TECHNEAU

WP5.2:

Combination of MAR and adjusted conventional treatment processes for an Integrated Water Resources Management

Deliverable 5.2.6:

Occurrence and fate of microbial pathogens and organic trace compounds at riverbank filtration sites in Delhi, India.

KOMPETENZZENTRUM Wasser Berlin

Freie Universität Berlin

TECHNEAU is an Integrated Project Funded by the European Commission under the Sixth Framework Programme, Sustainable Development, Global Change and Ecosystems Thematic Priority Area (contractnumber 018320).

Colophon

Title
Occurrence and fate of microbial pathogens and organic trace compounds at riverbank filtration sites in Delhi, India.

Author(s)
Christoph Sprenger (FUB), Gunnar Lorenzen (FUB), Asaf Pekdeger (FUB)

Quality Assurance
By Iris Huelshoff (KWB), Gesche Grützmacher (KWB)

Deliverable number
D 5.2.6

Address for correspondence:

- Dipl.–Geol. Gunnar Lorenzen, Freie Universität Berlin, Hydrogeology Group, Malteserstr. 74-100, 12249 Berlin, Germany, tel.: +49 30 83840612; fax: +49 30 83870742; e-mail: **lorenzen@zedat.fu-berlin.de**
- Dipl.–Geol. Christoph Sprenger, Freie Universität Berlin, Hydrogeology Group, Malteserstr. 74-100, 12249 Berlin, Germany, tel.: +49 30 83840612; fax: +49 30 83870742; e-mail: **chspreng@zedat.fu-berlin.de**
- Dr. Gesche Grützmacher, KompetenzZentrum Wasser Berlin gGmbH, Cicerostr. 24, 10709 Berlin, tel.: +49 30 53653 813, fax: +49 30 53653 888, e-mail: **gesche.gruetzmacher@kompetenz-wasser.de**
- Prof. Dr. Asaf Pekdeger, Freie Universität Berlin, Hydrogeology Group, Malteserstr. 74-100, 12249 Berlin, Germany, tel.: +49 30 83840612; fax: +49 30 83870742; e-mail: **pekdeger@zedat.fu-berlin.de**

This report is:
PU = Public

PREFACE

Riverbank Filtration (RBF) is a valuable method for the (pre-)treatment of surface water for drinking water production. It has successfully been used in different parts of Europe for more than one century. The main intention of work package 5.2 of the TECHNEAU integrated project is to analyze the function and relevance of Riverbank Filtration (RBF) to enable sustainable water resources management, especially in developing and newly industrialized countries. A review on the attenuation capacity of RBF with a main focus on the significance for developing and newly industrialized countries is given in the D 5.2.3.

This report (D 5.2.6) provides an overview on pathogen and organic trace compound content in water samples from the three TECHNEAU riverbank filtration (RBF) sites in Delhi, India. It is a follow up of the D 5.2.1 report that gives an introduction to the studies in Delhi, including regional information to water stressed mega city, environmental conditions at the three field sites and a summary of the hydrogeological investigations. Further information on hydrogeochemistry including inorganic ions (major ions, heavy metals and inorganic trace substabnces) and physicochemical parameters was submitted in D 5.2.2.

The data published in this report represents water samples that have been collected during several field campaigns between May 2007 and March 2008 and analysed in different laboratories in India and Europe. Microbiological analysis includes faecal bacteria and indicator bacteria, bacteriophages and enteric viruses. For the analysis of organic contaminants, a non target GC-MS screening was performed as well as a quantitative analysis of pesticides and other trace pollutants.

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Abbreviations and units:

mbgl – Meters below ground level
ppm – Parts per million (1ppm = 1 mg/L)
mg/L - Milligram per liter (1mg/L = 1 ppm)
EC - Electrical Conductivity [µS/cm]
Eh - Redoxpotential [mV]
PZ - Piezometer (observation well)
GW - Groundwater
SW - Surface water
MAR - Managed Aquifer Recharge
RBF - Riverbank Filtration
DW - Dug well (traditional shallow, open well with bricked wall)
DJB - Delhi Jal Board (Delhi water supplier)
EC - electric conductivity [µS/cm]
TDS - total dissolved solids [mg/L]
PA - Palla Well Field (field site)
NI - Nizamuddin Bridge (field site)
NA - Najafgarh Drain (field site)
UBA – Umweltbundesamt (Federal Environmental Agency, Germany)
MPN – Most probable number
CPCB – Central Pollution Control Board
IITD – Indian Institute of Technology Delhi

1. Introduction

Most waterborne diseases are caused by pathogenic microorganisms (protozoa, viruses, bacteria and intestinal parasites) which are directly transmitted to the human body when contaminated drinking water is consumed. Several studies have shown that Riverbank Filtration (RBF) is an effective technique to remove pathogens through subsurface passage (Mathess et al. 1988; Schijven et al. 2003a; Berger 2001).

Apart from pathogens, organic trace compounds are widespread pollutants in rivers and lakes. Most common sources are industrial or sewage effluents in urbanised areas or agrochemicals in rural regions. The capacity of RBF to effectively or even completely remove many organic contaminants has been confirmed in numerous investigations (Kuehn et al. 2000, Weiss et al. 2003, Sacher & Brauch 2002, Herberer et al. 2004, Grünheid et al. 2005).

RBF is a process during which surface water is induced to infiltrate into the subsurface either due to a natural hydraulic gradient or the depression cone of an abstraction well. During infiltration and soil passage, the quality of the surface water is substantially improved thanks to a combination of physical, chemical, and biological processes such as filtration, dilution with genuine groundwater, and sorption as well as biodegradation of pollutants (Kivimäki et al. 1998; Kuehn et al. 2000; Stuyfzand 1998; Tufenkji et al. 2002; Weiss et al. 2003). Depending on the infiltration conditions, RBF removes organic matter (Sontheimer 1980; Tufenkji et al. 2002), some organic trace pollutants like pesticides or pharmaceuticals (Kuehn et al. 2000), disinfection by-products (Weiss et al. 2003) as well as pathogens (Havelaar et al. 1995; Hijnen et al. 2004; Schijven et al. 2003b; Weiss et al. 2003). The effectiveness of RBF depends strongly on the formation of a colmation layer (Dizer et al. 2004; Wang et al. 2007), filter depth (Ellis 1985; Hijnen et al. 2004), the pore-water velocities, the residence time of the water in the soil (Dillon et al. 2002) and the quality of surface water (Goldschneider et al. 2007; Ray et al. 2002).

If suitable conditions are met, the effectiveness of RBF as a procedure for drinking water treatment can be excellent. For instance, in Berlin, the quality of bank filtrate does not require further disinfection and chlorination and has been ceased definitely in 1978 (West-Berlin) and 1992 (formerly GDR sectors) (Grohmann et al. 2000). In total, Germany uses RBF or artificial groundwater recharge for the production of 16% of the drinking water (Schubert 2002).

Because of its relatively low costs and because high-tech and highly skilled labour is not required, RBF as water purification tool is highly applicable in developing countries (Shamrukh et al. 2008).

1.1. Waterborne pathogens

Rivers receiving waste waters generated in human dwellings always contain the pathogens excreted by the infected persons who happen to be in the towns and cities at the time of discharge. Downstream users of that contaminated water are at risk of contracting those illnesses caused by the contaminating agents. The pathogenic microorganisms in the water might be multi or unicellular parasites, bacteria, and viruses. Regarding RBF the most critical pathogens are the viruses, since its small size, typically between 30 and 80 nm, makes them appear as the best candidates for trespassing the soil filter at significant rates. Therefore, the emphasis in this section will be placed on viruses only.

Worldwide contamination of drinking waters with enteric viruses such as noroviruses, rotaviruses, hepatitis viruses and enteric adenoviruses are, especially in developing countries, a huge health concern. (Ashbolt 2004; Caul et al. 1993; Hedberg et al. 1993; Muniain-Mujika et al. 2000; Van-Heerden et al. 2004). Due to the gastroenteritis caused by pathogens, the infected loose high amounts of water, what, especially in the very young, may lead to dehydration and even death. The main effects on humans are briefly described below:

Noroviruses are a major cause of acute viral gastroenteritis in all age groups. Symptoms include nausea, vomiting and abdominal cramps. Usually about 40% of infected individuals suffer from diarrhoea; some experiencing fever, chills, headache and muscular pain. The condition when the symptoms include vomiting but no diarrhoea is known as "winter vomiting disease." Infections by noroviruses induce a short-lived immunity. The symptoms are usually relatively mild and rarely last for more than 3 days. High attack rates in outbreaks indicate that the infecting dose is low.

Enteric adenoviruses cause a wide range of infections with a spectrum of clinical manifestations. These include infections of the gastrointestinal tract (gastroenteritis), the respiratory tract (acute respiratory diseases, pneumonia, pharyngoconjunctival fever), the urinary tract (cervicitis, urethritis, haemorrhagic cystitis) and the eyes (epidemic keratoconjunctivitis, also known as "shipyard eye"; pharyngoconjunctival fever, also known as "swimming pool conjunctivitis"). Different serotypes are associated with specific illnesses; for example, types 40 and 41 are the main cause of enteric illness. Adenoviruses are an important source of childhood gastroenteritis. In general, infants and children are most susceptible to adenovirus infections, and many infections are asymptomatic. High attack rates in outbreaks imply that infecting doses are low.

Hepatitis A virus (HAV) is highly infectious, and the infecting dose is considered to be low. The virus causes the disease hepatitis A, commonly known as "infectious hepatitis." Like other members of the group of enteric viruses, HAV enters the gastrointestinal tract by ingestion, where it infects epithelial cells. From here, the virus enters the bloodstream and reaches the liver, where it may cause severe damage to liver cells.

Hepatitis E virus (HEV) causes hepatitis that is in many respects similar to that caused by HAV. However, the incubation period tends to be longer (average 40 days), and infections typically have a mortality rate of up to 25% in pregnant women. In endemic regions, first infections are typically seen in young adults rather than young children. Despite evidence of antigenic variation, single infection appears to provide lifelong immunity to HEV. Global prevalence has a characteristic geographic distribution. HEV is endemic and causes clinical diseases in certain developing parts of the world, such as India, Nepal, central Asia, Mexico and parts of Africa. In many of these areas, HEV is the most important cause of viral hepatitis.

Human rotaviruses (HRVs) are the most important single cause of infant death worldwide. Typically, 50–60% of cases of acute gastroenteritis in hospitalised children throughout the world are caused by HRVs. Acute infection has an abrupt onset of severe watery diarrhoea with fever, abdominal pain and vomiting; dehydration and metabolic acidosis may develop, and the outcome may be fatal if the infection is not appropriately treated.

The short descriptions above have been taken in slightly modified form from: Guidelines for Drinking-Water Quality, 2006 WHO, Geneva.

In the study presented here, we investigated the presence of noroviruses, enteric adenoviruses, Hepatitis A and Hepatitis E viruses and viral indicators (see below) in both the Yamuna water and in the bankfiltrate.

1.2. Indicator organisms

The numbers of the pathogenic organisms present in polluted waters are vast: many of them are difficult to isolate and identify (Leclerc 2000). Therefore, indicator organisms (faecal coliforms, faecal streptococci, *E.coli*), which are more numerous and more easy to determine, are commonly used to indicate a faecal contamination of water samples.

The human intestinal tract contains countless rod-shaped coliform bacteria and their presence in an aquatic environment indicates the contamination by faecal matter but reversely their absence does not compulsory means that no pathogens are present (Grabow, 1996)

To indicate an enteric viral contamination of surface waters, bacteriophages such as somatic coliphages (Borrego et al. 1987; IAWPRC 1991; Wentsel et al. 1982), F-specific phages (Calci et al. 1998; Havelaar et al. 1984; IAWPRC 1991; Woody et al. 1995) and *Bacteroides fragilis* phages (IAWPRC 1991; Jofre et al. 1986; Lucena et al. 1996; Tartera et al. 1987) are widely used. Because of their similar size and structure to adenovirus, somatic coliphages are used as indicators in sand filtration processes (Schijven et al. 2000) (Figure 1).

Indicator bacteria and bacteriophages are usually not pathogenic; the main characteristics of the used microbial indicators are summarised in Table 1.

Table 1 Organisms that have been used as an indicator for potential pollution in this study and their main characteristics (modified from Metcalf and Eddy 2001).

Indicator organism	Characteristics
Coliform bacteria	Species of gram negative rods that may ferment lactose with gas production (or produce a distinctive colony within 24±2 h to 48±3 h incubation on suitable medium) at 35±0.5 °C. The total coliform group includes four genera in the Enterobacteria family. These are *Escherichia*, *Klebsiella*, *Citrobacter* and *Enterobacter*. Of the group, the Escherichia genus (*E.coli* species) appears to be most representative of the faecal contamination.
E.coli	The *E.coli* is one of the coliform bacteria population and is more representative of faecal sources than other coliform genera.
Enterococci	The *enterococci* are generally found in lower numbers than other indicator organisms; however they exhibit better survival in seawater
Clostridium perfringens	This is spore-forming anaerobic persistent bacteria, and its characteristic make it a desirable indicator where disinfection is employed, where pollution may occurred in the past, where the interval before analysis is protracted.
Bacteriophages Somatic phages or PRD1 bacteriophages	These organisms (20-62nm) are non- pathogenic, relatively stable in a variety of the groundwater environments and structurally similar to adenoviruses of public health concern.

E. coli is a faecal coliform bacterium that is found regularly and in high densities in human and warm-blooded animal faeces and thus, it is representative for the presence of physiologically similar enteric bacteria (Leclerc et al. 2001). Therefore, it was used as an indicator organism to examine the fate and transport potential of pathogenic bacteria through the subsurface by numerous authors (Mathess et al. 1988, Foppen and Schijven 2006).

Indicator bacteria have a limited significance for evaluating the presence or absence of viruses, since viruses often survive significantly longer in groundwater systems. Bacteriophages, which are harmless but occur in sewage polluted water in greater number than enteric viruses are used as an indicator for pathogenic viruses in this study.

