Organic chemical and heavy metal contaminants in wastewaters discharged from two textile manufacturing facilities in Indonesia

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Executive Summary

The manufacture of textile products involves the use of a diverse range of techniques and processes, many of which are chemically intensive. Some process and finishing chemicals used are known to have hazardous properties. For many other chemicals used, however, little or no information is publicly available on their toxicology or the potential impacts of their release to the environment.

Greenpeace International has recently reported the presence of a diverse range of hazardous chemicals within wastewaters discharged from manufacturing processes used by parts of the textile industry in China and Mexico. In addition, residues of hazardous chemicals have been found in clothing products manufactured and sold in many countries around the world.

The current study builds on this recent work by investigating releases of hazardous chemicals in effluents discharged by two facilities involved in the manufacture of textile products in Indonesia, namely the PT Gistex and PT Kahatex facilities, both located near Bandung City, West Java Province. Samples were collected from wastewater discharges at each of the two locations in May 2012. For each facility, a sample was collected from the main outfall on two separate days, while a single sample was collected from each of two smaller intermittent outfalls from the PT Gistex facility. The samples were not intended to provide a comprehensive understanding of the chemical make-up of discharged wastewater for each location, as this is something that can vary on a daily basis. Rather, the samples were intended to provide a snapshot of the type of hazardous chemicals present in the complex industrial wastewaters being discharged at the time of sampling, as an illustration of waste practice in the sector.

All samples were analysed at the Greenpeace Research Laboratories (University of Exeter, UK), using qualitative analysis to detect the presence (though not the concentrations) of semi-volatile and volatile organic compounds, as well as to determine the concentrations of a range of metals and metalloids. In addition, for samples from the PT Kahatex facility, the concentrations of a range of perfluorinated chemicals (PFCs, which cannot be identified using the technique employed for organic compounds qualitative analysis) were quantified at an accredited independent laboratory.

For one of the facilities (PT Gistex), a diverse range of chemicals was identified in the samples of discharged wastewater, many of which have known hazardous properties, and included compounds that are toxic to aquatic life, persistent, and able to bio-accumulate. Key findings for this facility can be summarised as follows:-

Chemicals found in wastewater samples discharged from the main outfall included:

- **Nonylphenol (NP)**, a well known persistent environmental contaminant with hormone disrupting properties, together with **nonylphenol ethoxylates (NPEs)**, which are used as detergents and surfactants in textile manufacture and washing, and which degrade to NP;

- **Tributyl phosphate (TBP)**, a hazardous chemical used in the textile industry, which is toxic to aquatic life and moderately persistent;

- Other substances for which little information is available on their toxicity, including quinoline-related compounds that are associated with the use of dyes, and certain ethylene glycol ethers;

- A high level of dissolved **antimony**, a toxic metalloid used in polyester manufacture.

Wastewater discharged from one of the two smaller intermittent outfalls was highly **alkaline** (pH 14) and contained a high loading of **p-terephthalic acid** (a raw material used in the manufacture of PET polyester) suggesting it had not received even the most basic of treatment prior to discharge. A high concentration of **antimony** was also found in this sample.

In addition, a significant proportion (in some cases, most) of the chemicals isolated from each of these samples could not be reliably identified, a characteristic that is not uncommon for complex industrial effluents. The properties and potential impacts of these substances cannot, therefore, be assessed.

This investigation has demonstrated the use and release of hazardous chemicals by the PT Gistex facility, and also highlighted very poor management of some wastewaters at this facility. Improved wastewater treatment, however, will not fully resolve the concerns identified in this study regarding hazardous chemical use. Conventional wastewater treatment processes are unable to treat effectively wastewaters containing NPEs and NP, chemicals that were identified in samples of effluent from this facility.

For the second facility (PT Kahatex), little was found in terms of hazardous organic chemicals related to the manufacture of textiles within samples of discharged wastewater, though these samples did contain residues of

chromium at concentrations that were moderately elevated above background concentrations commonly found in uncontaminated surface waters.

While this study, based on a small number of samples from two facilities, cannot be assumed to be representative of wastewaters released from textile manufacturing facilities throughout Indonesia, it does provide an illustration of what is likely to be a much wider problem of the discharge of effluents from this sector, some containing hazardous chemical contaminants.

Progressively replacing hazardous substances with less hazardous, and preferably non-hazardous, alternatives will help bring about rapid reductions and ultimate cessation in their discharges, emissions and losses, and ultimately reduce the burden of hazardous substances on the local and global environment. Making information on the current use and release of hazardous chemicals by individual facilities publicly available will be an important contribution towards that goal.

1. Introduction

The manufacture of textile products makes use of a diverse range of techniques and processes, many of which rely on the use of numerous process and finishing chemicals. Some of the chemicals currently employed within different parts of this sector are known to have intrinsic hazardous properties, while for many others little or no information is publicly available on their toxicology or other potential impacts should they be released to the environment.

Greenpeace has recently conducted a number of investigations of hazardous chemicals within wastewaters discharged from parts of the textile manufacturing industry in China and Mexico (Brigden *et al* 2009, Greenpeace 2011a, Brigden *et al* 2012b, Labunska *et al* 2012c), as well as their presence in various clothing products manufactured in many countries around the world (Brigden *et al* 2012a, Greenpeace 2011b). These studies have highlighted the ongoing use of a wide range of hazardous and potentially hazardous chemicals by parts of the textile manufacturing sector, and the resulting releases of such substances into the environment, particularly the aquatic environment.

This study extends the scope of the previous work by investigating releases of hazardous chemicals within wastewaters discharged into surface waters from two individual facilities involved in the manufacture of textile products in Indonesia. The two facilities are those operated by PT Gistex and by PT Kahatex, both located near Bandung City, West Java Province, Indonesia.

The PT Gistex textile facility is one of the largest manufacturing companies in Bandung Regency, Indonesia. This facility manufactures polyester fabrics, and includes weaving, dyeing, printing and finishing operations (Gistexgroup 2012). The PT Kahatex facility manufactures polyester fibres and fabrics, and also employs dyeing, printing and other finishing operations (Kahatex 2012).

2. Sampling programme

A total of seven water samples were collected in May 2012 from locations at which wastewaters were being discharged from each of the facilities. Details of the samples collected are provided below, and a summary of the sample descriptions and location of sampling points is presented in Tables 1a & 1b.

PT Gistex

Four samples of discharged wastewater samples (ID12001-ID12004) were collected on two separate days, 12 and 14 May 2012, from the outfalls of the PT Gistex facility to the Citarum River. The main outfall was sampled on both days (ID12001 & ID12002). For each of the two minor outfalls that were observed to discharge intermittently, a single sample was collected on 14 May (ID12003 & ID12004).

PT Kahatex

Three samples were collected in total – one on 12 May (ID12005) and two on 14 May 2012 (ID12006 & ID12007) – in the vicinity of the wastewater outfall of the PT Kahatex facility, where wastewater is discharged from a treatment plant via a channel that exits into a canal that flows through the facility. The canal may also receive other wastewaters from this and/or other facilities, upstream from the point at which the main discharge channel flows into it, though it has not been possible to confirm this. The canal that receives wastewaters from the PT Kahatex facility subsequently flows into the Cikijing River, which in turn joins the Citarik River, a tributary to the Citarum River.

The channel carrying discharged wastewater from this facility was sampled on both days (ID12005 & ID12006), upstream from its connection to the canal. In addition, a single sample was collected from the receiving canal itself (ID12007) on 14 May.

Due to the range of products manufactured by PT Kahatex, there were reasons to suspect that perfluorinated chemicals (PFCs) might be used at their facility to provide waterproof properties to certain products, and therefore samples associated with this facility were also collected to enable the quantification of a range of PFCs.

	Sample code	Collection point	Sample type	Time & Date	Description
PT Gistex	ID12001	Main outfall	Wastewater	08.30 12 May 2012	Discharge into River Citarum
	ID12002			10.10 14 May 2012	
	ID12003	Intermittent outfall #1		10.20 14 May 2012	Discharge into River Citarum, upstream of main outfall
	ID12004	Intermittent outfall #2			Discharge into River Citarum, upstream of main outfall and intermittent outfall #1
PT Kahatex	ID12005	Main discharge channel	Wastewater	14.00 12 May 2012	Prior to channel joining canal, approximately 3 m upstream of confluence
	ID12006			09.50 14 May 2012	
	ID12007	Canal flowing through facility	Canal water	09.55 14 May 2012	Approximately 10 m upstream of where canal is joined by main discharge channel

Table 1a: Details of samples collected from the vicinity of the PT Gistex and PT Kahatex textile manufacturing facilities near Bandung City, West Java Province, Indonesia, 2012.

