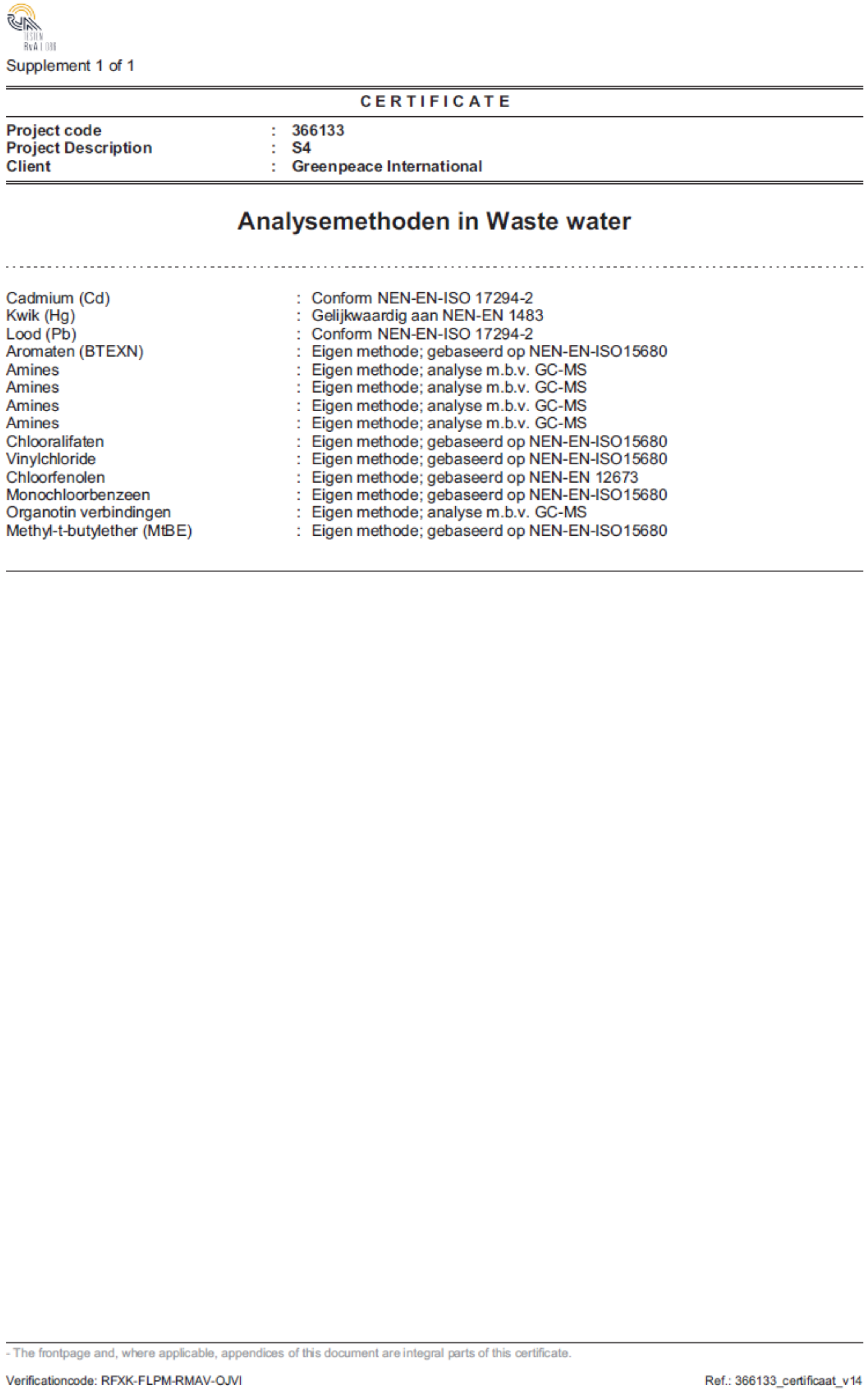
Verificationcode: RFXK-FLPM-RMAV-OJVI

Ref.: 366133_certificaat_v14



Supplement 1 of 1

CERTIFICATE

Project code : **366133**
Project Description : **S4**
Client : **Greenpeace International**