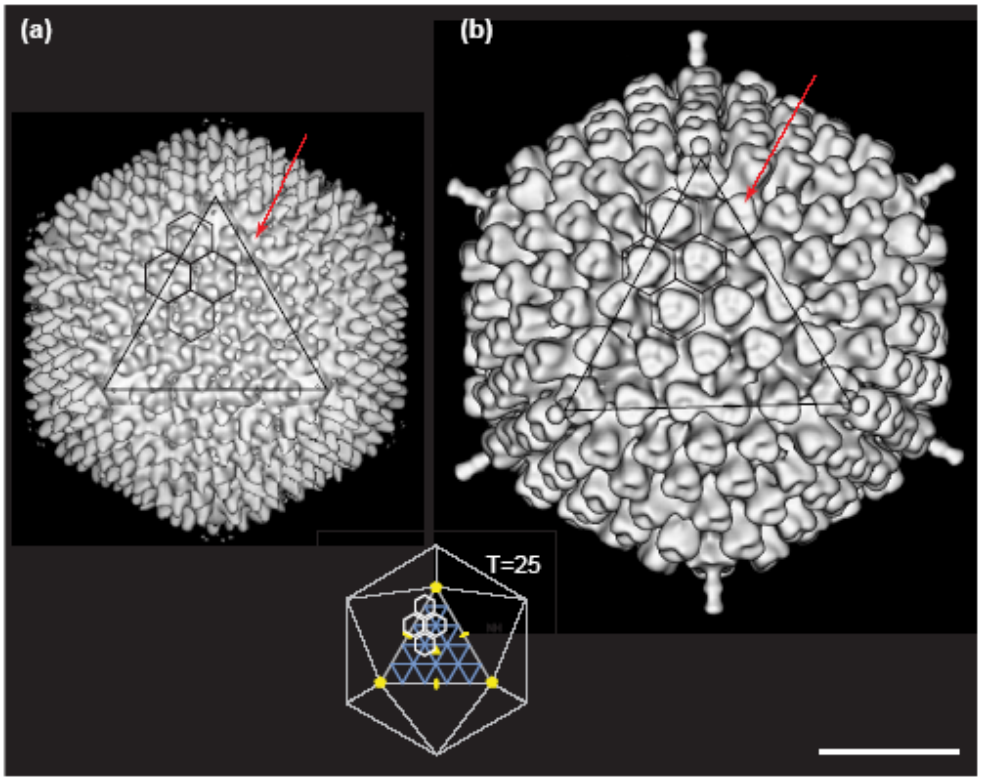

Figure 1 Comparison of bacteriophage PRD1 (a) and adenovirus (b) surface. Scale bar = 250 Å (from: Belnap and Steven 2000).

1.3. Outbreaks of waterborne infectious diseases in India

Many studies have been carried out on waterborne pathogens in India and it was found out that more than 70% of the epidemic outbreaks in India are either waterborne or water related (Kehra et al. 1996).

Sharma et al. 2003 studied the occurrence of different microbial contamination in surface-, ground- and drinking water in Delhi by means of indicators such as total coliform, faecal coliforms and faecal streptococci. The authors found that the Yamuna River had a 100 to 1000 fold increase in all indicator parameters as it passes through the city of Delhi in comparison to the water quality upstream of the city. The majority of groundwater samples (shallow and deep) were contaminated by coliforms. Since no exact sampling locations are shown in this study, it is not clear if these wells are under the influence of surface water. The source of the contamination could not be identified and it is unclear if the microbes were present in the ambient groundwater or rather originate from an insufficient well head protection. *Vibrio cholerae* O1 was shown to be present in all surface water samples, while only 5 % of the groundwater samples were contaminated by this bacterium. The authors also measured emerging pathogens such as *Vibrio cholerae* O139 and have shown that this cholera strain, though to be considered as a concern in developing countries and of documented outbreaks in Delhi in 1992 (NICD 2000), was not present in surface/groundwater samples at this time.

Sharma et al. 2003 were not able to detect the pathogenic *E.coli* O157:H7 neither in surface- nor in groundwater and explains this absence by a general geographical absence or by a presence in very low numbers.

Yersinia enterocolitica, another emerging water- and food borne pathogen has been isolated from surface water collected from sewage treatment plants in 1997/98 in Delhi (Sinha et al. 2000).

No studies have been found on other waterborne pathogens such as viz. *Cryptosporidium*, *Campylobacter jejuni* and Microsporidia in aqueous environments in India so far.

1.4. Organic trace compounds in the Delhi context

In India, rivers have been celebrated as sacred entities for thousands of years. One of the most prominent examples is the Yamuna, that is conceptualised religiously as a "divine goddess, flowing with liquid love" (Haberman 2006). Over the last decades, however, a large number of India's rivers, streams, lakes and wetlands have been polluted with all different kinds of contaminants. Rapid population growth along with agricultural revolution and industrialisation have a dramatic impact on the natural water cycle. Groundwater depletion and surface water contamination have become some of India's most urgent and significant problems. Water is extracted from ground- and surface water resources for domestic, agricultural or industrial use. In most cases, sewage (Figure 2), industrial waste and agrochemicals are discharged into drains, rivers and lakes without proper treatment (Pangare et al. 2006). Additionally, the Yamuna is subject to microbiological and organic contaminants due to its cultural and religious status: The *Times of India* (October 11th, 2008) reports for instance, that the Yamuna receives tons of toxic matters during Durga Puja festival when religious idols painted with toxic chemicals, flowers, leaves, coconut husks, clothes are immersed into the water (Figure 2). Cremations that are traditionally performed on the banks of a river have been observed regularly during this study and combustion residues are left for the next flooding along with personal belongings etc. (personal communication, P. Kumar, PhD student, IIT Delhi). In some cases (i.e. ascetics, small children or lepers and smallpox victims) dead bodies are rather immersed in a sacred river than cremated (Das 1982, Parry 1994, cited from Alley 2002). In December 2005, for instance, clothes of a child were found on the shoreline at Palla field site, along with pharmaceuticals[1] (Figure 2).

[1] including: Acetaminophen (Paracetamol), Dextromethorphan, Phenylpropanolamine, Chlorpheniramine, Levosalbutamol, Salbutamol, Ciprofloxacin (antibiotic), Promethazine, Cephalexin (antibiotic) and other substances.

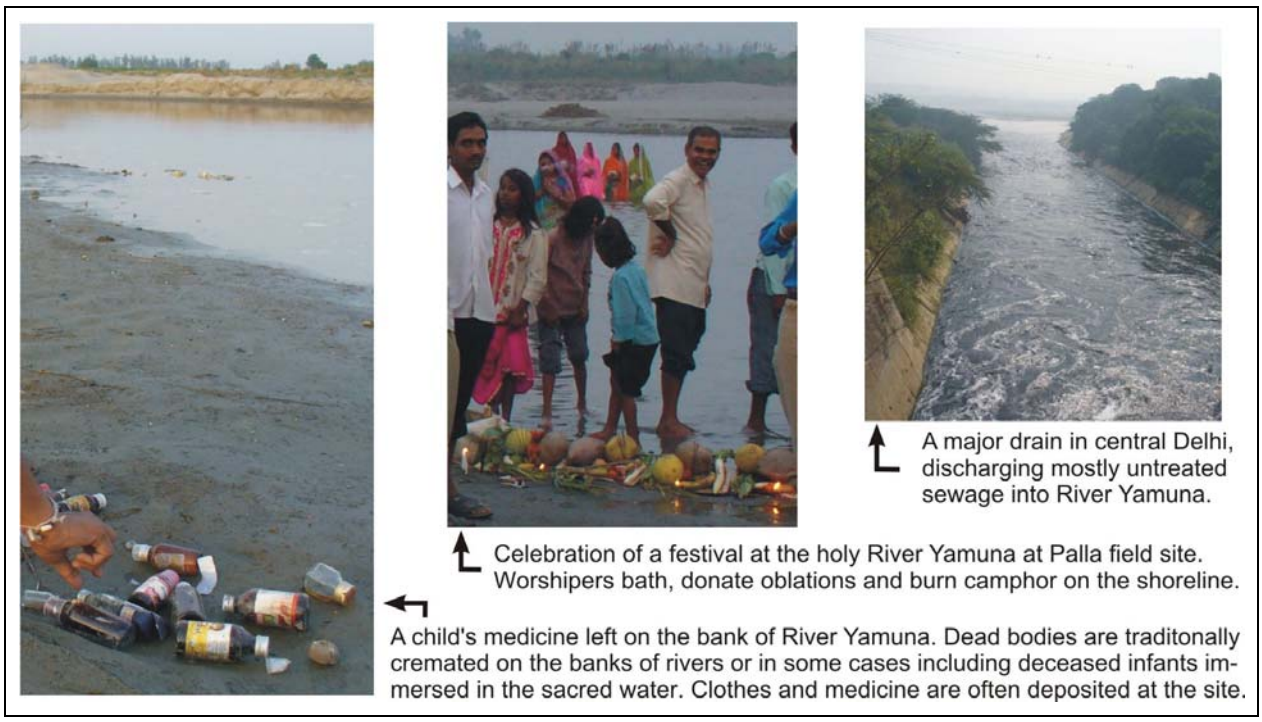

Figure 2 Pollution of Yamuna River with residual pharmaceuticals (left), the immersion of oblations during a religious ceremony (centre) and large scale discharge of untreated wastewaters in central Delhi (right).

The water quality status of River Yamuna has been subject to many investigations, testifying that it gets severely contaminated on its way from the Himalayan foothills towards the confluence with the Ganga. It reaches the worst state in the mega city of Delhi, because a major part of its discharge is extracted for the water supply of the metropolis and returned into the system as wastewater (Haberman 2006). In this stretch, that covers just about 2 % of the total length river, the Yamuna receives around 70 % of the total pollution load (CSE 2007). The wastewater reaches the river through a network of drains. Around 200 million litres of raw sewage and 20 million litres of waste are reaching the river every day mainly through the drains and reduce it to a sewage run-off (UNDP 2006). Furthermore, leachate and run-off from non engineered landfills with all sorts of non-biodegradable and toxic wastes, seeps to nearby drains and contributes to river pollution, especially during the rainy season (Zafar and Alappat 2004).

Karn and Harada (2001) identified high organic levels in the Yamuna with average annual biological oxygen demand (BOD) loads in the range of 20-25 mg/L. Values are even more critical during the dry season, when dissolved oxygen concentrations are almost zero. Kaushik et al. (2006) analysed pesticides in samples from the Yamuna in Delhi and found traces of HCH and DDT residues, with most samples being within Indian permissible limits of 1000 ng/L, but more than one third of them exceeding standards of the European commission (100 ng/L). Polycyclic aromatic hydrocarbons (PAHs) in sediments of the Yamuna in Delhi were analysed by Agarwal et al. (2006) and indicate a strong impact of urbanisation and industrialisation on the overall pollution status of the river. Some authors consider the water quality of the Yamuna River in Central Delhi to be among the worst of all rivers in India (Haberman 2006).

Such conditions offer an outstanding opportunity to study the attenuation capacity of RBF under a worst case scenario. No references have been found about

studies at comparable sites. However, promising results have been published by different authors, that have investigated the removal of organic compounds by RBF. Studies were performed with miscellaneous substances and under different field and laboratory conditions and in most cases certified an effective removal of organic trace compounds. For instance, Grünheid et al. (2005) compared the removal of bulk dissolved organic carbon (DOC) and trace organics at a BF site and an artificial recharge basin and concluded that travel time and redox conditions are the controlling factors. Herberer et al. (2004) monitored several pharmaceutically active compounds (PhACs) at BF transects and observed decreasing concentrations due to dilution and degradation or even the complete removal during subsurface passage. Sacher & Brauch (2002) analysed the degradation of micro-pollutants (industrial chemicals, pharmaceutical compounds, fuel additive) during BF and proved in laboratory experiments with test filters, that the degradation is mainly controlled by microbiology. Grützmacher et al. (2002) proved a high elimination potential for extracellular as well as cell-bound microcystins (algal toxins) during slow sand filtration processes comparable to bank filtration.

1.5. Overview of sampling locations

The sampling campaigns were carried out at three field sites, which were designed and equipped by the FUB. These field sites present a broad variety of hydrochemical and hydraulic conditions in the surface water and the adjacent aquifer. A spatial overview is given in Figure 3, more detailed information about the location and setup of the field sites can be found in TECHNEAU report D.5.2.1 and information about hydrochemical data can be found in D.5.2.2.

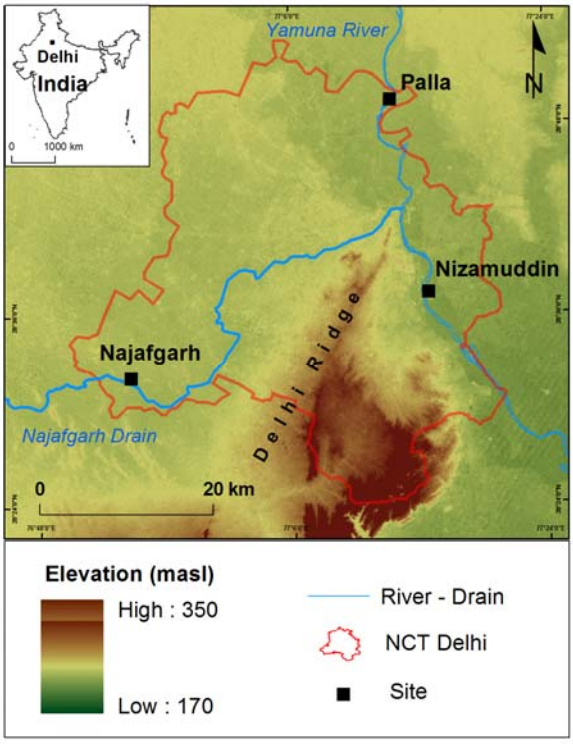

Figure 3 Location map of the three selected field sites with the geomorphology and the main river / drain.

2. Methods

In the following chapters several data sources are used to evaluate the pathogen removal efficiencies of RBF systems. Long-term measurements (May 2007 – March 2008) conducted by the IITD staff were compared with single measurements (February / March 2008) analysed by a commercial laboratory (SGS laboratory). The IITD measured total and faecal coliforms in the surface water and selected observation wells/tubewells frequently. In this report only the faecal coliform results are shown, since total coliforms are considered to be unreliable in terms of indicating faecal pollution. The SGS lab analysed several indictor parameters such as *E.coli*, coliforms, faecal streptococci and *clostridia*. The SGS results are used as a comparative value to evaluate the results from the IITD. A report from the Central Pollution Control Board (CPCB), the governmental environmental protection agency of India is used as a third source. An overview of sampling dates, involved persons, measured parameters and involved laboratories is given in Table 2. Each of the conducted field campaigns is described in detail in the following section.

Table 2 Overview of sampling campaigns for microbial and organic trace substances. SW = surfacewater, GW = groundwater, FC = faecal coliforms, TC = total coliforms, GC-MS = Gas chromatography-mass spectrometry.

Sampling date and frequency	Sampling campaign	Laboratory	Person	Parameters
May 07 - March 08 sampled monthly	SW/GW all field sites	IITD	IITD staff	FC, TC
February 2007 sampled once	SW and shallow GW at Palla, Nizamuddin	FUB	FUB staff	non-target GCMS screening
September 2007 sampled once	SW and shallow GW at Palla, Nizamuddin	UBA/IITD	UBA/FUB/IITD staff	somatic bacteriophages
				human-pathogen viruses
December 2007 - March 2008 sampled monthly	SW and shallow GW at Nizamuddin	IITD	IITD staff	somatic bacteriophages
March 2008 sampled once	SW and shallow GW at Palla, Nizamuddin and Najafgarh	SGS	FUB staff	General multiresidue screening
				E.Coli, coliforms, *streptococci*, *clostridia*

2.1. Bacteria

Total and faecal coliform analysis by the IITD

The water samples for bacteriological analysis were transported to the IITD laboratory in cooled ice boxes to keep temperature as low as possible (4°C recommended). This was sometimes challenging because the ambient temperatures often reached >30°C and the transport was very time consuming due to the heavy traffic. In most of the cases the sample was not pumped with a sterilised pump, but it was always paid attention to remove at least three volumes of the static water within the observation well. The surfacewater samples were taken with a beaker and all samples were filled immediately in prior sterilised glass bottles.