	Sample code	North		East	
		Degree (°)	Minutes ('), Seconds (")	Degree (°)	Minutes ('), Seconds (")
PT Gistex	ID12001*	06	56'33"	107	32'05"
	ID12002*				
	ID12003*				
	ID12004*				
PT Kahatex	ID12005	06	57'48"	102	17'32.79"
	ID12006				
	ID12007	–	–	–	–

Table 1b: GPS coordinates of sample collection locations
* GPS location of river bank opposite the three outfalls

Each sample therefore consisted of two or three subsamples collected in different types of bottles depending on the subsequent analyses to be carried out on each subsample. In all cases, the first subsample was collected in a 0.5 litre screw-cap glass bottle, for use in the quantitative analysis of metals and metalloids, as well as the qualitative analysis of solvent extractable (semi-volatile) organic compounds. In addition, a second sub-sample was collected in a separate 50 ml amber glass bottle with a ground-glass stopper (filled to leave no headspace) to be analysed for volatile organic chemicals (VOCs). All glass bottles had been pre-cleaned, including being rinsed thoroughly with nitric acid and analytical grade pentane in order to remove all heavy metal and organic residues.

For the three samples associated with the PT Kahatex facility (ID12005-ID12007), a third sub-sample was collected in a 500 ml polypropylene bottle previously rinsed with analytical grade methanol, for use in the quantification of a range of perfluorinated chemicals (PFCs) in the water.

All samples in glass bottles were immediately chilled and kept cool and dark, while those collected for PFC analysis were frozen and kept frozen and dark, during transit to the Greenpeace Research Laboratories at the University of Exeter in the UK. Upon arrival, samples in glass bottles were stored cool and dark at the Greenpeace Research Laboratories prior to analysis at that laboratory, while those collected for PFC analysis were cooled to -20°C for 24 hours, before being transported in a frozen state to an accredited independent laboratory where they were subsequently analysed.

3. Methodology

For those samples analysed at the Greenpeace Research Laboratories, each was analysed quantitatively for the concentrations of a range of metals and metalloids, and qualitatively for the presence of semi-volatile (solvent-extractable) organic compounds and volatile organic compounds (VOCs).

Metal and metalloid concentrations were determined for all samples by ICP atomic emission spectrometry (AES) following acid digestion and using intra-laboratory standards. Both the total concentrations in the whole (unfiltered) sub-sample and the concentrations of dissolved forms in a filtered sub-sample were determined separately for each sample. Hexavalent chromium concentrations (in filtered samples only) were determined colourimetrically, using a diphenylcarbazide method.

Semi-volatile organic compounds were isolated from each sample using solid phase extraction (SPE) with ethyl acetate, pentane and toluene. Extracted compounds were subsequently identified as far as possible, using gas chromatography – mass spectrometry (GC/MS). Volatile organic chemicals (VOCs) were identified in all samples as received (with no pre-treatment), using GC/MS with HeadSpace sample introduction technique. More detailed descriptions of the sample preparation and analytical procedures are presented in the Appendix 1.

Those samples in which PFCs were quantified were analysed at an accredited independent laboratory using liquid chromatography-mass spectrometry (LC-MS/MS), employing appropriate quality control procedures, including the use of certain ^{13}C labelled PFCs as internal and recovery standards.

One sample (ID12004) was highly alkaline (pH 14), and upon neutralisation (to pH 6) a substantial amount of white solid precipitated from solution. This white solid was isolated by filtration, dissolved in acetone, and analysed separately by GC/MS. The analysis of semi-volatile (solvent-extractable) organic compounds in the neutralised solution was conducted separately.

4. Results and Discussion

The results for the samples are outlined in the following two sections, divided into the two locations from which the samples were collected. For all samples, semi-volatile (solvent-extractable) organic compounds identified in each of the individual samples are summarised in Table 2, with a full list of organic chemicals identified in each sample provided in Appendix 2. No VOCs were detected in any of the samples (ID12001-07). The concentrations of metals and metalloids in filtered (dissolved metals) and in whole waters (dissolved and suspended metals) are reported in Table 3. In addition, the concentrations of PFCs in samples ID12005-ID12007 are given in Appendix 3.

	PT Gistex				PT Kahatex		
	Main outfall		Intermittent outfall #1	#2	Discharge channel		Canal
Sample code	ID12001	ID12002	ID12003	ID12004	ID12005	ID12006	ID12007
Number of compounds isolated	82	90	3	5	22	16	27
Number identified to >90%	47	37	0	1	14	13	8
Percentage identified to >90%	57%	41%	0%	20%	64%	81%	30%
Nonylphenol isomers	13	11					
Nonylphenol ethoxylates isomers	19	9					
Nonylphenol diethoxylate isomers	6						
Tributyl phosphate (TBP)	✓	✓					✓
Quinoline and methyl derivatives	2	3					
Isoquinoline	✓	✓					
Tetrahydro quinolines		2					
Para-terephthalic acid				✓			
Phthalate: DEHP		✓*					
Ethylene glycols and derivatives	✓	3					
Ketones		2					
Long-chain alcohols	3	2					
Long-chain fatty acids	✓	✓					✓
4-methylphenol (*p*-cresol)		✓					
4-chloro-3-methyl-phenol					SIM	SIM	
Alkanes					13	12	
2-Butoxyethanol phosphate							✓
Tris-(1,3-dichloroisopropyl)phosphate							✓*
Benzotriazoles							✓
Triazine triamines							2*

Table 2: Summary of organic contaminants identified in samples of wastewater (ID12001-06) or canal water (ID12007) from the PT Gistex and PT Kahatex facilities in which semi-volatile (solvent extractable) organic compounds were identified (to >90% reliability). The number indicates, where appropriate, if more than one compound was identified for a group. SIM indicates compounds detected at trace levels only using Selected Ion Monitoring (SIM) mode of analysis.

* minor component of the total ion chromatogram

	PT Gistex								PT Kahatex					
	Main outfall				Intermittent outfall				Discharge channel				Canal	
					#1		#2							
	ID12001		ID12002		ID12003		ID12004		ID12005		ID12006		ID12007	
	Filt.	Wh.	Filt.	Wh.	Filt.	Wh.	Filt.	Wh.	Filt.	Wh.	Filt.	Wh.	Filt.	Wh.
Antimony	199	266	73	74	<50	<50	759	814	<50	<50	<50	<50	<50	<50
Arsenic	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50
Barium	15	51	35	37	<10	12	60	63	34	38	41	42	89	97
Cadmium	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5	<5
Chromium	<20	<20	<20	<20	<20	<20	<20	<20	194	251	154	198	<20	<20
Chromium (VI)	<50	-	<50	-	<50	-	<50	-	<50	-	<50	-	<50	-
Cobalt	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Copper	<20	<20	<20	<20	<20	<20	<20	<20	32	60	32	55	<20	24
Lead	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50	<50
Manganese	83	228	43	118	40	40	22	29	78	98	78	93	90	118
Mercury	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2	<2
Nickel	<20	<20	<20	<20	<20	<20	<20	<20	20	23	<20	23	<20	<20
Selenium	<200	<200	<200	<200	<200	<200	<200	<200	<200	<200	<200	<200	<200	<200
Vanadium	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20	<20
Zinc	43	103	35	92	18	26	88	86	45	91	60	88	67	177

Table 3: Concentrations of metals and metalloids (µg/l) in filtered (Filt.) and whole (Wh.) samples of wastewater (ID12001-06) or canal water (ID12007) from the PT Gistex and PT Kahatex facilities

4.1 PT Gistex

4.1.1 Organic contaminants

The sample of wastewater collected from the main outfall of the PT Gistex facility on 12 May 2012 (ID12001), and to a lesser extent the sample collected on 14 May 2012 (ID12002), were dominated by the presence of nonylphenol (NP) and nonylphenol ethoxylates (NPEs), consisting of mono- and di-ethoxylate isomers. It may be that these samples also contained additional longer-chained NPEs, though it was not possible to identify them using the GC/MS method employed. Other predominant compounds in these samples were tributyl phosphate (TBP) and certain quinoline-related compounds.