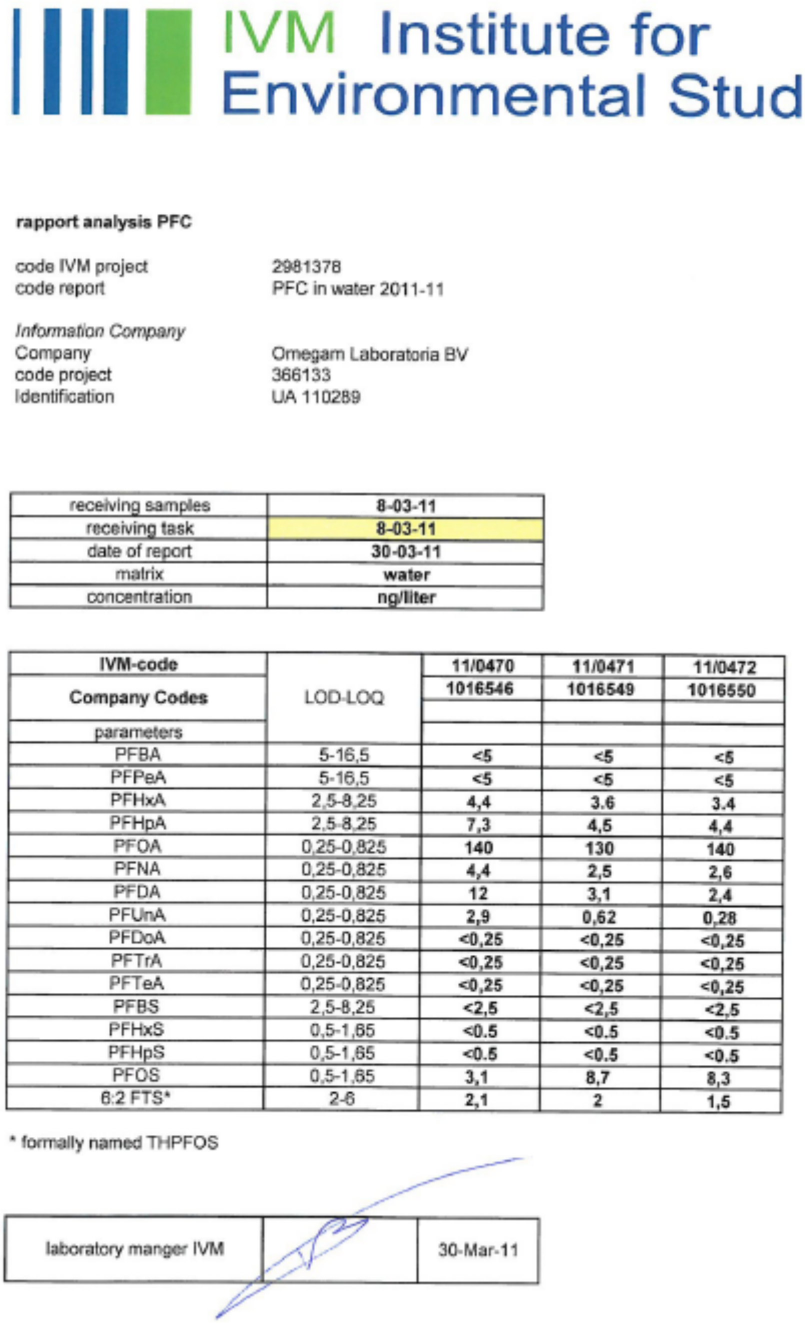
Analysemethoden in Waste water

Cadmium (Cd)	Conform NEN-EN-ISO 17294-2
Kwik (Hg)	Gelijkwaardig aan NEN-EN 1483
Lood (Pb)	Conform NEN-EN-ISO 17294-2
Aromaten (BTEXN)	Eigen methode; gebaseerd op NEN-EN-ISO15680
Amines	Eigen methode; analyse m.b.v. GC-MS
Amines	Eigen methode; analyse m.b.v. GC-MS
Amines	Eigen methode; analyse m.b.v. GC-MS
Amines	Eigen methode; analyse m.b.v. GC-MS
Chlooralifaten	Eigen methode; gebaseerd op NEN-EN-ISO15680
Vinylchloride	Eigen methode; gebaseerd op NEN-EN-ISO15680
Chloorfenolen	Eigen methode; gebaseerd op NEN-EN 12673
Monochloorbenzeen	Eigen methode; gebaseerd op NEN-EN-ISO15680
Organotin verbindingen	Eigen methode; analyse m.b.v. GC-MS
Methyl-t-butylether (MtBE)	Eigen methode; gebaseerd op NEN-EN-ISO15680

- The frontpage and, where applicable, appendices of this document are integral parts of this certificate.

Verificationcode: RFXK-FLPM-RMAV-OJVI

Ref.: 366133_certificaat_v14



IVM Institute for Environmental Studies

rapport analysis PFC

code IVM project	2981378
code report	PFC in water 2011-11

Information Company

Company	Omegam Laboratoria BV
code project	366133
Identification	UA 110289

receiving samples	**8-03-11**
receiving task	**8-03-11**
date of report	**30-03-11**
matrix	**water**
concentration	**ng/liter**

IVM-code	LOD-LOQ	**11/0470**	**11/0471**	**11/0472**
Company Codes		**1016546**	**1016549**	**1016550**
parameters				
PFBA	5-16,5	**<5**	**<5**	**<5**
PFPeA	5-16,5	**<5**	**<5**	**<5**
PFHxA	2,5-8,25	**4,4**	**3.6**	**3.4**
PFHpA	2,5-8,25	**7,3**	**4,5**	**4,4**
PFOA	0,25-0,825	**140**	**130**	**140**
PFNA	0,25-0,825	**4,4**	**2,5**	**2,6**
PFDA	0,25-0,825	**12**	**3,1**	**2,4**
PFUnA	0,25-0,825	**2,9**	**0,62**	**0,28**
PFDoA	0,25-0,825	**<0,25**	**<0,25**	**<0,25**
PFTrA	0,25-0,825	**<0,25**	**<0,25**	**<0,25**
PFTeA	0,25-0,825	**<0,25**	**<0,25**	**<0,25**
PFBS	2,5-8,25	**<2,5**	**<2,5**	**<2,5**
PFHxS	0,5-1,65	**<0.5**	**<0.5**	**<0.5**
PFHpS	0,5-1,65	**<0.5**	**<0.5**	**<0.5**
PFOS	0,5-1,65	**3,1**	**8,7**	**8,3**
6:2 FTS*	2-6	**2,1**	**2**	**1,5**

* formally named THPFOS

laboratory manger IVM		30-Mar-11

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