Total and faecal coliforms analysis was conducted according to the procedures of the American Public Health Association (APHA 1998). The method 9221 B is known as the standard total coliform fermentation technique and uses lauryl tryptose broth. The fermentation tubes were arranged in sets of five per sample. To statistically ensure the density of organisms, samples of 10 mL, 1 mL and 0.1 mL were used during the entire analysis. After incubating the samples for a period of 24±2 h to 48±3 h at 35±0.5 °C, development of gas and acid reaction (yellow colour) was considered as a positive indication for the presence of coliform bacteria. The results are given in MPN/100 mL (most probable number per 100 mL).

E.coli analysis by SGS

E.coli analysis was carried out according to the Indian standard IS 1622:1981 by the SGS laboratory. Results are given in cfu/100 mL.

Enterococci (faecal streptococci) by SGS

Enterococci analysis was carried out according to IS 15186/ ISO 7899-2 by the SGS laboratory. Results are given in cfu/100 mL.

Clostridium perfringens by SGS

Clostridium perfringens analysis was carried out according to ISO 7937 by the SGS laboratory. Results are given in cfu/100mL.

2.2. Viruses

Somatic bacteriophages (PRD1) by UBA/IITD/FUB

The groundwater samples were taken with previously disinfected pumps. A day before sampling, the pump with the hose and the electric wires were immersed into a disinfection solution of 1% sodium hypochlorite. In order to neutralise the disinfection solution, the whole pumping equipment was then immersed into a solution of 1% thiosulphate for two hours. In both steps, the pump was run for at least 30 minutes in order to sterilise/neutralise also the inner mechanics. The water samples for the somatic bacteriophages analysis were transported to the laboratory in cooled ice boxes to keep temperature as low as possible (4°C recommended). In order to avoid any contamination by the sampling equipment, the sampling order was always from the assumed least contaminated well to the assumed most contaminated well. Viruses are stable under dark conditions, whereas 80 – 90 % will die after a three-hour exposure to solar radiation (Gabriel et al. 1978, Gameson and Gould 1975). Therefore, immediately after filling the water into plastic (PP) bottles, these were placed into the dark ice box. In order to prevent cross-contamination of surface water and groundwater samples, both sample types were stored separately.

Human pathogenic viruses by UBA/University of Barcelona

In addition to the bacteriophages, the concentration of four human-pathogenic viruses was analysed in surface- and groundwater samples. Adenoviruses and noroviruses (type GII) were quantified by molecular methods based on the quantitative polymerase-chain reaction (qPCR) and reverse-transcriptase-PCR

(RT-PCR), respectively. Hepatitis-A and Hepatitis-E viruses were detected semi-quantitatively using PCR or nested PCR by the Department of Microbiology of the University of Barcelona. Whereas the river water was processed without being concentrated, the well water was subjected to concentration, in order to increase the chances for detection. For the concentration procedure of the well water and for the detection of adenovirus and noroviruses we proceeded according to the methods developed by the EU-project "Virobathe". The supervision of the sampling, concentration procedure and the subsequent analysis were done by Prof. Dr. Lopez-Pila and Dipl. Biol. Andreas Grunert in August 2007. Ten litres of well water, that has been conditioned by lowering the pH to 4.5, are passed through a column filled with glass wool. The viruses sorb to the glass wool and are retained inside the column. With the aid of a proteinaceous solution at a pH of 9.5, the viruses are eluted from the glass wool into a volume of 200 ml. This volume is reduced even further by precipitating the viruses and collecting the precipitate by centrifugation. At the end of the procedure, the viruses which were present in 10 litres of water are concentrated in 10 ml.

2.3. Organic trace compounds

At the beginning of the monthly sampling campaigns within WP 5.2 of the TECHNEAU project, IITD reported technical problems regarding the analysis of organic trace substances. The procedure of sample processing was not established yet and the recently acquired Gas Chromatograph Mass Spectrometer (GC-MS) was not ready for operation. Consequently, a non target analysis for organic compounds was initialised by FUB team, with the aim to get an overview on contamination load by the qualitative identification of critical substances.

In the last field campaign, when there was still no data available from the Indian cooperation partner, it was decided by KWB and FUB, to perform a survey for at least one quantitative analysis for surface water samples and bank filtrate at each field site. A commercially operating local laboratory (SGS, Gurgaon) was assigned for the qualitative analysis of pesticides and specific organic trace substances.

Each of these analysis could only be made for one sampling campaign, so the results will represent only snapshots and not necessarily typical values. In the groundwater or bank filtrate, peaks will be levelled off due to dispersion or mixing and represent the input of a longer period. In the surface, in contrast, the contamination input may be the result of short time events or fluctuate seasonally or even daily. This is especially important to consider at Palla field site, where the Yamuna has a high flow velocity. At Nizamuddin Bridge and Najafgarh Drain in contrast, short term fluctuations are less probable, because relatively low discharge in relatively broad river beds lead to longer residence times.

GC-MS screening (qualitative non target analysis) by FUB

For each sample, a volume of at least 1 L is necessary. The sample bottle should be cooled down to about 4°C immediately after sampling and brought to the laboratory for further processing as soon as possible. Therefore, it was decided to take only four samples in the last days of the sampling campaign of February 2007 and bring them to FUB laboratory by airplane. Two samples were taken to analyse the contamination load of the Yamuna River, one at the Palla field site (PA-SW) upstream the urban parts of Delhi and one at Nizamuddin Bridge (NI-SW) in the centre of the Mega City. Additionally, at each of these two sites a sample was

pumped from a shallow piezometer with high bank filtration share (PA-PZ-4 and NI-PZ-1). A specification of the locations, sampling well design and sampling procedure is given in TECHNEAU deliverable D5.2.1 and D5.2.2. Due to unexpected transport delays, the samples arrived in Berlin more than 48 hours late and without proper cooling. The samples were brought to the Environmental Organic Geochemistry at Department of Earth Sciences at FUB and cooled down immediately for conservation.

For the GC-MS analysis, the organic compounds must be in a solution of a volatile and organic solvent (Hites 1997). The preparation of the samples and extraction was performed as follows: Each sample was filtered through a 1.2 µm glass fibre membrane (Whatman GF/C, previously extracted with acetone/hexane) and transferred into a separating funnel. For the sequential extraction of three fractions, three different solvent were used:

- Fraction F1 (non polar): 50 ml pentane
- Fraction F2 (medium polar): 50 ml DCM (dichloromethane)
- Fraction F3 (polar): 50 ml DCM with HCl suprapure (to adjust a pH of ~ 2)

After adding the solvent, the funnels were agitated intensely for at least five minutes and than left for phase separation (figure 3). After about 20 minutes the solvent was transferred to a 100 ml injection plunger and 10g Na_2SO_4 were added to remove the residual water from the extraction. The fractions were concentrated with nitrogen gas. Fraction F1 and F2 were directly vialed (final volume 100 µl). For fraction F3 a derivatization with TMSH (Trimethylsulfonium Hydroxide) before the final volume (100 µl) was vialed. Before the measurement, internal standards werde added to the samples, including deuterated molecules of dichlorobenzene, acenaphtene, chrysene, perylene and naphtalene.

For each fraction of the four samples, a qualitative screening was performed with a gas-chromatography–mass spectrometry (GC–MS) on a Hewlett-Packard 5890 II GC coupled to a Hewlett-Packard MSD 5971 A (Hewlett-Packard, Palo Alto, USA). For the gas chromatographic run, an operation time of 100 min was fixed.

In some cases, characteristic peaks at a specific retention time are already adequate to identify certain substances. For instance, in the F1 fraction of the NI-SW sample, a huge peak after around 44 minutes indicates elemental sulphur (S1). For a more detailed analysis, relevant GC peak maxima were identified at specific retention time steps to plot the corresponding mass spectra (figure 3). Therefore the normalised ion abundance was plotted in bars over the mass-to-charge (m/z) ratio. The characteristic patterns were compared with the mass spectra of specific organic substances collected in the WILE 275 library, a database with more than 275,000 entries. For this step, an automated library search was carried out for each fraction, with the default settings of the HP Chemstation Data Analyzer software. In the library search report, the best fitting matches for each peak are indicated, with information on the name of the substance, Chemical Abstracts Service (CAS) Registry Number, relative extent of the corresponding peak (area %) and match quality. A summary of each report, including only the best fitting match for each peak and only those with a match quality better than 75 % are given in the appendix of this report. An additional column was added for further information and comments on the substances. The search results, however, have to be interpreted with caution, because precise quality control could not be realised for each matcht, within the frame of this investigation.

For the interpretation of the findings of the screening, the probability of secondary contamination has to be assessed. With the highly sensitive method, even smallest amounts of organics released into the sample during sampling, storage, processing of the sample or from the analytical instrument itself may cause significant peaks and lead to misinterpretations. Typical examples are considerable peaks from plasticisers, released from the tube of a piezometer or sampling equipment or siloxanes from the inner membranes of the GC.

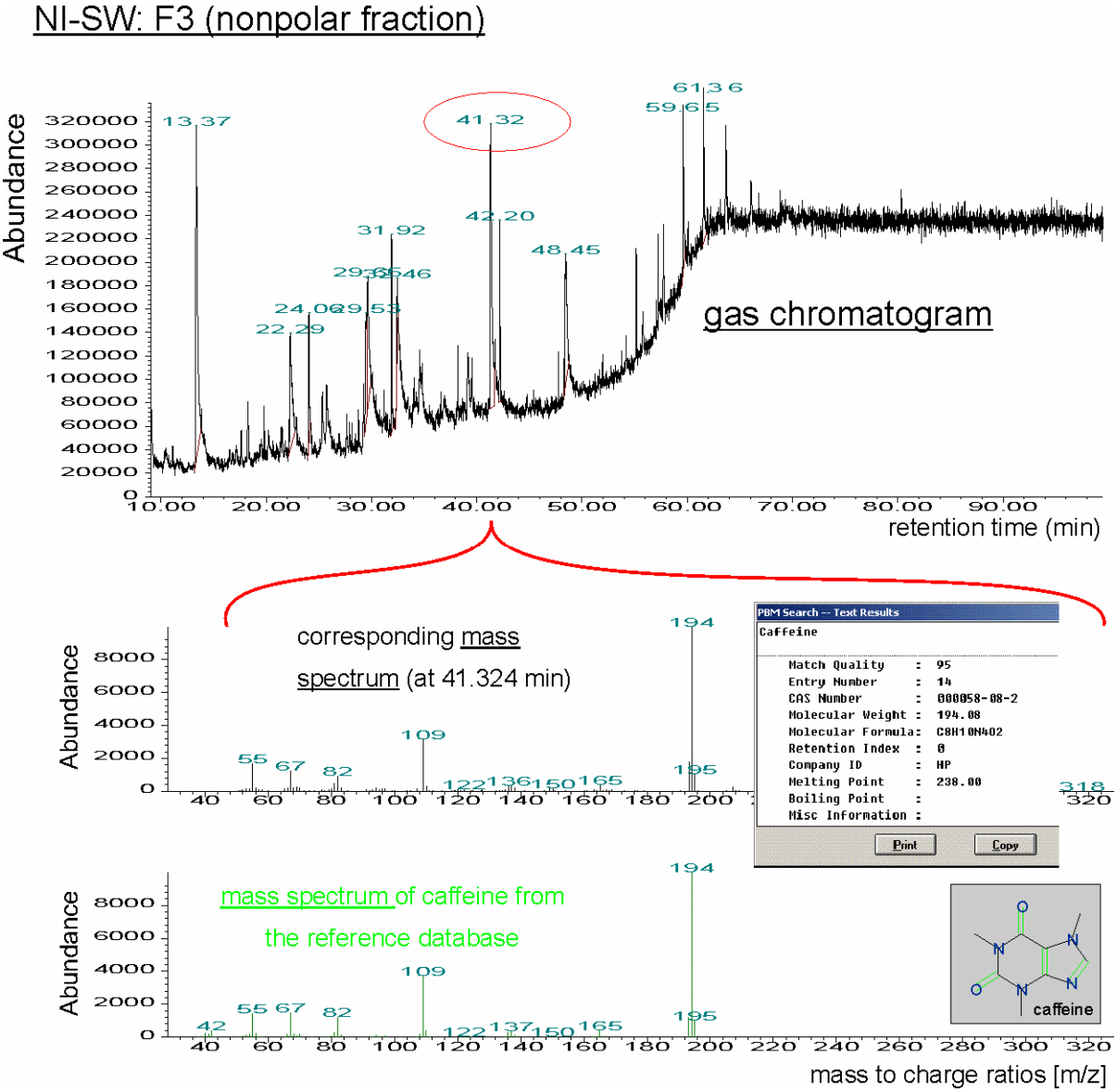

Figure 4 Example of the GC-MS based identification of a specific substance in the Yamuna River Water: The gas chromatic scan (above) represents the nonpolar fraction (F3) of the NI-SW sample. The mass spectrum for one specific peak at a retention time of 41.32 minutes (middle) is compared to the one of caffeine (below) as given in the reference database.

Quantitative analysis of specific trace organics by SGS

For the quantitative analysis, a number of specific organic trace contaminants have been selected, in order to identify the contamination load and attenuation by RBF.

Further, concentrations were compared to the limits of the Indian standard specifications for drinking water (IS 10500) and WHO (2004) guidelines. The samples were taken with a submersible pump and filled into 1L glass bottles. The bottles were immediately secured into a portable cool box with ice packs, brought to a refrigerator within less than four hours and analysed within less than two weeks after sampling. The samples were analysed in the facilities of SGS laboratory in Gurgaon, Haryana. The commercially operating laboratory is accredited by the National Accreditation Board for Testing and Calibration Laboratories of the Department of Science & Technology, India. A summary of the parameters, methods and detection limits is given in Table 3.

The laboratory is equipped with the following analytical instruments:

- GC-MS/MS (VARIAN-IonTrap), Agilent- Quadrouple (GC-MS)
- LCMS/MS(ThermoFinnigan)
- GC –Agilent-microECD (6890N),NUCON-FID
- HPLC–DAD(LACHROM-MERCK-HITACHI),AGILENT,WATER's-Fluorescence
- Solid phase extraction unit –Varian.
- Software -HSM SYSTEM MANAGER, SATURN MS WORKSTATION, CHEMSTATION

Table 3 Selection of organic substances for quantitative analysis, detection limits and drinking water standards.