NP is a group of closely related compounds mainly produced for use in the manufacture of NPEs, which are widely used as detergents and surfactants in many industrial sectors, including in the manufacture of textiles. Within wastewater treatment facilities, or following release into the aquatic environment, NPEs readily degrade back to shorter-chain NPEs (such as mono- and di-ethoxylate isomers), and ultimately to NP, which is highly resistant to further degradation and therefore persistent within the environment. In addition, NP is able to bio-accumulate and is toxic, primarily due to its hormone-disrupting properties that can lead to altered sexual development in some organisms, most notably the feminisation of fish in contaminated waters (OSPAR 2004, Jobling et al 1995, 1996). As a result of their hazardous properties, the manufacture, use and release of NPEs and NP are regulated in some regions (OSPAR 1998, EU 2003b). Additional information on NPEs and NP, including on their hazardous properties, is available in Box A.

Box A. Nonylphenol (NP) and nonylphenol ethoxylates (NPEs)

Nonylphenol (NP) is a group of related (isomeric) chemicals that are manufactured primarily for use in producing nonylphenol ethoxylates (APEs), a group of non-ionic surfactants. NP is also used directly for a range of specialised industrial uses. NPEs are the most widely used type of a broader group of substances known as alkylphenol ethoxylates APEs). 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. Assessments in the early part of the last decade noted that the largest share were used 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).

Once released to the environment, NPEs can degrade back to NP, which are persistent, bio-accumulative, and toxic to aquatic life. Both NPEs and NPs are widely distributed in fresh and marine waters, and – in particular – sediments, in which persistent NPs tend to accumulate (Ying *et al* 2002, Jonkers *et al* 2005, David *et al* 2009, Shue *et al* 2010). Because of their releases to water, NPEs and NPs 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), and residues 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 NPEs, and close to sewer outfalls (Lye *et al* 1999, Rice *et al* 2003, Mayer *et al* 2008). NPs are known to accumulate in the tissues of fish and other organisms, and have been found as common contaminants of seafood in some locations (OSPAR 2004, Basheer *et al* 2004, Brigden *et al* 2010b). NPs have also been reported in human tissues (Lopez-Espinosa *et al* 2009).

The most widely recognised hazard associated with NPs is their estrogenic activity, i.e. 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. 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). NP is classified under the Globally Harmonised System (GHS) for classification and labelling of chemicals[1] as toxic to reproduction (category 2) H361FD), harmful if swallowed (H302), and very toxic to aquatic life, with the potential for long-lasting effects (H400, H410).

In Indonesia, the manufacture, use and release of NP and NPEs are not currently regulated. Outside Indonesia, regulations addressing the manufacture, use and release of NP and NPEs, together with certain other APE/APs, 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, in 2000, another AP (tert-octylphenol, t-OP) was also listed under this category (OSPAR 2006). Since then, NP has been ncluded as a "priority hazardous substance" under the 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).

Octylphenols (OPs) have been designated as "priority substances" under the Water Framework Directive. Even before their isting 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, since January 2005 within the EU, products (formulations used by industry) containing greater than 0.1% NP or NPEs may no longer be placed on the market within Europe, with some minor exceptions principally for "closed loop" industrial systems (EU 2003b). Restrictions on the sale of textile products containing residues of NPEs within the EU do not currently exist, though such a regulation is currently proposed by one EU member state (KEMI 2012). In addition, Germany has recently announced its intention to propose both NP and t-OP as substances of very high concern (SVHC) under the EU REACH Regulation (ECHA 2012a).

TBP has various industrial uses including as a carrier for certain dyes and as a plasticiser in plastics and textiles (WHO 1991, UNEP 2001, ATSDR 2009).

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[1] The Globally Harmonized System of Classification and Labelling of Chemicals (GHS), a system set up under the UN, provides a way of assessing the hazardous properties of chemicals through the use of hazard statements (UN 2011). The hazard statements used for individual substances within Section 3.4 are a composite of information drawn from a number of sources, including material safety data sheets (MSDSs) supplied by Sigma-Aldrich (http://www.sigmaaldrich.com); Landolt-Börnstein (http://lb.chemie.uni-hamburg.de); Merck Millipore (http://www.millipore.com); Alfa (http://www.alfa.com); ACROS (http://www.acros.be)

TBP is toxic to aquatic life and moderately persistent, and has previously been detected in wastewaters discharged to surface waters from wastewater treatment facilities (Reemtsma *et al* 2008, ATSDR 2009), including textile manufacturing wastewater (Greenpeace 2011, Labunska *et al* 2012c). More information on TBP is given in Box B.

Box B. Tributyl phosphate (TBP)

TBP has various industrial uses, which include use as a solvent, a flame retardant in hydraulic fluids, a carrier for certain dyes, a plasticiser in plastics and textiles, and as an antifoaming agent (WHO 1991, UNEP 2001, ATSDR 2009). TBP does not occur naturally in the environment (WHO 1991) but has been commonly detected in surface waters (Reemtsma *et al* 2008, ATSDR 2009, Regnery & Puttmann 2010), and freshwater sediments (Ishikawa *et al* 1985). TBP has also been detected in wastewaters discharged to surface waters from wastewater treatment facilities (Reemtsma *et al* 2008, ATSDR 2009), and was recently reported in wastewater discharged from a textile manufacturing facility in China (Greenpeace 2011) and another in Mexico (Labunska *et al* 2012c).

The biodegradation of TBP is moderate or slow, depending on conditions, and TBP can also be photochemically degraded in aquatic environments (Regnery & Puttmann 2010). TBP has been estimated to have a moderate potential to bioaccumulate in aquatic organisms (Sasaki *et al* 1981), and has been detected in fish and in liver samples from seabirds (Evenset *et al* 2009). TBP toxicity has been reported for a range of aquatic organisms (WHO 1991, UNEP 2001, Verbruggen *et al* 2005), and also in rodents. The acute toxicity to fish (LC_{50}) ranges from 4.2 to 11.8 μg/litre (WHO 1991), with the lowest reported no effect no observed effect concentration (NOEC) for fish of 0.82 μg/l (UNEP 2001). A maximum permissible concentration of 66 μg/l has been derived for TBP in freshwaters to protect all species from adverse effects (Verbruggen *et al* 2005). It has also been shown that TBP is toxic to certain protozoa species (Bowen *et al* 2006). In rodents, long term exposure to TBP has been associated with effects on the liver in mice, including increased incidence of benign liver tumours and an increase in liver weights (Auletta *et al* 1998), and in the bladder of rats (Arnold *et al* 1997). 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). TBP has been classified under the Globally Harmonised System (GHS) for classification and labelling of chemicals[1] as harmful if swallowed (H302), irritating to skin (H315) and suspected of causing cancer (H351). In the EU, TBP is included on the Community Rolling Action Plan (CoRAP) list of substances for evaluation to clarify the initial suspicion that its manufacture and/or use could pose a risk to human health or the environment (ECHA 2012b).

The quinoline-related compounds identified in the wastewater samples (ID12001 & ID12002) were isoquinoline, quinoline, two methyl quinoline derivatives (2-methyl- and 6-methyl-quinoline) and two tetrahydroquinolines. Quinoline and its derivatives are often associated with the manufacture and use of dyes, and some of these compounds were also identified in wastewaters analysed in a previous investigation of textile manufacturing facilities in China (Brigden *et al* 2011). More information on quinoline and quinoline derivative is given in Box C.

Box C. Quinoline and quinoline derivatives

Quinoline is a nitrogen-containing heterocyclic aromatic compound that exists in two isomeric forms, quinoline and isoquinoline. Synonyms of quinoline are 1-benzazine and benzo[b]pyridine; synonyms of isoquinoline are 2-benzazine and benzo[c]pyridine (Budavari *et al* 2000).