Parameter / substance	detection limit	Drinking water standards	
		Indian standard IS: 10500	WHO guideline (2004)
Mineral oil	0.01 mg/L	Desirable 0.01 mg/L Permissable 0.03 mg/L	-
Anionic detergents as as Methylene Blue Active Substances (MBAS)	0.05 mg/L	Desirable 0.2 Permissable 1.0 mg/L	-
Phenolic compounds (as C6H5OH)	0.001 mg/L	Desirable 0.001; Permissable 0.002 mg/L	-
Benzo(α)pyrene	0.1 mg/L	-	0.7 µg/L
Benzene	0.1 µg/L	-	0.01 mg/L
Epichlorohydrine	0.01 µg/L	-	0.4 µg/L
1,2-Dichloroethane	0.1 µg/L	-	0.03 mg/L
Tetrachloroethane	0.1 µg/L	-	0.04 mg/L
Trichloroethane	0.1 µg/L	-	0.07 mg/L
Vinyl chloride	0.1 µg/L	-	0.3 µg/L
Trihalomethanes (Total)	0.1 µg/L	-	i.e. 0.2 mg/L for Chloroform
Polynuclear aromatic hydrocarbons (PAHs)	0.1 mg/L	-	-
General multiresidue screening = Pesticides (61	0.1 µg/L	Desirable absent;	-

different substances)*		Permissable 0.001 mg/L	
*Alpha-BHC, Beta-BHC, Lindane (gamma BHC), Delta BHC, o, p-DDD, o, p DDEo, p' DDT, p,p' DDD, p,p' DDE, p,p' DDT, Endodulphan I, Endodulphan II, Endosulphate sulphate, Aldrin, Dieldrin, Heptachlor, Heptachlor epoxide, Endrin, Methoxychlor, Alachlor, Butachlor, Chlordane, Dicofol, Hexachlorobenzene (HCB), Chlopyrifos, Chlopyrifos methyl, Phosphamidon, fenitrothion, Parathion, Malathion, Methyl paraoxon, Methyl parathion, Malaoxon, Dimethoate, Phosalone, Quinalophos, Primiphos methyl, monocrotophos, Fenchion, Phorate, Phorate sulphone, Phorate sulphoxide, Isoproturon, Methidathion, Dichlorvos, Carbofenotion, Clofenvinfos, Diazinon, Azinphos methyl, Tri azophos, Ethion, Cyfluthrin, Cypermethrin, Deltamehrin, Esfenvalerate, Lambda cyhalothrin, Premethrin, Carbaryl, Carbofuran, Aldicarb, Methomyl			

Samples for the quantitative analysis were taken from each of the three field sites, including one surface water sample (PA-SW, NI-SW, NA-SW) and at least one sample from a shallow piezometer in March 2008 (see Table 2). A detailed description of well designs and locations is available from D 5.2.1. At Palla field site, the sample was extracted from piezometer number PA-PZ-2, with high bank filtration share and a similar depth and distance to the river, than the PZ-4 that had been chosen for the non target screening described above. At Nizamuddin Field site, groundwater samples with high bank filtration shares were taken very close from the shoreline from NI-PZ-2a and NI-PZ-2c (figure 5) and from NI-PZ-2 that has an equal distance to the river than the piezometer that was chosen for the non target analysis (NI-PZ-1). At the Najafgarh Drain, the ground water sample was taken from the NA-PZ-1 piezometer. This sample does not necessarily represent bank filtrate, because at this site loosing river condition prevails only a few month a year during dry season.

3. Results

3.1. Palla well field

Bacteriological analysis

At the Palla field site the coliform concentrations analysed by the IITD in the Yamuna River show a mean concentration of 7 x 10^3 mpn/100 mL, again without any significant difference between non-monsoon and monsoon time (Figure 5). The coliform concentrations measured by the SGS lab (Table 4) are in the same order of magnitude (1.2 x 10^3 cfu/100 mL) and support the IITD results, but again the IITD results show slightly increased concentrations.

In the groundwater samples (PZ2), however, the results from the labs differ significantly – the IITD always measured faecal coliforms in the range between 6 – 64 mpn/100 mL, while the SGS lab results are below the detection limit (<1 cfu/100 mL). This can be explained by contamination which possibly occurred during sampling and/or during analytical procedures in the lab. It has to be considered, that the samples for SGS lab were taken with caution to avoid any secondary contamination. All equipment, including the pump and hose was sterilised and only one or two groundwater samples were taken per day. When two samples were taken, the less contaminated observation well was pumped first, to ensure that residual water would not contaminate the subsequent sample. The IITD samples, in contrast, were taken under time pressure by students, with up to 7 samples a day including in situ measurements and quick tests.

The faecal coliform count in the tubewell are always below the detection limit.

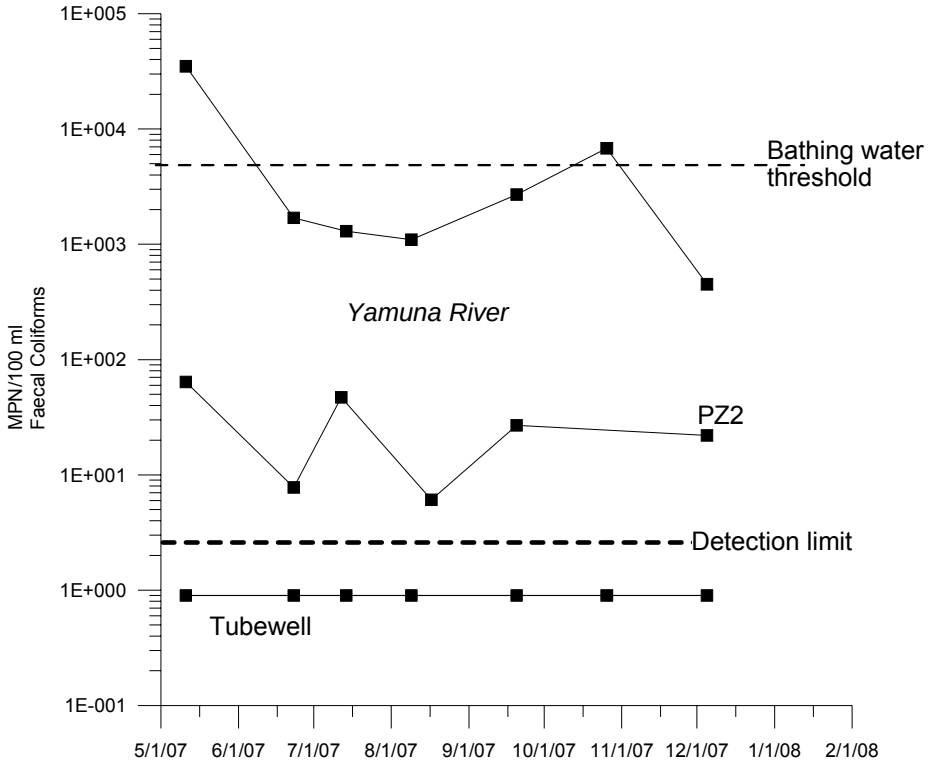

Figure 5 Faecal coliforms at the Palla well field analysed by the IITD.

Table 4 Microbial parameters analysed by the SGS laboratory.

Observation point	*E.coli* (cfu/100 mL)	Coliform (cfu/100 mL)	*Enterococci* (feacal streptococci) (cfu/100 mL)	*Clostridia* (cfu/mL)
Yamuna River	4.00E+02	1.20E+03	20	<1
PZ-2	<1	<1	<1	<1

Organic trace substances: non target screening

The results of the non target analysis at Palla Well Field are plotted in Figure 6. The three plots show the results of the GC scan from each of the three fractions of two samples. The red lines in the background of each graph show the results for the Yamuna river water. The black lines in the foreground represent the sample from the shallow piezometer, with a very high share of bank filtrate (see D 5.2.2).

In the diagrams, the number of major peaks from the baseline is relatively limited, especially in the F1 and F2 fractions. The MS analysis shows that except from the internal standards, most of these peaks are caused by the presence of traces of alkane hydrocarbons (paraffins), phenoles and phthalates (DIPB, DBP, DEHO).

Alkanes may occur in nature in various ways, but in a densely populated region like the Gangetic plain their increased concentrations must be related to anthropogenic pollution like oil and gas. Their source in the Yamuna river may be spills of fuel, motor oil or lubricants from industry, households, tanks, vehicles or agriculture i.e irrigation pumps extracting water from the rivers and canals. Traces of alkanes extracted with the water sample from the shallow piezometer, however, may also be residues of motor oil and lubricants, that were unavoidably contaminating the boring sludge during the drilling of the piezometers. This is probably the origin of the alkanes in the F1 fraction and may contribute to those of the F2 fraction. Consequently, it is not possible to evaluate whether the alkanes present in the Yamuna water (F2 fraction) are attenuated during RBF.

Phthalate esthers have been used for more than 40 years and are among the most common industrial chemicals with a yearly production of some million tons worldwide (Fromme et al. 2002). They are most commonly used as plasticisers in the production of resins (especially PVC) and other materials, but also as industrial solvents and lubricants, in pesticide formulations, as additives in the textile industry and in personal care products (Koch et al. 2003). They can enter the environment through leaching from final products or losses during manufacturing processes and are considered ubiquitous in the environment and tend to bioaccumulate in animal fat (Fromme et al. 2002, Jobling 1995). Due to their persistence, they are even found in the effluent of modern wastewater treatment plants (Jackson & Sutton 2008). Phtalates show low acute toxicity, but many of them (also DBP and DEHP) and their metabolites are suspected of having chronic effects including teratogenic and endocrine disrupting effects including estrogenic activity (Fromme et al. 2002, Koch et al. 2003).

Phthalates are also known as frequent laboratory and sampling contaminants (Jackson & Sutton 2008). Possible sources of secondary contamination are (i) the PVC piping[2] of the piezometers (ii) and unknown solvent that was used by the drilling company to agglutinate piping units and (iii) the flexible hose, that was connected to the (iv) submersible plastic pump for groundwater extraction. Considering that the abundance in the bank filtrate sample is even much higher than that from the Yamuna water it is impossible to conclude whether these substances are removed during RBF.

[2] *For the analysis of organic compunds, it is recommended to built piezometers with metal pipes. Contrariwise, for the analysis of inorganic ions, which are a crucial aspect of this study, PVC pipes are favourable.*

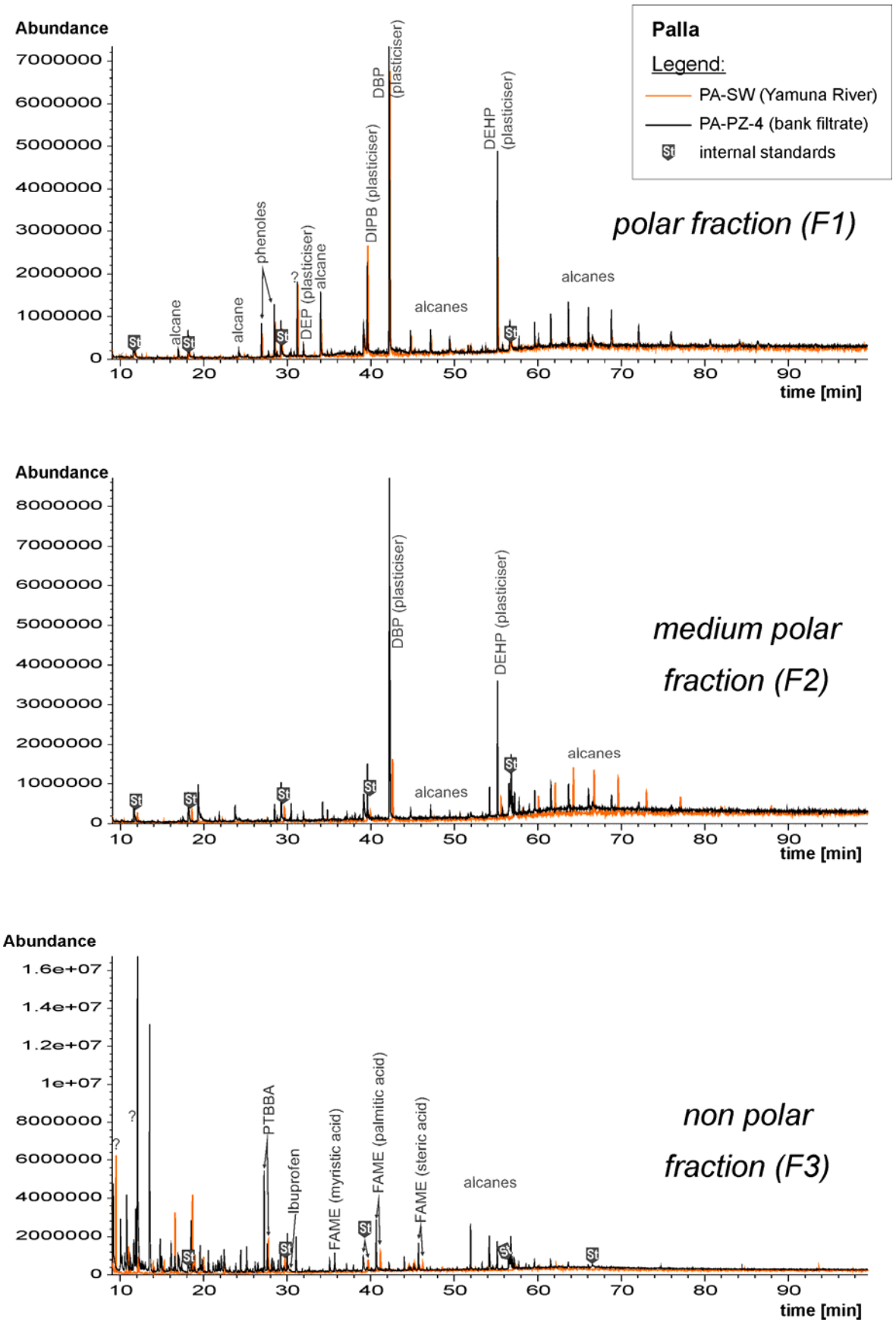

Figure 6 Trace organic compounds at the Palla field site: The three sections prepared for the GC-MS-screening are plotted separately. The sample from the piezometer with high bank filtration share is shown in the foreground, with the surface water in the back.

In the F3 fraction a series of additional peaks indicates the presence of several polar contaminants. Several peaks within below the retention time of 20 minutes could not be related with any of of the WILEY 275 reference database (275.000 entries).

Fatty acids like palmitic acid, oleic acid, stearic acid, myristic acid or palmitoleic acid for instance are the major components of vegetable and animal fats like butter fat, olive oil, palm oil, or soy oil. The fatty acids are separated with the gas chromatograph in form of their derivates (i.e. fatty acid methyl esthers, **FAME**). Apart from food ingredients and food preparation using frying oil, many fatty acids are used as ingredients in personal care products or other applications. For instance, steric acid is applied in the production of candles, plastics, cosmetics, as a softener for rubber or to harden soaps. The occurence in the Yamuna water probably originates from untreated sewage effluents. Surprisingly, the contents in the bank filtrate samples are higher than those in the surface water. As concentrations in the bank filtrate represent medium values of attenuated input from the river, it is assumed that medium values of the surface water are much higher in general.

The traceability of **ibuprofen,** in the Yamuna river upstream Delhi is remarkable. It is one of the most common non-steroidal anti-inflammatory drugs (NSAID) with diverse healing applications and widely sold in India under the brand name *Brufen*. In the groundwater, no traces of this pharmaceutical were found, so if it is commonly present in the river, it must be removed effectively by RBF.