Quinoline is mainly used as an intermediate for manufacture of other chemicals. It is also used in metallurgical processes as a catalyst and corrosion inhibitor; in wood treatment; and in the manufacture of dyes, including water-soluble dyes (EPA US 2001). Its methylated derivatives, 2-methylquinoline and 4-methylquinoline, are precursors to cyanine dyes (Gu *et al* 2010, Shindy *et al* 2002). Quinoline and its derivatives are known environmental contaminants, they have been found in soil and groundwater at tar oil contaminated sites (Reineke *et al* 2007, Thomsen *et al* 1999), and in the wastewater from dyes and intermediary products industry (Yuan *et al* 2006).

As for many of heterocyclic aromatic compounds, quinoline is a toxic chemical. Liver damage has been observed in rats chronically exposed to quinoline by ingestion. An increased incidence of liver vascular tumours has been observed in rats and mice orally exposed to quinoline. The US EPA has provisionally classified quinoline as a Group C, possible human carcinogen (EPA US 2001). In addition, vapours of quinoline may irritate the eyes, nose and throat, and may cause headaches, dizziness and nausea in humans. Quinoline and its mono-methylated derivatives have also been shown to initiate skin tumours in SENCAR mice (LaVoie *et al* 1984). Aquatic toxicity of quinoline-related compounds has also been investigated on luminescent bacteria (*Photobacterium phosphoreum*) and fish bioassays (*Salmo gairdneri*) (Birkholz *et al* 1990). Twenty-one quinoline derivatives were investigated by those authors, which included isoquinoline and three mono-methyl quinolines. It was shown that all compounds are moderately toxic according to both microtox and rainbow trout assays.

Other organic chemicals identified in samples from this site included three ethylene glycol ethers, three long-chain alcohols (fatty alcohols) and corresponding ketones, a long-chain fatty acid, and a cresol. In addition, the di(2-ethyhexyl)phthalate or DEHP was identified as a minor component

Of the three ethylene glycol ethers identified, 2-(2-phenoxyethoxy)-ethanol (also known as diethylene glycol monophenyl ether) was detected in both samples collected from the main outfall (ID12001 and ID12002). The two other compounds, 2-phenoxy-ethanol (also known as glycol monophenyl ether), and 2-[2-(2-phenoxy-ethoxy)ethoxy]-ethanol) (also known as triethylene glycol monophenyl ether) were detected only in the second sample (ID12002).

Ethylene glycol ethers (EGEs) are a large group of chemicals defined by the formula: $R-(OCH_2CH_2)_n-OR'$, where n=1, 2 or 3; R is either an alkyl chain, phenyl or an alkyl substituted phenyl; R' is an alkyl chain, OR' consists of a carboxylic acid ester, sulphate, phosphate, nitrate or sulfonate. EGEs have numerous uses in industry as solvents in manufacture of lacquers, varnishes, resins, printing inks, textile dyes, and anti-icing additives (Klaassen *et al* 1986). Additionally, they are used in consumer products such as latex paints and cleaners. Glycol monophenyl ether is also commonly used as a preservative in personal care products.

Most reported toxicity studies were conducted on alkyl-substituted ethers in which they have shown a range of effects in experimental animals, including on reproductive systems, as well as some compounds exhibiting a potential for teratogenicity (Klaasen *et al* 1986). Short-chain EGEs are absorbed by all routes of exposure, and may cause irritation of skin, eyes, and mucous membranes; direct or indirect kidney and liver damage; immunotoxicity and nervous central system depression (Fogle 2002).

There is limited information on the specific EGEs detected in our study. For these three EGEs, it was possible to find toxicological data only in relation to glycol monophenyl ether (2-phenoxy-ethanol). This chemical is classified under the GHS as "harmful if swallowed" and "causes serious eye irritation". If inhaled, glycol monophenyl ether may cause coughs, sore throats, headaches, drowsiness and slurred speech, as well as effects on the central and peripheral nervous systems (IPCS 2003). It was also found to cause toxic effects on the reproduction of *Ceriodaphnia dubia* (Devillers *et al* 2003), a freshwater crustacean that is commonly used for assessing the chronic toxicity of single chemicals and effluents.

Long-chain alcohols, also known as fatty alcohols, are used as starting materials in the synthesis of alcohol ethoxylates (AEs), which are widely used as non-ionic surfactants and detergents, and can be substitutes for NPEs in many applications. Although both AEs and NPEs have similar toxicity to aquatic organisms, AEs are far more readily biodegradable than NPEs. Fatty alcohols are biodegradation intermediates of AEs. These alcohols are less toxic than the AEs, and are also readily biodegradable themselves. Ketones of the type also identified in one of the samples (ID12002) are intermediates in the biodegradation of certain fatty alcohols (Rehm & Reiff 1981).

The opposite is observed in the case for NPEs, where the biodegradation intermediates (nonylphenol) are far more toxic than NPEs, as well as being persistent and bio-accumulative (see Box A for more detail). The Predicted No-Effect Concentration (PNEC) for AEs is over two orders of magnitude less than that for NP (Campbell 2002). The use of AEs as surfactants, detergents, wetting and dispersing agents is widespread in industries such as paper manufacture, oil refining, as well as textiles. Other more minor uses are as pigment dispersants in decorative emulsion paints and water based inks, as well as in cosmetics and agrochemicals.

One of the fatty alcohols detected, 1-dodecanol, is a high production volume chemical primarily used for the production of dodecyl ethoxylates (an AE) and dodecyl sulfates. Although 1-dodecanol exhibits toxicity to aquatic organisms at concentrations of about 1 µg/l (IPCS 1998), it is readily degradable (Federle & Itrich 2006), as are other fatty acid alcohols, and does not generally give rise to long-term environmental concerns. 1-Dodecanol has also been found to be practically non-toxic in experiments with other laboratory animals, and no concerns have been identified to date as result of human use experiences (IPCS 1998).

Esters of long-chain fatty acids, such as 9-octadecenoic acid (also known as oleic acid) – which was identified at this location – are present in animal or vegetable fats, oils, or wax. These substances are used in a variety of textile manufacturing processes, including as defoaming and dispersing agents in dye formulations, and as sizing agents (IPPC 2003). These compounds are not of particular toxicological concern due to their low toxicity and being readily biodegradable.

4-methylphenol (also known as *p*-cresol) was identified in one of the two samples collected from the main outfall (ID12002). *p*-Cresol has many uses including the manufacture of certain antioxidants, in disinfectant and preservative formulations, and in many applications in the fragrance and dye industries. However, *p*-cresol is also a widely occurring natural compound, which could account for its presence in the sample ID12002 (ATSDR 2008). *p*-Cresol has been classified under GHS as toxic if swallowed or in contact with the skin (H301, H311).

The phthalate DEHP was detected in only one sample (ID12002), and in that as only a minor component. DEHP does have uses in the manufacture of textiles, though there are also numerous other uses for this phthalate, primarily as a plasticisers in PVC and other plastics, as well as uses as components of inks, adhesives, surface coatings and personal care product. DEHP is a common environmental contaminant, primarily due to losses from products in which they have been incorporated (Fromme *et al* 2002).

The presence of DEHP in the wastewater may be due to its use during textile manufacturing. However, given the widespread use of this compound, and that it was present as only a minor component of the mix of chemicals isolated from one wastewater sample, other sources cannot be excluded, including leaching from plasticised materials with which the wastewater had been in contact.

DEHP is a toxic substance, particularly due to its ability to exert toxic effects on reproductive systems in animals and humans (Howdeshell *et al* 2008, Lin *et al* 2008, Swan *et al* 2005), and as a result has been classified as "toxic to reproduction" within Europe, and listed as a "substance of very high concern" under the European REACH Regulation, together with some other phthalates (ECHA 2011). Furthermore, DEHP has been classified under the GHS as having the potential to damage fertility or the unborn child (H360DF).

In addition to samples of wastewater collected from the main outfall, samples were also collected from intermittent wastewaters discharges via two smaller adjacent outfalls. No organic compounds were identified in the sample collected from the intermittent outfall #1 (ID12003).

In contrast, the sample collected from the intermittent outfall #2 (ID12004) was highly alkaline (pH 14) which in itself poses a hazard to the receiving river and organisms within it. A white solid that precipitated from this wastewater upon neutralisation was present at a high concentration (10.8 g per litre, or 10800 μg/l). Subsequent analysis identified this white solid as 1,4-benzenedicarboxylic acid (also known as *p*-terephthalic acid). *p*-Terephthalic acid is one of the raw materials used to produce one form of polyester fibre known as poly(ethylene terephthalate), or PET. The term "polyester" refers to a group of polymers rather than a single substance, of which PET is the most significant in terms of quantities produced. *p*-Terephthalic acid has been classified under the GHS as irritating to eyes and skin (H315, H319).