Organic trace substances: quantitative analysis

The concentrations of all pesticides and other substances mentioned in Table 3 remain below detection limit in both samples. However, it has to be considered, that the sample from the Yamuna River is presenting only a snapshot. The Yamuna has a considerable flow velocity at Palla. Consequently, it can not be excluded, that the river discharges significant amounts of one or more of the analysed contaminants in different seasons or on other days. In contrast to the surface water, the concentrations of contaminants in the bank filtrate represent averaged values as a consequence of dispersion and mixing. The fact that the bank filtrate is free of these substances demonstrates that they are either absent in the surface water for several days or weeks or attenuated sufficiently by bank filtration processes.

3.2. Nizamuddin Bridge

Bacteriological analysis

The variation of the faecal coliform concentration in the Yamuna River ranges from 4.5 x 10^5 - 9.2 x 10^7 mpn/100 mL, without any significant difference between monsoon and non-monsoon time with an average for both seasons of 1.6 x 10^7 mpn/100 mL (Figure 7). Literature data from CPCB 2006 reports an annual average concentration of faecal coliforms in the range of 1 – 8 x 10^6 mpn/100mL. The SGS laboratory analysed coliforms in the range of 1.5 x 10^5 cfu/100mL (Table 5). It is obvious that the analytical results from the IITD laboratory of the Yamuna River show higher concentrations than the comparative values from the SGS lab or from CPCB 2006.

The faecal coliform concentrations in the groundwater from both laboratories (IITD and SGS) are in the same order of magnitude. Despite of all the differences between measuring methods, frequency of measurements and sampling techniques a secondary contamination of the samples from the IITD is most likely. This secondary contamination can be derived (i) from the ground surface where cattle and birds are in close proximity to the unprotected wellheads or (ii) from contamination by the sampling equipment itself or (iii) from cross contamination in the laboratory. Since the sampling for the SGS lab was conducted under strict conditions a contamination by the sampling equipment seems to be not likely. Therefore, a direct contamination by animals (i.e. birds) in combination with cross contamination in the IITD lab is assumed. This underlines the importance of a good designed observation well and a sterile analytical procedure.

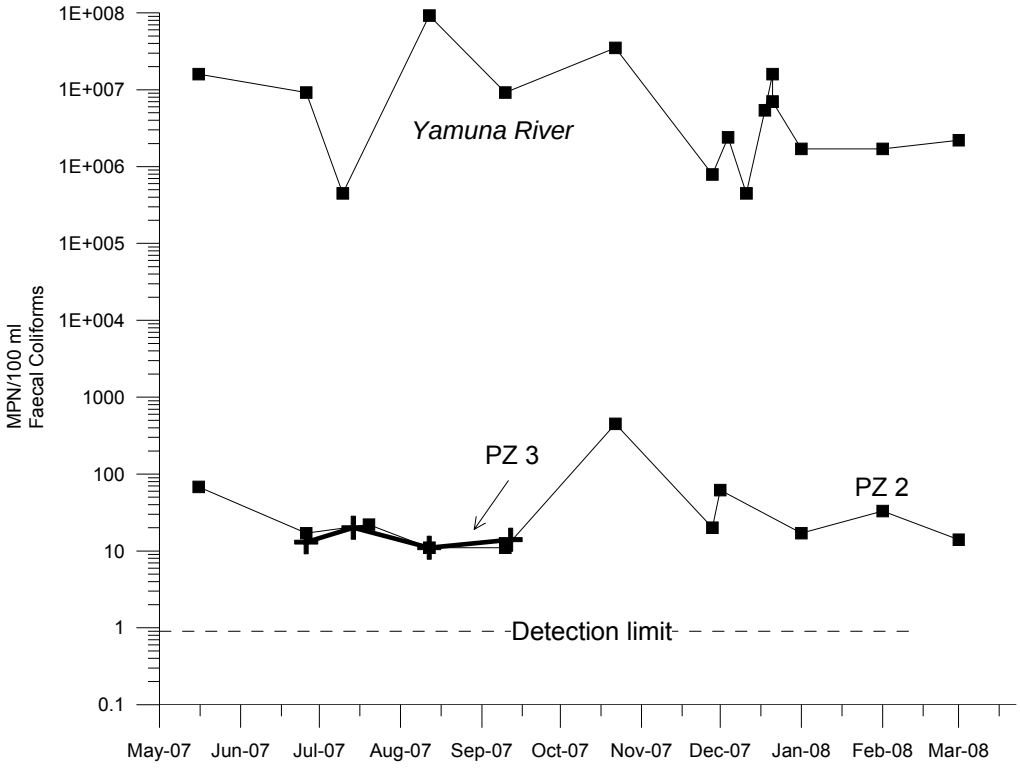

Figure 7 Faecal coliforms at the Nizamuddin site against time (analysed by IITD).

Moreover, *E.coli* concentrations of different observation wells, with different distances from the river and with different depth of the filter screen, are in the

same order of magnitude which seems unlikely. Observation well PZ-3, for instance, has its sampling depth between 31 and 37 mbgl. Regarding aquifer properties, hydraulic conditions and water chemistry it is not supposed to contain any bank filtrate or recently recharged water, thus faecal coliforms are not expected to be present at all.

Table 5 Microbial parameters analysed by the SGS laboratory.

Observation point	*E.coli* (cfu/100 mL)	Coliform (cfu/100 mL)	*Enterococci* (feacal streptococci) (cfu/100 mL)	*Clostridia* (cfu/mL)
Yamuna River	1.20E+05	1.50E+05	5000	<1
PZ-2a	6.80E+04	8.00E+04	3600	<1
PZ-2c	5.00E+02	4.00E+02	<100	<1
PZ-2	2.00E+02	1.80E+03	<100	<1

Despite of all the uncertainties of the IITD data, it is concluded that the peak of faecal coliform concentration in October 2007 (Figure 7) can be seen as increased background contamination from the monsoonal flood from the previous month. During this event the floodplain around the field site was inundated, so the flow path and residence time of the infiltrating river water was shortened significantly.

Virological content

After almost one year of sampling, a second drilling campaign was carried out at the end of the year 2007 (supported by Dr. A. K. Mittal (IITD)). Three shallow piezometers were built up at the Nizamuddin Bridge field site (Figure 8), to investigate the fate of bacteriophages during the first meters of subsurface passage. The analysis of bacteriophages were initially conducted by UBA staff (Prof. Lopez-Pila and Dipl. Biol. Andreas Grunert) and later continued by the IITD (MSc. Medalson Ronghang). This data gives realistic removal rates for bacteriophages under the given field conditions.

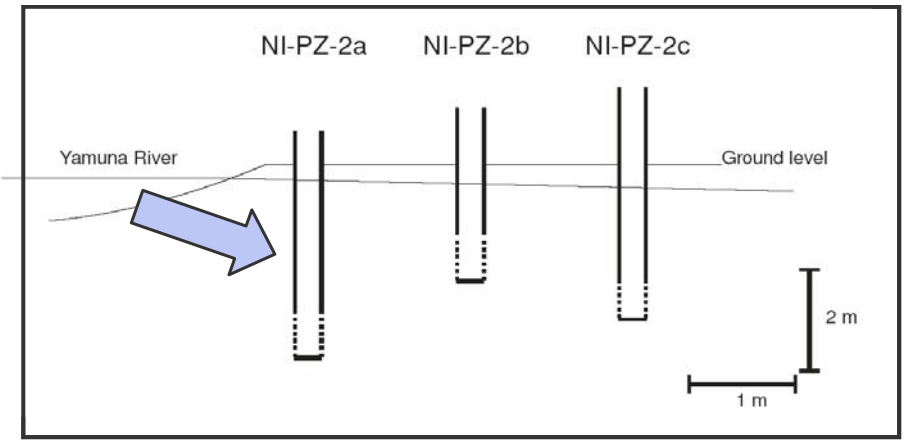

Figure 8 Observation wells at the bank of the Yamuna River (the blue arrow indicates influent hydraulic conditions).

A detailed and comprehensive overview of processes affecting the removal of viruses during subsurface passage is given by Schijven and Hassanizadeh 2000. The removal of viruses is defined as the logarithmic reduction of virus concentration ($\log_{10} C/C_0$). The main removal processes are reversible adsorption /desorption processes and inactivation of both free and adsorbed viruses (Schijven and Hassanizadeh 2000). Advection and dispersion processes transport the virus (and other bio-colloids) in the porous media. Adsorption of viruses to the grain surface will take place when both phases are oppositely charged.

In the Yamuna River somatic bacteriophages in the range of $1.2*10^5$ pfu/100mL were found. In Berlin i.e. the concentration of somatic bacteriophages in the surface water of the lake Wannsee were given with a maximum of 800 pfu/100 mL (Lopez-Pila and Szewzyk 2002). The high concentrations of bacteriophages in the river allowed to make an estimate of the elimination of viruses "*in situ*", without having to resort to artifially spiking the site, as such estimates are usually carried out. To our knowledge, this opportunity to observe the authentic removal performance of RBF is unique.

Generally, most surfaces of grains in the aquifer matrix are unfavourable for the attachment of viruses, since both are negatively charged (Ryan and Elimelech 1996). Surface charge heterogeneities of the grains, expressed as positively charged patches in a negatively charged area, provide favourable sites for virus attachment (Schijven and Hassanizadeh 2000). Iron- , aluminium- and manganeseoxides are considered to be the most common sources of surface heterogeneities and are present in many aquifers as surface coatings of the grains. Series of sequential extraction procedure, where different binding forms of arsenic were investigated, carried out with soil samples from the Nizamuddin site revealed that most Fe was present in the aquifer as amorphous (or weak crystalline) ferrihydrite. A smaller fraction of the Fe-oxides is present in the crystalline form of goethite or lepidocrocite. The isoelectric point is the pH range where a surface positive and negative charge of a mineral phase is in equilibrium. Mineral phases with a high isoelectric point are better virus adsorbents then those with a low isoelectric point (Gerba 1984). The isoelectric point of goethite is in the range of 7.6 – 8.1 and lepidocrocite 7.8 – 8 (Parks 1962), which means that under the given pH range of 7 – 7.2 positive charged surface heterogeneities will enhance attachment of virus colloids to the grain surface.

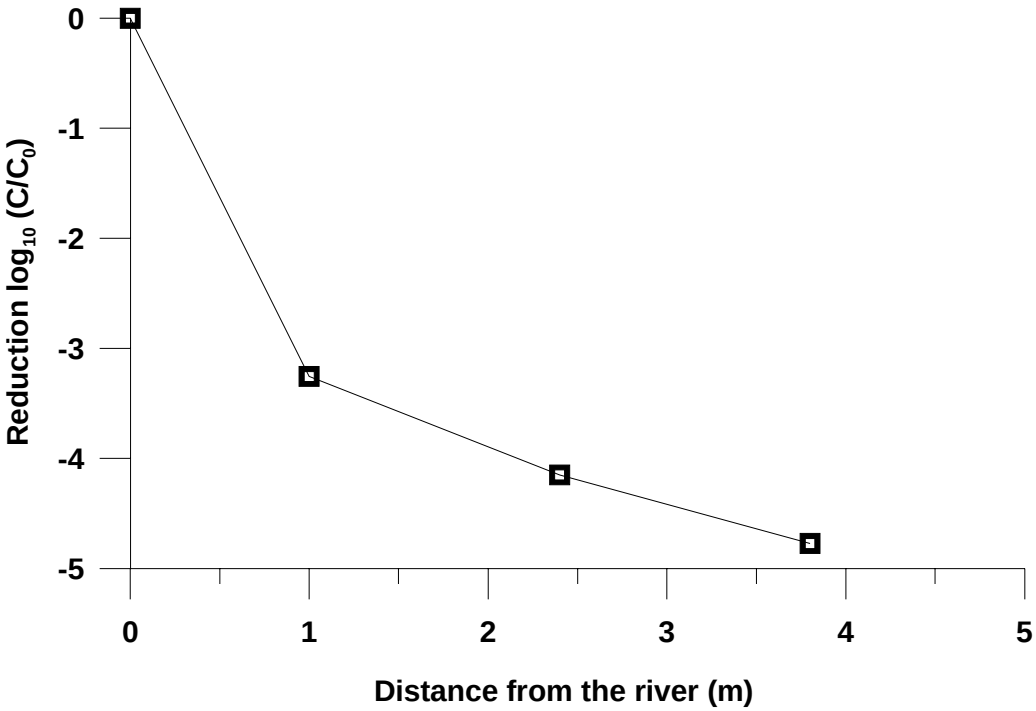

Figure 9 Log reduction of somatic coliphages against distance (in meters).

Removal of bacteriophages is not linear with distance (Figure 9). During the first meter of subsurface passage 3.3 $\log_{10}$ removal is achieved, while at the following 3 m only 1.5 $\log_{10}$ removal can be observed. Dilution effects can be excluded since conservative tracer calibrated hydraulic modelling indicate 100% proportion of bankfiltrate at the sampled observation wells. The observed initial higher removal of viruses is found in several other field and column studies and can be explained by soil and/or virus heterogeneities (Schijven and Hassanizadeh 2000). Soil heterogeneities like larger proportion of silt and clay seems unlikely to be taken into account for higher removals since Schijven et al. 1999 reported of even lesser proportion of clay and silt during dune recharge at the first meter with an increased initial removal rate. Soil heterogeneities in terms of more available attachment sites for viruses at the first meters or centimetres of infiltration may be the reason. Unfortunately, no soil analysis of the clogging layer of the Yamuna River at the Nizamuddin Bridge is available. Anyhow, extensive soil analyses of the sediments from drilling campaigns of the observation wells at this field site suggest that organic matter is distributed in small lenses up to 3 % fraction of the total mass (Table 6). It seems likely that the presence of the organic rich clogging layer at the riverbed is removing viruses by hydrophobic interactions more effectively than during the following passage. Other studies like Schijven et al. 1999 found 3 $\log_{10}$ removal in a field study after 2.4 m flow distance in a dune recharge experiment with higher flow velocities, under oxic conditions and lesser f_{OC} than at the Nizamuddin site. Recent studies of bacteriophages removal at an injection experiment on field scale states that virus removal was considerable lower under anoxic conditions than under oxic conditions (van der Wielen et al. 2008). In this study the authors argue that under the considered worst case scenario of a leaking sewer in an anoxic aquifer, with relatively high pH and a shallow abstraction well, the groundwater protection zone of 50 – 60 days should be extended to 110 days to meet the infection risks regulations. This finding cannot be confirmed from this field study since the removal rates under anoxic

conditions at the Nizamuddin site are much higher than at the field site from van der Wielen et al. 2008. These higher removal rates may be attributed to an increased content of bonded organic matter, higher proportion of iron hydroxides and/or lower effective porosities.

However, a linear regression line calculated only with measurements from the observation wells shows a high correlation (r^2=0.82) and supports the idea that removal follows a linear trend after the passage of the riverbed.

Virus-soil interactions are affected by changes in pH, ionic strength, multivalent ions and organic matter (Gerba 1984). Table 6 gives hydrological and chemical parameters affecting the removal capacity of pathogens and provides the possibility of comparison with other field studies. The Nizamuddin field site is characterised by anoxic conditions. Ammonia is infiltrating into the aquifer through riverbank filtration in considerable amounts, while nitrate is present only during monsoon time when dissolved oxygen reaches levels of 1-2 mg/L.

Table 6 Hydrological and chemical properties of the studied aquifer at Nizamuddin Bridge (DO = Dissolved oxygen, f OC = fraction of organic matter, EC = electrical conductivity, DOC=dissolved organic carbon, mbgl = meters below ground level).

	Nizamuddin (0-2 mbgl)
Grain size d_{50} (mm)	0.4
Porosity	~ 0.2
Clay (%)	1
Silt (%)	5
Sand (%)	94
Gravel (%)	0
Hydraulic conductivity (m/d)	~ 35
Pore water velocity (m/d)	~ 1.1
f_{OC} (%)	0.3 - 2
Surface Fe (III) (g/kg)	~ 2
pH	7 - 7.2
EC (µS/cm)	800 - 1500
Temp (°C)	17 - 24
DO (mg/L)	0 - 1
Nitrate (mM)	0
Ammonia (mM)	0.3 - 0.8
Sulfate (mM)	0.05 - 0.75
HCO_3^- (mM)	9 - 11
DOC (mg/L)	4.4 - 5.7
Ca^{2+} (mM)	1.7 - 2.2
Mg^{2+} (mM)	1.3 - 1.5

Human pathogenic viruses

Usually adenoviruses excreted in human faeces and urine, are far more abundant in sewage than any other type of enteric virus (Metcalf et al. 1995). High numbers of adenoviruses and noroviruses have been found in the Yamuna River (Table 7) at the Nizamuddin Bridge, but none in the observation well in 50 m distance from the river. Please note that the observation well water was subject to concentration steps (approximately 1000 fold) prior to attempting the detection of human pathogenic viruses.

Adenoviruses are endemic worldwide and are found in raw sewage in magnitudes of 10^5 - 10^6 genome copies/100mL (He and Jiang 2005).

Table 7 Human pathogenic viruses in the Yamuna River and the PZ2 (travel time approx. 50 d).

Observation point	Adenoviruses	Noroviruses	Hepatitis A*	Hepatitis E*
Yamuna River	$3.6*10^4$ (genome copies/100mL)	$5.4*10^4$ (genome copies/100mL)	positive in 100 mL	positive in 100 mL
PZ-2	negative in 2000 mL	negative in 2000 mL	negative in 1000 mL	negative in 1000 mL

*detected semi-quantitavely using PCR or nested PCR.

Organic trace substances: non target screening

At the Nizamuddin field site in central Delhi, the Yamuna River receives all kinds of pollutants present in wastewater from the Mega-City. The increasing load of organic contaminants becomes visible by comparing the GC scan of the surface water sample (NI-SW) with the one of the sample from upstream Delhi (PA-SW)(Figure 10).

Accumulation of organic trace contaminants

Accumulation of organic trace contaminants in the urban environment is reflected by an increased counta rise in peaks. Higher concentrations of many substances lead to higher abundances and broader peaks in the diagram (the integrated value for the surface under the peaks is given as area % in the appendix). The increase in the total organic load becomes most obvious in the F3 fraction: Whereas the PA-SW curve is characterised by a relatively flat baseline, interrupted by a number of peaks, the NI-SW consists of a cluster of major and minor peaks, partly overlapping each other. The baseline is considerably elevated (up to an abundance of almost 2,000,000) due to the background noise from miscellaneous substances without specifiable peaks.

Comparing the samples makes also makes clear, that the type of organic micropollutants at both field sites is very distinct: results suggest that **phthalate** plasticisers that were detected with high abundances at Palla field site and medium polar alkanes are present in much lower concentrations at Nizamuddin Bridge. Some substances found in the Yamuna can directly be attributed to sewage effluents or industrial wastewater discharge:

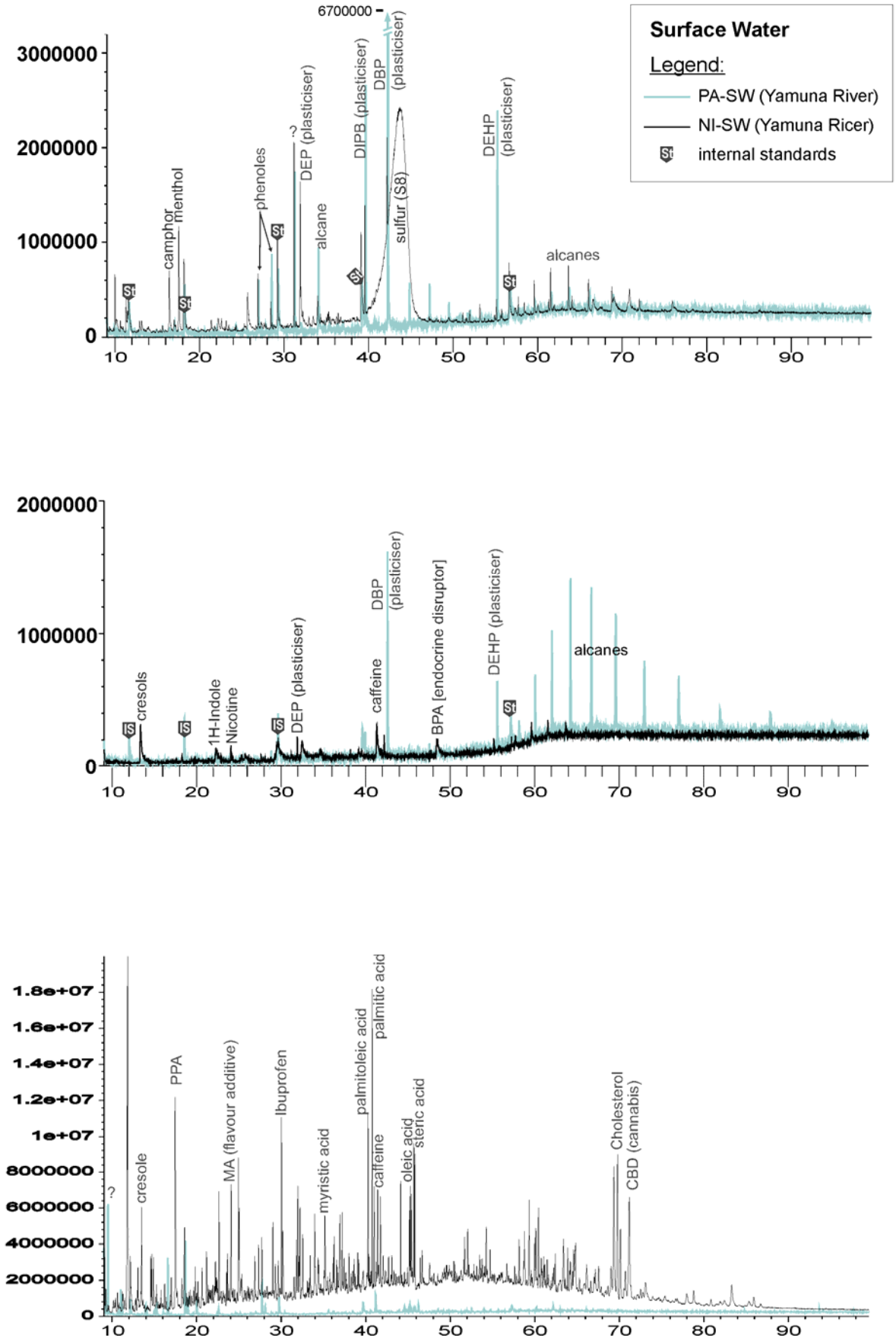

Figure 10 GC-MS analysis of surface water samples from Palla field site (PA-SW) upstream Delhi and Nizamuddin Bridge (NI-SW) indicate a drastic increase of organic contaminants in urbanised parts of the Mega City.

The huge peak in the middle appearing between around 40 to 44 minutes in the non polar fraction F1 fraction is produced by elementary **sulfur** (S8), which can only remain stable in this reduced form in waters where oxygen is virtually absent. It underlines the enormous chemical and biological oxygen demand due to the extreme loads of organic substance.

The presence of the oils of **menthol** and **camphor** in the F1 fraction are not surprising, considering that both are produced and consumed plentiful in Northern India. Srivastava et al. (2001) estimates the Indian contribution to the total world menthol production to exceed 70 % and most of it originates from menthol mint plants *(Mentha arvensis)*, cultivated on industrial scale in the Indo-Gangetic plains. The oil is exported but also used for the production of different types of products, like personal care items (i.e. in tooth paste), food and fragrance industries, pharmaceuticals and ayuverdic medicine, or even as insect repellent. Camphor is extracted from the wood of the camphor laurel tree *(Cinnamomum camphora)* and other plants and is widely used in India as an spice for traditional ayuveric medicine and for religious ceremonies (Figure 2). Whether the contents of these oils in the Yamuna River is related to processing industries in Delhi or originating from residues of final products from the consumers is not clear.

In the F2 and F3 fraction, some other products appear, which are related to sewage:

Traces of **drugs** *(substances that alter normal bodily function)* have been identified, including residues of omnipresent everydaylife stimulants (tea/coffee, cigarettes), medicines (pain killer) and illegal psychoactive drugs (hashish, marijuana):

Caffeine appears with a noticable peak in the F2 as well as in the F3 fraction. In India, it is consumed predominantly as tea (chai), which is probably the most comon baverage in India.

Nicotine is found as minor peak of the F2 fraction. Tobacco is consumed in India in various forms such as bidis (tobacco wrapped in a leaf rolls), cigarettes, or hookah (water pipe) but also sold as chewing tobacco or snuff. Nicotine was detected in the urine of consumers in India, in levels from around 0.2 up to 1 mg/L (Behera et al. 2003).

The metabolites of **ibuprofen** that were already found in the river water at Palla Field site (see Figure 6; F2 fraction) was again identified in Central Delhi (NI-SW). A larger peak indicates a higher concentration. The presence in both samples confirms, that the pharmaceutical residues were not originating from a single contamination event in the river, but seem to be permanently present.

The last noticeable peak in the F3 fraction comes from traces of **cannabidiol (CBD)**, which is a major constituent of the cannabis plant and often consumed as a mind-altering substance. CBD is not an intoxicant itself, but can influence the effects of THC, which is the main narcotic substance of the plant. Different from CBD, THC can not be detected with the methods described in this report. The controlled use of cannabis products for medical, religious, and social function has a tradition of well over a thousand years in India. In the form of charas (hashish) or ganja (marijuana), it is consumed in all parts of the countrys as "the poor mans liquor" or served as bhang (beverage of the plants leaves and flowers) or in cakes,

sweet dishes, and snacks, especially for religious events (Charles & Britto 2001). Hence, considerable traces of CBD in sewage water are not unexpected.

Other substances related with human urine and excrements in sewage are several **biomarkers**, detected in the F3 fraction of the NI-SW sample: At a retention time >50, sterols like **cholesterol** and its derivates are identified (see appendix).

Most of the major peaks in the F3 fraction of NI-SW correspond to a series of **fatty acid methyl esthers (FAME)**. Many of these substances were already present in the PA-SW, but with much lower abundance values. Considering the amount of frying fats and oils that must be used in the preparation of food for Delhi's more than 16 million inhabitants, it is not surprising to find the metabolites of these fatty acids. A huge share of these fatty acids thus may originate from food preparation like frying oils. Some of the fatty acids, however, originate from industrial application or are used for the manufacturing of different products like personal care products, candles or plastics.

Methyl Anthranilate (MA) which shows a significant peak after 24.1 minutes is an essential oil with a grape-like flavour that is commonly used as a food additive, i.e. in grape soda. **PPA** (F3, 17.46 mintues) is a substance with a strong, naturally occurring flavour i.e. in honey. It is a frequently additive in the food production and perfumes industry.

Cresoles were identified in the F2 and F3 fraction of NI-SW. They are used as industrial chemicals and for the production of different items, especially as solvents for disinfectants, deodorizers, insecticides or household cleaners.

Several other industrial chemicals were found in the Yamuna of Central Delhi (see appendix) like the toxic **DMF** (polar organic solvent) or **phthalic acid** (a common starting substance for the production in the chemical industry).

Bisphenol A (BPA) is another industrial chemical with miscellaneous applications. It is frequently used in the manufacturing of resins, flame retardants or epoxy lining of food and beverage cans (Fromme et al. 2002, Jackson & Sutton 2008). It is a persistent, synthetic chemical with occurence and behaviour similar to the phthalates and also a relatively high solubilty in water (~ 360 mg/L). It is considered as a xenoestrogen and cancerogenic substance (Fromme et al. 2002).

Furthermore, two substances were detected in the urban environment of Central Delhi, which would rather have been expected in the rural environment of Palla field site: **2,4-D** (F3, 32.55 min) and **2,4,5-T** (F3, 36.57 min.) are widely used **herbicides**, applied to defoliate or kill broad-leafed plants (including aquatic weeds). According to the WHO (2004) guideline the cancerogenic 2,4-D should not be present in drinking water in concentrations above 0.03 mg/L, but usually remains below that value because it is rapidly biodegraded in the environment. 2,4,5-T is considered toxic and cancerogenic, so the WHO (2004) guideline value is set at 0.009 mg/L. The substance has half-lives in the environment in the order of several days (WHO 2004). The mixture of both became famous in the large scale application in warfare as "agent orange" in the Vietnam War.

Attenuation during Riverbank Filtration

The organic trace components that accumulate in the Yamuna River in Central Delhi get largely attenuated during RBF. Figure 11 illustrates the GC scans from the samples of surface water (NI-SW) and the adjacent piezometer (NI-PZ). Most of the peaks from the NI-SW sample (especially from the F3 fraction where the number and abundance of identified substances is by far the highest) are removed or at least largely reduced.

Alkanes and plasticisers in the F1 and F2 fraction may again be secondary contaminants, getting released from the PVC pipe and sampling equipment or residues from contamination that occurred during the drilling and construction of the piezometer.

Other substances from the F1 and F2 fractions, including camphor, menthol, cresole as well as the endocrine disruptor and suspected cancerogen BPA are no more traceable in the bank filtrate. Likewise, none of the drugs that were found in the river (nicotine, caffeine, ibuprofen, CBD) could be detected in the sample. The same applies for the biomarkers (i.e. cholesterol), herbicides and most other substances. Only the fatty acids or their methyl esthers (FAME) respectively are not completely removed: the most abundant ones, palmitic acid and steric acid, still have traceable peaks in the groundwater sample. Considering the abundance of the peaks, it is assumed that these fats and oils are effectively degraded during infiltration and soil passage, but the travel time and distance are still insufficient for a complete removal of the fats and oils under the given environmental conditions.

One substance is detected in considerable abundance in groundwater, and was not found in the river: **MCH** (F3 fraction, 14.1 min) is a pesticide that acts as a beetle repellent. The substance, however, is not expected an environmental risk in minor concentrations, because it shows no or only very minor adverse effects on nontarget species and has an overall low toxicity. Anyhow, the exposure to humans should be minimal to non-existent and not acceptable in drinking water. The concentration is assumed to be linked to agricultural activities near the field site: The terrain is used for crop farming and the farmers apply pesticides regularly and irrigate agricultural land with water from the Yamuna River, so seepage of pesticides and other agrochemicals (fertilisers etc.) to the shallow groundwater can be assumed.