The discharge of wastewater that is both highly alkaline and contains a high loading of *p*-terephthalic acid suggests that it had not received even the most basic of treatment prior to discharge.

No volatile organic compounds (VOCs) were detected in any of the samples associated with the PT Gistex facility (ID12001-ID12004).

4.1.2 Metals

In addition to the organic compounds identified, high levels of dissolved antimony were found in the samples of wastewater collected from the intermittent outfall #2 (ID12004, 759 μg/l) and from the main wastewater outfall (ID12001, 199 μg/l; and ID12002, 73 μg/l). These three samples also contained some additional antimony bound to particulates suspended in the wastewater, as indicated by the moderately higher concentrations in the whole (unfiltered) samples. Concentrations of antimony in unpolluted surface waters are typically below 1 μg/l (Filella *et al* 2002).

The polymerisation process used to produce PET is commonly catalysed by antimony trioxide, which is likely to result in wastewater containing antimony (Jaffe & East 2007). Furthermore, polyester fibres typically contain residues of antimony trioxide used in the manufacture. These fibres have a high surface area and are often subjected to harsh conditions during processing, during which the catalyst may be expected to leach out into processing water (Lacasse & Baumann 2004).

Antimony compounds have been associated with dermatitis and irritation of respiratory tract, as well as interfering with normal function of the immune system (Kim *et al* 1999). In addition, antimony trioxide has been listed by the International Agency for Research on Cancer (IARC) as "possibly carcinogenic to humans", with inhalation of dusts and vapours the critical route of exposure (IARC 1989).

Concentrations of the other metals included in the analysis showed little sign of elevation above typical background concentrations for uncontaminated surface waters.

4.2 PT Kahatex

4.2.1 Organic contaminants

Organic chemicals identified in the samples of wastewater collected from the main discharge channel of the PT Kahatex facility (ID12005 & ID12006) were predominately linear alkanes, the presence of which is likely to indicate contamination by oil or other petrochemicals (Overton 1994).

The only other compound identified in these samples was 4-chloro-3-methyl-phenol (also known as *p*-chlorocresol), which was present in both samples at only trace levels. *p*-Chlorocresol is used as a general biocide/preservative in a wide range of applications, including textile and leather goods, as well as in glues, paints, inks and pharmaceutical products. However, another possible source is from the reaction of chloramines, chemicals commonly used as water disinfectants, with cresol, a chemical that can occur naturally and can be commonly found in surface waters (Heasley *et al* 2004). *p*-Chlorocresol has been shown to be toxic to a number of freshwater fish and invertebrate species (EC 2002). Classifications for *p*-chlorocresol under the GHS include being very toxic to aquatic life (H400), harmful if swallowed or in contact with the skin (H302, H312), and able to cause serious eye damage (H318). *p*-Chlorocresol is, however, readily biodegradable, and it has been shown that it can be readily removed from wastewaters within treatments plant (Körner *et al* 2000).

In addition to the organic chemicals identified using qualitative chemical screening, as described above, one PFC was identified in one sample of discharged wastewater (ID12006). This compound, perfluorooctanoic acid (PFOA), was present at 12 ng/l (0.012 µg/l). All other quantified PFCs were below method detection limits (see Appendix 3 for details).

Many diverse uses of PFCs can give rise to PFOA, which is highly persistent in the environment once released. As a result, PFOA is a widespread environmental contaminant, including within surface water environments. The concentration of PFOA in this sample is with the range commonly reported in surface waters in several parts of the world (Loos *et al* 2009, Jin *et al* 2009, So *et al* 2007) though relevant data for Indonesia could not be found. The low concentration of PFOA, together with all other quantified PFCs being below detection limits, does not provide strong evidence that the presence of PFOA in the sample is due to specific use of PFCs within the PT Kahatex facility.

Additional organic chemicals were identified in the sample of water (ID12007) collected from the canal that flows through the PT Kahatex facility, into which treated wastewater from this facility is discharged. These included TBP (discussed in Section 4.1) and two other phosphates, a benzotriazole, two triazine triamine derivatives, and a long-chain fatty acid. No PFCs were detected in this sample (see Appendix 3 for details). The sources of any additional inputs of wastewater to this canal, upstream of where the main wastewater discharge channel from the PT Kahatex facility enters it, are not known, including any additional discharges from the PT Kahatex facility. Therefore, the origins of chemicals identified within the canal water are unknown, and their properties or potential sources are not discussed.

4.2.2 Metals

The two samples of wastewater collected from the main discharge channel (ID12005 & ID12006) contained dissolved chromium (154 µg/l and 194 µg/l respectively) at concentrations that were elevated above background concentrations expected for chromium in uncontaminated waters (median values are commonly around 10 µg/l, ATSDR 2012). Total chromium concentrations in these two samples were slightly higher, at 198 µg/l and 251 µg/l respectively. To a far lesser extent, the concentrations of dissolved copper (32 µg/l in both samples) were also somewhat elevated above background concentrations in surface waters, which are typically below 10 µg/l (ATSDR 2004, Comber *et al* 2008).

Chromium is a naturally occurring element that is an essential trace nutrient at very low concentrations but which, in common with many other metals, can be toxic to plants and animals at high concentrations. It exists in two main forms, chromium (III) and hexavalent chromium (IV), the latter being generally considered to be the most toxic (and carcinogenic) form of chromium. Chromium IV was not detected in either of the filtered wastewater samples. Even without detectable concentrations of chromium (VI), there could be toxicity of total chromium in dissolved forms to the aquatic environment (especially to algae and fish) following release of wastewaters at the detected levels. In the aquatic environment there are instances where chromium (III) has been found to exert greater toxicity than chromium (VI) to algae and cyanobacteria at the base of the food web (Thompson *et al* 2002, Jasso-Chavez *et al* 2010, Vignati *et al* 2010), and has also been found to induce greater oxidative stress in the liver and kidneys of goldfish (Lushchak *et al* 2009). The toxicity of chromium (III) is often limited by its low water solubility, though this can be influenced through the formation of soluble complexes with other chemicals present in water or wastewater,

including organic chemicals (Walsh & O'Halloran 1996, Belay 2010), which appears to be the case in this wastewater.

Chromium (III) compounds, and also copper compounds, have been used in certain dye formulations, though primarily for dyeing wool. In addition, chromium (VI) compounds have been used as oxidising agents for certain dyes. Although this use has been replaced in some regions, there may be ongoing uses in other places, including Indonesia. Chromium (VI) is usually readily converted into chromium (III) during wastewater treatment (Gregory 2007, IPPC 2003).

5. Conclusions

For one of the two facilities that were investigated in this study (PT Gistex), a diverse range of chemicals was identified in samples of discharged wastewater. The mix of chemicals included many with known hazardous properties, while for other little or no information is publicly available on their toxicological properties and, therefore, potential impacts of their release to the environment remain unknown. Certain compounds identified (NP and NPEs) are regulated in some counties with respect to their manufacture, use and release, as a result of the toxicity, persistence and bio-accumulative potential of NP.

This study has also highlighted particularly poor management of certain wastewaters at this facility, for which data suggest discharge can occur without even the most basic of treatment. Improvements in wastewater treatment, however, will not fully resolve the concerns identified in this study regarding the use and release of hazardous chemicals. Conventional wastewater treatment processes are unable to address effectively the presence of some of the key substances identified in effluent samples (NPEs and NP).

Overall, this study has demonstrated that the PT Gistex facility provides a clear example of the use and consequent release of hazardous chemicals from a textile manufacturing facility in Indonesia.

While this study, based on a small number of samples from two facilities, cannot be representative of wastewaters released from textile manufacturing facilities throughout Indonesia, it does provide a further illustration of what is likely to be a much wider problem of the discharge of effluents from this sector containing hazardous chemical contaminants.

Progressively replacing hazardous substances with less hazardous, and preferably non-hazardous, alternatives will help bring about rapid reductions and ultimate cessation in their discharges, emissions and losses, and ultimately reduce the burden of hazardous substances on local and global environment. Making information on the current use and release of hazardous chemicals by individual facilities publicly available will be an important contribution towards that goal.