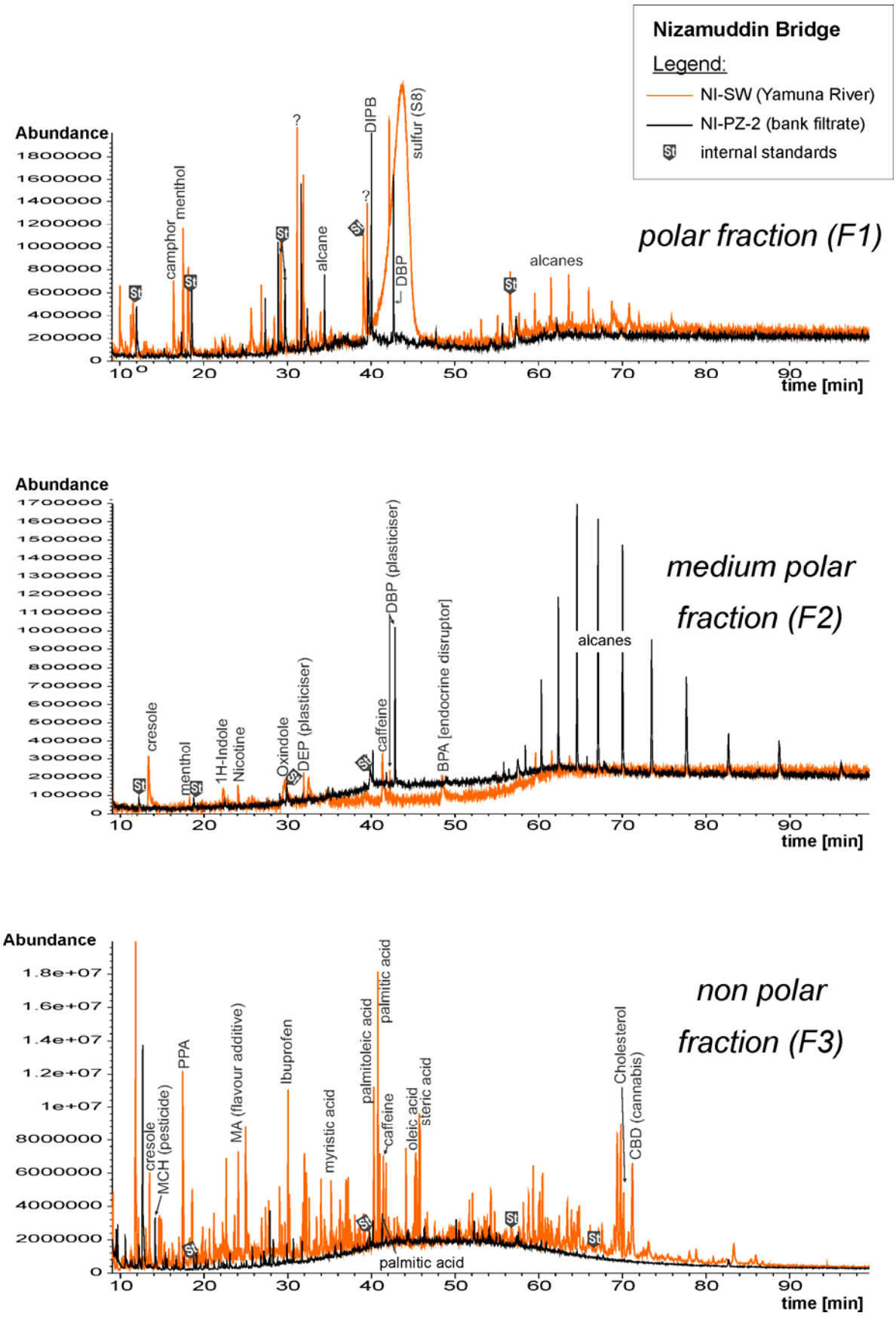

Figure 11 Trace organic compounds at the Palla field site: The three sections prepared for the GC-MS-screening are plotted separately. The sample from the piezometer with high bank filtration share is shown in the foreground, with the surface water in the back.

Organic trace substances: quantitative analysis by SGS

The concentrations of all pesticides and most other substances mentioned in Table 3 remain below the detection limit for all samples from Nizamuddin Bridge.

Only **anionic detergents** (e.g. MBAS) were detected in the Yamuna River in a concentration of 0.18 mg/L, which remains below the desirable limit of 0.2 mg/L, and the permissable maximum of 1.0 mg/L of the Indian Standards. Whether the detected value is a typical value for the Yamuna water in Central Delhi is not clear, it could as well be a maximum or minimum peak. In all the samples of the piezometers, concentrations of these detergents remain below the detection limit of 0.05 mg/L. This indicates that after only 1.5 m of subsurface passage, a possible pollution with anionic detergents is effectively attenuated. **B**enzene was also detected but not in the Yamuna. Concentrations remain below the detection limit of 0.1 µg/L in all samples except the one from NI-PZ-2c where benzene was detected with 0.65 µg/L. This trace contamination remains far below the WHO guideline value of 10 µg/L. It is probably a secondary contamination, because it is rarely found in bank filtrate at a distance of around 4 m from the shoreline, whereas it is absent in the river and the piezometer in between. A potential source of secondary contaminination, are exhaust fumes from the generator that was used for power supply for the sampling pump or exhaust fumes from huge diesel operated irrigation pumps of local farmers.

With regard to the apparent absence of the other organic compounds, it should be considered that the samples are only representing snapshots and are not necessarily characteristic values for the system.

3.3. Najafgarh Drain

Bacteriological analysis

At the Najafgarh field site the faecal coliform concentrations in the Najafgarh Drain show an increase with time (Figure 12). However, it does not reach the high contamination level found in the Yamuna River at Nizamuddin. This increase over time is attributed to an increasing discharge of untreated sewage water. The SGS analysis of *E.coli* and coliforms at the Najafgarh Drain (Table 8) supports the results obtained by the IITD. The SGS results from the observation point at the Najafgarh Drain II, which is situated approx. 2 km upstream of the Najafgarh field site, show that the contamination level at this stretch of the Najafgarh Drain is low (Figure 3). It must be taken into account that the SGS measurements give only a snapshot of the contamination level since sampling was here carried out only once. Anyhow, the faecal contamination of the Najafgarh Drain, compared to the contamination of the drain in the central part of the city, is still low. It was thus concluded that this particular sites provides good opportunities for removal of microbial contaminants by RBF systems.

The dug well shows faecal contamination in the same magnitude as the Najafgarh Drain. Pathogens present in the shallow open well may come from various sources: Villagers use the same bucket for water extraction, personal hygiene and household use. In addition, livestock and other animals are in proximity of the unprotected dug well.

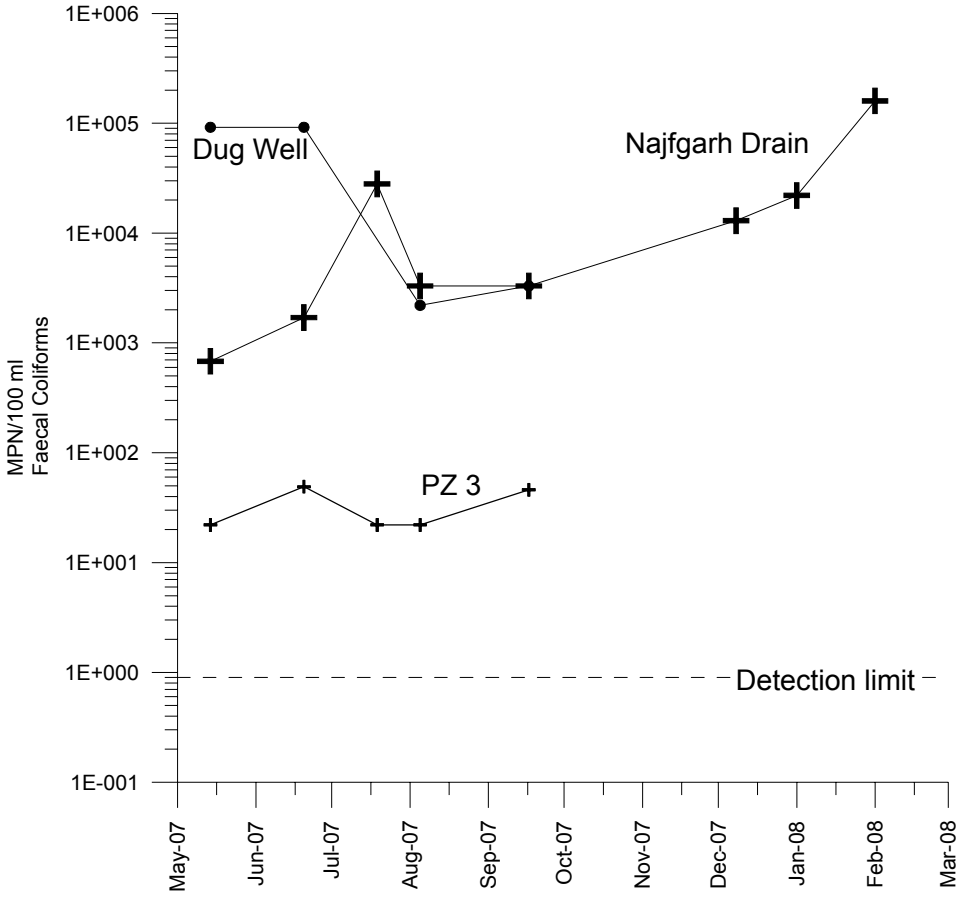

Figure 12 Faecal coliforms at the Najafgarh site analysed by the IITD.

The observation well PZ-3 also shows some degree of faecal contamination, but since this is the deepest of all observation wells (filter screen depth 35mbgl), the data obtained is not very representative for conditions in the aquifer. It was also shown by the SGS analysis of an shallow observation well (PZ-1) that the groundwater is free of indicator organisms, suggesting the absence of faecal contamination.

Table 8 Microbial parameters analysed by the SGS laboratory (Najafgarh site)

Observation point	*E.coli* (cfu/100 mL)	Coliform (cfu/100 mL)	*Enterococci* (feacal streptococci) (cfu/100 mL)	*Clostridia* (cfu/mL)
Najafgarh Drain	8.70E+04	2.50E+05	3500	<1
Najafgarh Drain II	<1	<1	<1	<1
PZ-1	<1	<1	<1	<1

Organic trace substances: quantitative analysis

A non target analysis was not performed at Najafgarh Drain. In the quantitative analysis, the two samples from the drain itself and the shallow piezometer (NA-PZ-1) nearby did not have any traceable contamination of pesticides or organic pollutants as listed in Table 3. The only exception is a concentration of 1.2 µg/L of benzene found in the groundwater sample. It is most probably again a secondary contamination from exhaust fumes of the generator used for sampling (see above) or vehicles driving past (the piezometer is standing on a roadside).

4. Conclusions

Based on the microbial results given in this report and the literature reviewed, it is concluded that Riverbank Filtration, effectively reduces (indicator) bacteria and viruses even when the surface water is heavily polluted with pathogens,. The following conclusions can be drawn for the specific field sites:

Palla well field

- No significant difference between monsoon and non-monsoonal season in faecal contamination in the Yamuna River can be found
- After ~30 days of travel time and a share of bankfiltrate between 30 – 60 % no faecal contamination is found in the tubewell
- The faecal pollution level of the Yamuna River is about bathing water quality (10^{+3} mpn/100 mL)

Nizamuddin Bridge

- No significant difference between monsoon and non-monsoon season in faecal contamination in the Yamuna River can be found
- Faecal pollution level of the Yamuna River is very high (~10^{+7} mpn/100 mL)
- Under the given field conditions 3 $\log_{10}$ removal of somatic bacteriophages after 1 m of travel distance was achieved. Given the fact that RBF wells should be located more distant from the shoreline, the results are very reassuring; however, further studies under different hydraulic conditions are required
- High numbers of up to 10^4 genome copies/100 mL of human pathogenic viruses (adeno- and norovirus) were found in the Yamuna River
- Hepatitis A and E virus have been detected semi-quantitatively in the Yamuna River

Najafgarh Drain

- A significant increase of faecal contamination over time can be observed at the Najafgarh Drain
- Dugwells of the area constitute a potential input path for faecal contaminants into groundwater

Based on the non target screening and quantitative analysis of organic trace compounds, a good overview of contamination load, potential sources of contaminants and the attenuation potential of RBF systems is provided. The results, however, have to be interpreted with caution because they are based only on singular measurements from each field site and may not reflect average values. Anyhow, the following conclusions can be drawn for the specific field sites:

Palla well field

- None of the 61 pesticides or organic pollutants selected for quantitative analysis was traceable within the detection limits.
- Number and abundance of organic trace compounds in the surface water is still comparably low.
- Some substances (i.e. fatty acids) are more abundant in the bank filtrate than in the river. Their average concentrations in the Yamuna may be considerably higher than those in the surface water sample.
- Phthalate plasticisers and alkanes in the groundwater samples may result from secondary contamination source (e.g. residues from the drilling sludge, leaching from PVC tubes).

Nizamuddin Bridge

- Anionic detergents were found in Yamuna water, but not detected in the bank filtrate, so they are probably attenuated after only a few meters of subsurface passage.
- Benzene was found in shallow groundwater but not in the surface water. It may originate from a secondary contamination source, e.g. exhaust fumes.
- Except from the above mentioned, none of the substances selected for quantitative analysis (including 61 pesticides) was traceable within the detection limit.
- The non target screening confirmed the presence of all kinds of organic trace contaminants from industrial waste sources to biomarkers. Especially in the polar fraction, a multitude of significant peaks indicate a huge variety of organic trace substances accumulating in the Yamuna River.
- A comparison of surface water sample and bank filtrate shows that the major amount of organics is either strongly reduced or completely removed during infiltration and subsurface passage. Examples for pollutants that were identified in the river sample but not traceable in the bankfiltrate include the following substances and derivatives:
    - Drugs: nicotine, caffeine, ibuprofen, cannabidiol [absent]
    - Biomarkers: i.e. cholesterol [absent]
    - Herbicides: 2,4-D and 2,4,5-T [absent]
    - Fatty acids: i.e. palmitic acid, steric acid [almost completely reduced]
- Phthalate plasticisers and alkanes in the groundwater samples may be resulting from secondary contamination (residues from the drilling sludge, leaching from PVC tubes).

- None of the 61 pesticides or organic pollutants selected for quantitative analysis was traceable within the detection limits, except from benzene.
- Benzene was found in shallow groundwater but not in the surface water. It may originate from a secondary contaminantion source (e.g. exhaust fumes).

5. Outlook

In order to evaluate the results obtained from bacteriophages, it is recommended to carry out a monitoring program for coliforms under sterile conditions. In order to achieve more reliable results, the sampling for bacteriophages should be repeated under varying conditions. The elimination capacity should be tested with induced flow regime, since the flow velocity is considered to be crucial for the removal of viruses. These results can contribute to the understanding of the processes for virus removal during sediment passage.