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Appendix 1: Details of methodologies

Analysis for Volatile Organic Compounds (VOCs)

Methods

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 program included an initial temperature of 35°C (held for 4min), rising to 55°C at 5°C/min, and then to 210°C at 15°C/min. The carrier gas was helium, supplied at 1ml/min. From each sample, three 10ml portions were sub-sampled into 20 ml 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. Additionally, Selected Ion Monitoring (SIM) analysis was performed simultaneously with SCAN analysis to identify the presence of the selected VOCs listed below in Table A1. 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 stored for duplicate quantitative analysis, if required, for any VOCs that had been detected in the samples through screening.

Compound	Compound
Benzene	Ethene, tetrachloro-
Benzene, 1,2,3-trichloro-	Ethene, trichloro-
Benzene, 1,2,4-trichloro-	Hexachlorobutadiene
Benzene, 1,2,4-trimethyl-	*m*- & *p*-Xylene
Benzene, 1,2-dichloro-	Methane, bromo-
Benzene, 1,3,5-trichloro-	Methane, bromochloro-
Benzene, 1,3,5-trimethyl-	Methane, bromodichloro-
Benzene, 1,3-dichloro-	Methane, chloro-
Benzene, 1,4-dichloro-	Methane, dibromo-
Benzene, bromo-	Methane, dibromochloro-
Benzene, chloro-	Methane, dichloro-
Benzene, tert-butyl-	Methane, dichlorodifluoro-
Bromoform	Methane, tetrachloro-
Chloroform	Naphthalene
Ethane, 1,1,1,2-tetrachloro-	*o*-Xylene
Ethane, 1,1,1-trichloro-	Propane, 1,2,3-trichloro-
Ethane, 1,1,2,2-tetrachloro-	Propane, 1,2-dibromo-3-chloro-
Ethane, 1,1-dichloro-	Propane, 1,3-dichloro-
Ethane, 1,2-dibromo-	Propene, 1,3-dichloro-, trans-
Ethane, 1,2-dichloro-	Styrene
Ethane, chloro-	Toluene
Ethane, hexachloro-	Toluene, 2-chloro-
Ethene, 1,1-dichloro-	Toluene, 4-chloro-
Ethene, 1,2-dichloro-, cis-	Vinyl chloride
Ethene, 1,2-dichloro-, trans-	Ethene, tetrachloro-

Table A1. List of VOC compounds for SIM analysis.

Quality control

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.

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

For the clean-up stage, each extract was shaken with 3 ml isopropyl alcohol and 3 ml 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 1 ml 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 (30 m, 0.25 mm 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 program 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 8 minutes). The carrier gas was helium, supplied at 1 ml/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. Any background contaminants detected in blanks are subtracted from the chromatograms obtained for the samples before mass spectra are interpreted.

Analysis for metals

Preparation

To obtain total metal concentrations, a representative portion of each whole water sample was acidified by the addition of concentrated nitric acid to give a final concentration of 10% v/v. Separately, a portion of each whole sample was filtered through a 0.45 micron filter and then acidified in the same way to enable determination of dissolved metal concentrations. 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 5.0 μg/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.

Concentrations of chromium (VI) were determined colourimetric for each water sample following filtration. 0.5 ml of a 1,5-diphenylcarbazide testing solution (freshly prepared from 0.4 g of 1,5-diphenylcarbazide, 20 ml acetone, 20 ml ethanol, 20 ml orthophosphoric acid solution and 20 ml of demineralised water) was added to 9.5 ml of each filtered sample. The solution was mixed and let to stand for 10 minutes to allow full colour development. Concentrations were determined using a spectrophotometer at 540 nm, correcting with a blank prepared from deionised water, using standards freshly prepared from potassium dichromate at concentrations of 0, 50, 100, 200 and 500 μg/l respectively. Any sample exceeding the calibration range was diluted accordingly, in duplicate, and re-analysed.

Quality control

Two samples were prepared for ICP analysis 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 μg/l for each metal, 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 4 μg/l and 0.4 μg/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.

For chromium (VI) determination, two samples were prepared and analysed in duplicate, along with a blank sample, and two quality control solutions of 100 and 400 μg/l prepared in an identical manor to the samples.

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

Appendix 2: Detailed semi-volatile organic analytical screening data

Detailed screening data arising from GC-MS analysis of solvent extracts of each of the samples are presented below. These data list only those semi-volatile organic compounds identified following solvent extraction and do not include any volatile organic compounds (VOCs) identified through separate headspace GC-MS analysis of sub-samples; No VOCs were identified in each of the samples. Only those semi-volatile substances identified to greater than 90% quality match (following verification by expert interpretation) are listed here.

Sample code	ID12001
Location	PT Gistex facility, near Bandung City, West Java Province
Sample type	Wastewater
Date & time	12.05.12; 08.30
Description	Discharge from main outfall into River Citarum
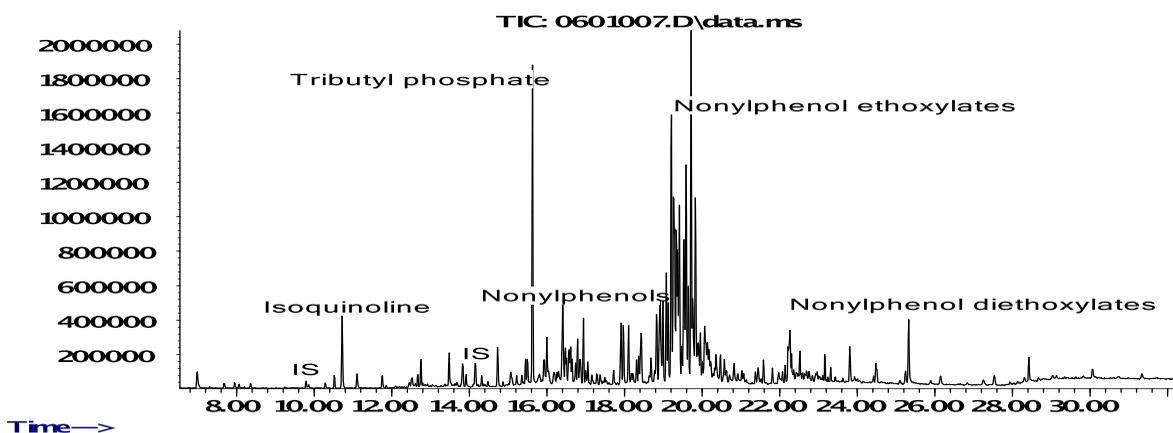
Number of compounds isolated: 82

Compounds identified to better than 90%:

CAS#	Name
000119-65-3	Isoquinoline
000126-73-8	Tributyl phosphate
000112-53-8	1-Dodecanol
000104-76-7	1-Hexanol, 2-ethyl-
001653-31-2	2-Tridecanol
000112-80-1	9-Octadecenoic acid (z)-
000104-68-7	Ethanol, 2-(2-phenoxyethoxy)-
084852-15-3	Nonylphenol, mixture of 13 isomers
000000-00-0	Nonylphenol ethoxylate, mixture of 19 isomers
000000-00-0	Nonylphenol diethoxylate, mixture of 6 isomers
000091-22-5	Quinoline
000091-63-4	Quinoline, 2-methyl-

Sample code	ID12002
Location	PT Gistex facility, near Bandung City, West Java Province
Sample type	Wastewater
Date & time	14.05.12; 10.10
Description	Discharge from main outfall into River Citarum
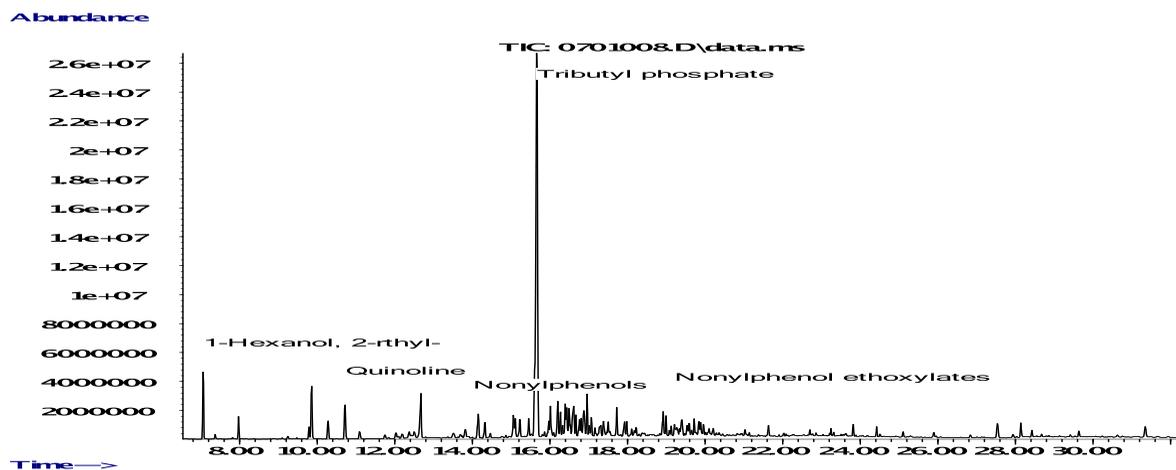
Number of compounds isolated: 90

Compounds identified to better than 90%:

CAS#	Name
000117-81-7	1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester*
000506-52-5	1-Hexacosanol
000104-76-7	1-Hexanol, 2-ethyl-
006175-49-1	2-Dodecanone
030692-16-1	5-Tridecanone
000112-79-8	9-Octadecenoic acid, (e)-
000104-68-7	Ethanol, 2-(2-phenoxyethoxy)-
007204-16-2	Ethanol, 2-[2-(2-phenoxyethoxy)ethoxy]-
000122-99-6	Ethanol, 2-phenoxy-
000119-65-3	Isoquinoline
084852-15-3	Nonylphenol, mixture of 11 isomers
000000-00-0	Nonylphenol ethoxylate, mixture of 9 isomers
000106-44-5	Phenol, 4-methyl-
000091-22-5	Quinoline
000635-46-1	Quinoline, 1,2,3,4-tetrahydro-
001780-19-4	Quinoline, 1,2,3,4-tetrahydro-2-methyl-
000091-63-4	Quinoline, 2-methyl-
000091-62-3	Quinoline, 6-methyl-
000126-73-8	Tributyl phosphate

* minor component of the total ion chromatogram

Sample code	ID12003
Location	PT Gistex facility, near Bandung City, West Java Province
Sample type	Wastewater
Date & time	14.05.12; 10.20
Description	Discharge from smaller outfall #1 into River Citarum
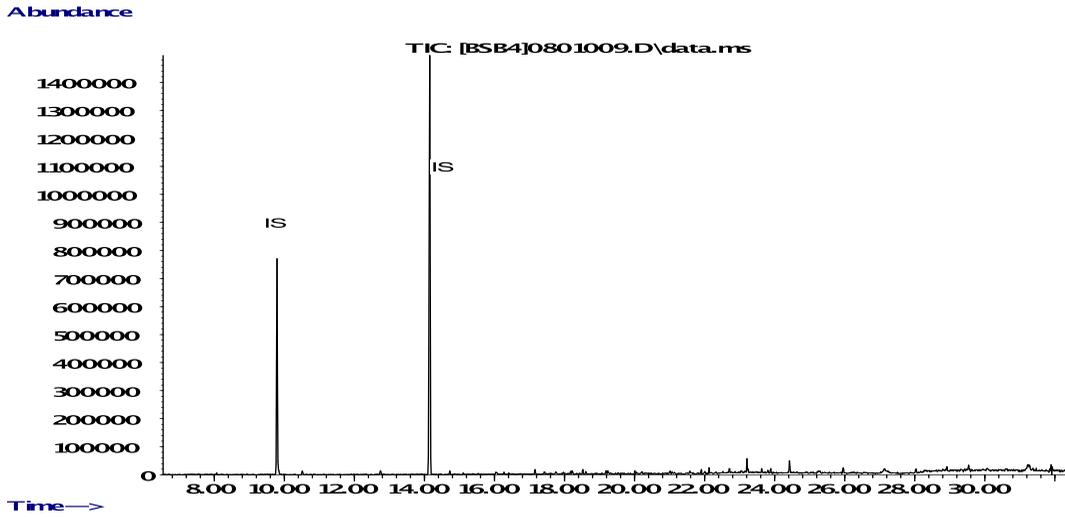
Number of compounds isolated: 3

Compounds identified to better than 90%:

None

Sample code	ID12004
Location	PT Gistex facility, near Bandung City, West Java Province
Sample type	Wastewater
Date & time	14.05.12; 10.30
Description	Discharge from smaller outfall #2 into River Citarum

Abundance

TIC: [BSB3]0401004.D\data.ms
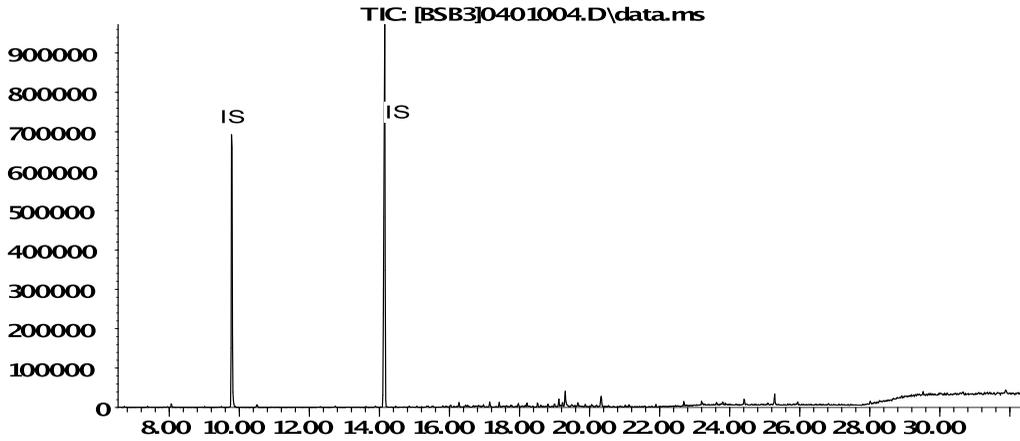
Time—>

Chromatogram of sample extract prepared after removal of terephthaliic acid

Number of compounds isolated: 5

Compounds identified to better than 90%:

CAS#   Name

000100-21-0   1,4-Benzenedicarboxylic acid (synonym: para-terephthalic acid)

Sample code	ID12005
Location	PT Kahatex facility, near Bandung City, West Java Province
Sample type	Wastewater
Date & time	12.05.12; 14.00
Description	Collected from main discharge channel

TIC: [BSB2]0401004.D\data.ms
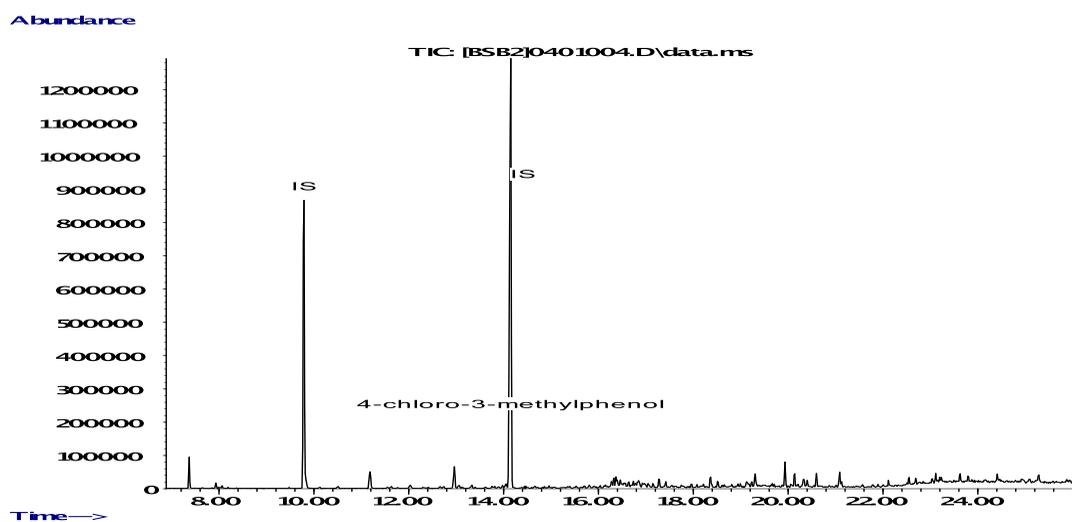
Number of compounds isolated: 22

Compounds identified to better than 90%:

CAS#	Name
000629-97-0	Docosane
000544-85-4	Dotriacontane
000112-95-8	Eicosane
000629-94-7	Heneicosane
000629-78-7	Heptadecane
000630-01-3	Hexacosane
000544-76-3	Hexadecane
000629-92-5	Nonadecane
000630-02-4	Octacosane
000593-45-3	Octadecane
000629-99-2	Pentacosane
000059-50-7	Phenol, 4-chloro-3-methyl-
000646-31-1	Tetracosane
000638-68-6	Triacontane

Sample code	ID12006
Location	PT Kahatex facility, near Bandung City, West Java Province
Sample type	Wastewater
Date & time	14.05.12; 09.50
Description	Collected from main discharge channel
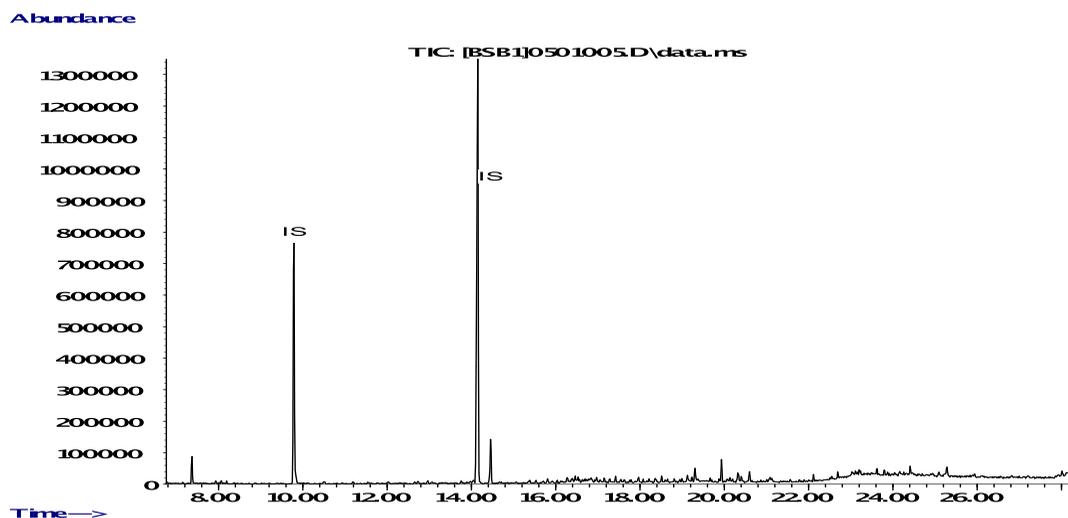
Number of compounds isolated: 16

Compounds identified to better than 90%:

CAS#	Name
000629-97-0	Docosane
000544-85-4	Dotriacontane
000112-95-8	Eicosane
000629-94-7	Heneicosane
000629-78-7	Heptadecane
000630-01-3	Hexacosane
000544-76-3	Hexadecane
000629-92-5	Nonadecane
000630-02-4	Octacosane
000593-45-3	Octadecane
000059-50-7	Phenol, 4-chloro-3-methyl- (SIM)
000629-99-2	Pentacosane
000646-31-1	Tetracosane

Sample code	ID12007
Location	PT Kahatex facility, near Bandung City, West Java Province
Sample type	Wastewater
Date & time	14.05.12; 09.55
Description	Collected from canal flowing through facility at a location prior to the junction with main discharge channel
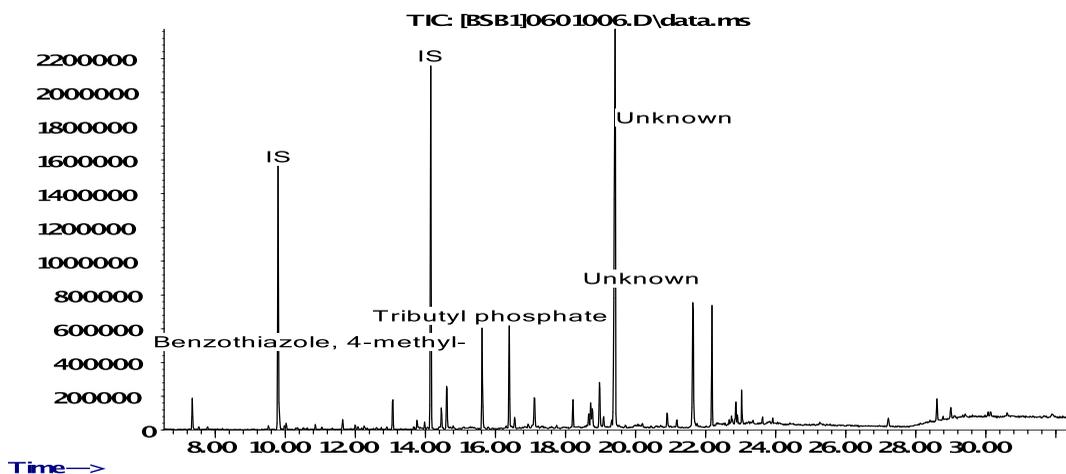
Number of compounds isolated: 27

Compounds identified to better than 90%:

CAS#	Name
000000-00-0	1H-Benzotriazole, 4-methyl-
000108-78-1	1,3,5-Triazine-2,4,6-triamine*
001014-70-6	1,3,5-Triazine-2,4-diamine, N,N'-diethyl-6-(methylthio)-*
020200-86-6	2H-indol-2-one, 1,3-dihydro-1,3,3-trimethyl-
000078-51-3	Ethanol, 2-butoxy-, phosphate (3:1)
000057-10-3	Hexadecanoic acid
000000-00-0	Tris-(1,3-dichloroisopropyl)phosphate*
000126-73-8	Tributyl phosphate

Appendix 3: Data for PFCs quantified in water samples associated with the PT Kahatex facility

Description	Main discharge channel		Canal flowing through facility
Type	wastewater	wastewater	canal water
Sample code	ID12005	ID12006	ID12007
	(ng/l)	(ng/l)	(ng/l)
Perfluorobutanoic acid (PFBA)	< 3	< 3	<10
Perfluoropentane acid (PFPeA)	< 3	< 3	<10
Perfluorohexanoic acid (PFHxA)	< 3	< 3	<10
Perfluorheptanoic acid (PFHpA)	< 3	< 3	<10
Perfluorooctanoic acid (PFOA)	< 3	12	<10
Perfluorononanoic acid (PFNA)	< 3	< 3	<10
Perfluordecanoic acid (PFDA)	< 3	< 3	<10
Perfluoroundecanoic acid (PFUnA)	< 3	< 3	<10
Perfluorododecane acid (PFDoA)	< 3	< 3	<10
Perfluorotridecane acid (PFTrA)	< 3	< 3	<10
Perfluorotetradecane acid (PFTA)	< 3	< 3	<10
Perfluorbutansulfonate (PFBS)	< 5	< 5	<15
Perfluorohexane sulfonate (PFHxS)	< 5	< 5	<15
Perfluoroheptane sulphonate (PFHpS)	< 5	< 5	<15
Perfluorooctane sulfonate (PFOS)	< 3	< 3	<10
Perfluorodecane sulphonate (PFDS)	< 5	< 5	<15
Perfluorooctane-sulfonamide (PFOSA)	< 3	< 3	<10
7H-Dodecafluoroheptanoic acid (HPFHpA)	< 7	< 7	<20
2H,2H-Perfluorodecane acid (H2PFDA)	< 7	< 7	<20
2H,2H,3H,3H-Perfluoroundecane acid (H4PFUnA)	< 7	< 7	<20
4:2 Fluorotelomer sulfonate (FTS)	< 7	< 7	<20
6:2 Fluorotelomer sulfonate (FTS)	< 5	< 5	<15
8:2 Fluorotelomer sulfonate (FTS)	< 7	< 7	<20

Table A3: Concentrations of PFCs (ng/l) quantified in wastewater discharged from the PT Kahatex facility (ID12005 & ID12006), and water from the receiving canal that flows through the facility (ID12007)