Further studies are needed to evaluate the well head protection at the Palla field site during flood events.

Since the sampling for organic trace compounds is only a snapshot, it is recommended to carry out at least three campaigns in one year to cover the broad climatic variations during post- pre- and monsoon periods and the associated contaminant load during the respective season. Within these investigations, surface water (especially at Palla Field site where residence times are minimal) would have to be sampled more frequently to identify short term fluctuations. To avoid secondary contamination and estimate its influence on the above presented results, it would be useful to built at least one piezometer at each field site with metal tubing than instead of PVC.

For some organic substances (i.e. drugs, herbicides) that could be identified in the non target screening, a quantitative analysis would be relevant. The piezometers at Nizamuddin field at only a few meters distance from the River were built after the GC-MS screening. The transect which is now available would be adequate to study degradation rates of organic trace contaminants.

6. Acknowledgements

We would like to thank Prof. Dr. J. López-Pila and Dipl. Biol. A. Grunert (Federal Environmental Agency of Germany) for their indispensable commitment to the microbiological investigations. Without their experience, know-how and manpower provided during the organisation and implementation of microbiological sampling, analysis and data interpretation, this report would not have been possible. We are also grateful to Prof. Dr. R Girones for the analysis of human pathogenic viruses at the Department of Microbiology of the University of Barcelona.

Furthermore, we would like to express our gratitude to Dr. A. K. Mittal from the Environmental Engineering Laboratory of the Civil Engineering Department at the Indian Institute of Technology in Delhi (IITD). He supported parts of these

sampling campaigns by providing assistants and logistical help and was responsible for bacteriological analysis in India. In addition, he offered us space in his laboratory for associated work and implementation of the analysis of bacteriophages. We want to thank IITD students, particularly M-Tec. Pravin Kumar and B-Tec. M. Ronghang, who were a great help for all work in India. M. Ronghang collected most of the data on indicator bacteria and pathogens, showed an extraordinary commitment and contributed considerably to the succes of the project.

Last but not least we acknowledge the scientists of the Environmental Organic Geochemistry at Department of Earth Sciences from FUB for conducting the GC-MS analysis as a courtesy. The processing of the samples was performed by cand. geol. S. Kuhn under the supervision of M. Ricking (head of the laboratory). The GC-MS analysis of organic trace substances, was done by M. Ricking who also provided advice on data interpretation has reviewed this report. We would like to thank them most sincerely for their efforts.

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APPENDIX

PA-SW (March 2007) F1 (non polar fraction)

Peak no.	ret. time	area %	substance	CAS number	Qua-lity	*comment*
1	11,7	3,03	1,2-DICHLOROBENZENE-D4	000000-00-0	91	*INTERNAL STANDARD*
2	17,06	0,66	Cyclododecane (CAS)	000294-62-2	93	*cycloalkane*
3	18,24	3,95	Naphthalene-d8; Naphthalene-d8-	001146-65-2	91	*INTERNAL STANDARD*
4	24,3	0,27	1-Tetradecene (CAS); n-Tetradec-1-e	001120-36-1	96	*alkane*
5	27,02	1,96	2,6-di(t-butyl)-4-hydroxy-4-methyl-2,5-cyclohexadien-1-o		95	*(BHTOH) alkane*
6	28,57	3,03	Phenol, 2,4-bis(1,1-dimethylethyl)- (	000096-76-4	94	*phenole*
7	29,36	5,16	Z-jasmone	000000-00-0	91	
9	32,04	0,44	1,2-Benzenedicarboxylic acid, diethyl	000084-66-2	96	*phthalate: DEP (plast.)*
10	34,1	4,73	Heptadecane (CAS); n-Heptadecane	000629-78-7	95	*alkane*
11	36,99	0,46	Octadecane (CAS); n-Octadecane; O	000593-45-3	97	*alkane*
12	39,27	5,42	Anthracene-D10	000000-00-0	93	*INTERNAL STANDARD*
13	39,68	10,5	1,2-Benzenedicarboxylic acid, bis(2-methyl	000084-69-5	95	*phthalate: DIBP (plast.)*
14	42,32	8,29	1,2-Benzenedicarboxylic acid, dibutyl	000084-74-2	91	*phthalate: DBP (plast.)*
15	44,87	2,22	HENEICOSANE	000629-94-7	97	*alkane*
16	47,26	2,4	Docosane (CAS); n-Docosane; C22H4	000629-97-0	98	*alkane*
17	49,55	1,16	Tricosane (CAS); n-Tricosane	000638-67-5	97	*alkane*
18	51,76	0,56	N-EICOSANE	000112-95-8	87	*alkane*
20	55,28	8,46	1,2-Benzenedicarboxylic acid, bis(2-e	000117-81-7	90	*phthalate: DEHP (plast.)*
21	56,81	4,18	PERDEUTERO-CHRYSENE; Chrysene-d	001719-03-5	90	*INTERNAL STANDARD*
25	63,81	1,24	Eicosane (CAS); n-Eicosane	000112-95-8	91	*alkane*

PA-SW (March 2007) F2 (medium polar fraction)

Peak no.	ret. time	area %	substance	CAS number	Qua-lity	*comment*
1	12,04	1,73	1,2-DICHLOROBENZENE-D4	000000-00-0	95	*INTERNAL STANDARD*
2	18,55	3,25	Naphthalene-d8; Naphthalene-d8-	001146-65-2	91	*INTERNAL STANDARD*
4	28,84	0,53	Phenol, bis(1,1-dimethylethyl)- (CAS)	026746-38-3	87	*phenole*
	29,65	4,65	Acenaphthene-d10	015067-26-2	93	*INTERNAL STANDARD*
6	32,32	0,28	1,2-Benzenedicarboxylic acid, diethyl	000084-66-2	93	*phthalate: DEP (plast.)*
9	39,64	4,68	Anthracene-D10	000000-00-0	93	*INTERNAL STANDARD*
11	42,61	1,17	1,2-Benzenedicarboxylic acid, dibutyl	000084-74-2	91	*phthalate: DBP (plast.)*
12	45,16	0,46	Heneicosane (CAS); n-Heneicosane	000629-94-7	93	*alkane*
13	47,56	0,8	Docosane (CAS); n-Docosane; C22H4	000629-97-0	92	*alkane*
15	55,6	3,15	1,2-Benzenedicarboxylic acid, bis(2-e	000117-81-7	87	*phthalate: DEHP (plast.)*
17	57,2	4,23	PERDEUTERO-CHRYSENE; Chrysene-d	001719-03-5	91	*INTERNAL STANDARD*
21	60,07	3,55	HENEICOSANE	000629-94-7	95	*alkane*
23	62,07	5,67	N-DOCOSANE	000629-97-0	97	*alkane*
24	64,26	9,05	Triacontane (CAS); n-Triacontane	000638-68-6	96	*alkane*
26	66,73	9,5	HENEICOSANE	000629-94-7	97	*alkane*
29	69,62	9,99	Tricosane (CAS); n-Tricosane	000638-67-5	95	*alkane*
30	73	6,03	Eicosane (CAS); n-Eicosane	000112-95-8	93	*alkane*
32	77,09	5,66	Eicosane (CAS); n-Eicosane	000112-95-8	95	*alkane*

PA-SW (March 2007) F3 (polar fraction)

Peak no.	ret. time	area %	substance	CAS number	Qua-lity	*comment*
12	11,71	0,2	Heptanoic acid, methyl ester (CAS)	000106-73-0	80	
16	12,58	0,11	Ethane, 1,2-bis(methylthio)- (CAS)	006628-18-8	78	
20	13,98	1,15	Cyclopentasiloxane, decamethyl- (CAS)	000541-02-6	90	
24	15	0,73	Benzoic acid, methyl ester (CAS); M	000093-58-3	80	
25	15,29	1,19	Octanoic acid, methyl ester (CAS)	000111-11-5	95	*FAME (caprylic acid)*
29	16,99	0,24	Trimethylsilyl derivative of 1-(3,4-d	000000-00-0	78	
36	18,97	2,34	Nonanoic acid, methyl ester (CAS)	001731-84-6	94	
47	22,61	0,85	Decanoic acid, methyl ester (CAS)	000110-42-9	96	*FAME (capric acid)*
58	26,36	0,16	methyl 4,4,7-trimethyl-4,7-dihydroind	121013-28-3	83	
61	27,01	0,39	3',4'-(Methylenedioxy)acetophenone	003162-29-6	87	
64	27,74	3,85	PARA-T-BUTYL-BENZOIC ACID, METH	026537-19-9	95	*PTBBA*
65	28,12	1,35	1,2-Benzenedicarboxylic acid, dimethy	000131-11-3	91	*DMF (i.e. insecticide)*
70	29,42	0,57	METHYL DODECANOATE; METHYL LAU	000000-00-0	93	
71	29,76	2,97	Acenaphthene-d10	015067-26-2	83	*INTERNAL STANDARD*
74	30,43	0,16	Benzeneacetic acid, .alpha.-methyl-4-	061566-34-5	93	*Ibuprofen*
80	32,62	0,21	10b,10c-dimethyl-1,2,10b,10c-tetrahyd	113305-20-7	86	
87	35,6	0,83	Tetradecanoic acid, methyl ester (CAS	000124-10-7	98	***FAME (myristic acid)***
95	37,63	0,27	Tetradecanoic acid, 12-methyl-, methy	005129-66-8	83	
97	38,46	0,46	Pentadecanoic acid, methyl ester (CAS	007132-64-1	94	
100	39,69	3,23	DECADEUTEROPHENANTHRENE	001517-22-2	95	*INTERNAL STANDARD*
103	40,59	0,38	9-Hexadecenoic acid, methyl ester, (Z	001120-25-8	78	***FAME(palmitoleic acid)***
106	41,16	3,44	Hexadecanoic acid, methyl ester (CAS)	000112-39-0	98	***FAME (palmitic acid)***
108	41,64	0,32	3,4-dihydro-7,12-dihydroxy-7,12-dimet	000000-00-0	83	
119	43,77	0,17	Tetradecanoic acid, 5,9,13-trimethyl-	056196-55-5	89	
124	44,84	0,45	3-ETHOXYPHENYLACETONE HYDRO	000000-00-0	90	
128	45,7	0,87	9-Octadecenoic acid (Z)-, methyl este	000112-62-9	94	***FAME (oleic acid)***
131	46,24	1,24	Octadecanoic acid, methyl ester (CAS)	000112-61-8	98	***FAME (stearic acid)***
151	50,9	0,12	Eicosanoic acid, methyl ester (CAS)	001120-28-1	93	
159	52,52	0,43	1-Phenanthrenecarboxylic acid, 1,2,3,	001740-19-8	89	
164	53,6	0,06	3-METHYL-5-DIPHENYLDIHYDRAFUR	000000-00-0	93	
174	56,31	0,12	Hexacosane (CAS); n-Hexacosane	000630-01-3	90	
175	57,28	2,19	PERDEUTERO-CHRYSENE	001719-03-5	91	*INTERNAL STANDARD*
180	58,28	0,31	Docosane (CAS); n-Docosane; C22H4	000629-97-0	91	*alkane*
183	59,38	0,06	3-METHYL-5-DIPHENYLDIHYDRAFURA	000000-00-0	93	
187	60,19	0,46	Octacosane (CAS); n-Octacosane	000630-02-4	98	*alkane*
192	62,19	0,61	Docosane (CAS); n-Docosane; C22H4	000629-97-0	93	*alkane*
198	64,4	0,49	Docosane (CAS); n-Docosane; C22H4	000629-97-0	93	*alkane*
202	66,89	0,39	Octadecane (CAS); n-Octadecane; O	000593-45-3	78	*alkane*
204	67,52	0,57	Perylene-d12	001520-96-3	86	*INTERNAL STANDARD*
209	69,8	0,48	TETRACOSANE	000000-00-0	89	*alkane*
217	79,08	0,07	Spiro[cyclopenta[c]pyran-7(1H),2'-[1,	103384-82-3	86	

PA-PZ (March 2007) F1 (non polar fraction)

Peak no.	ret. time	area %	substance	CAS number	Qua-lity	*comment*
1	11,58	1,53	1,2-DICHLOROBENZENE-D4	32086	94	*INTERNAL STANDARD*
2	11,74	0,53	1,4-DICHLOROBENZENE-D4	32087	83	*INTERNAL STANDARD*
3	16,95	0,75	2-Dodecene, (Z)- (CAS)	53763	96	
4	18,12	2,69	Naphthalene-d8; Naphthalene-d8-	19926	91	*INTERNAL STANDARD*
5	24,19	0,33	3-Tetradecene, (E)- (CAS)	81333	95	
6	26,92	1,59	2,6-di(t-butyl)-4-hydroxy-4-methyl-2,	12217	97	
7	27,77	0,35	2,5-Cyclohexadiene-1,4-dione, 2,6-bis	10543	93	
8	28,46	2,98	Phenol, 2,4-bis(1,1-dimethylethyl)- (	91574	93	
10	29,23	2,92	Acenaphthene-d10	40987	87	*INTERNAL STANDARD*
11	29,41	0,31	Acenaphthene-d10	40987	93	*INTERNAL STANDARD*
13	30,82	0,34	3-Hexadecene, (Z)- (CAS); cis-3-Hex 1	10849	98	
15	31,95	0,95	Isopropyl dodecanoate; ISOPROPYL ES 1	28006	95	
16	34	4,21	HEPTADECANE 1	26483	97	*alkane*
19	39,13	4,25	Anthracene-D10	63087	94	*INTERNAL STANDARD*
20	39,57	5,05	1,2-Benzenedicarboxylic acid, bis(2-m 1	60093	83	
21	40,89	0,29	1,2-Benzenedicarboxylic acid, butyl 2 2	2251	80	
22	42,2	6,57	1,2-Benzenedicarboxylic acid, dibutyl 1	60079	90	
23	44,76	1,46	Heneicosane (CAS); n-Heneicosane 1	75419	97	*alkane*
24	47,14	1,67	Docosane (CAS); n-Docosane; C22H4 1	86056	99	
25	49,44	0,84	Tricosane (CAS); n-Tricosane 1	95910	98	
26	51,64	0,37	Tetracosane (CAS); n-Tetracosane 2	4919	96	
31	55,16	9,81	1,2-Benzenedicarboxylic acid, bis(2-e 2	30979	90	
32	55,77	0,34	Eicosane (CAS); n-Eicosane 1	63881	95	
33	56,64	3,78	PERDEUTERO-CHRYSENE; Chrysene-d	14792	91	*INTERNAL STANDARD*
35	57,73	0,62	Eicosane (CAS); n-Eicosane 1	63881	95	
36	59,61	1,24	Octadecane (CAS); n-Octadecane; O 1	39444	95	*alkane*
38	61,56	1,83	Nonacosane (CAS); n-Nonacosane; C 2	37991	97	*alkane*
39	63,67	2,78	Eicosane (CAS); n-Eicosane 1	63878	97	*alkane*
40	66,04	2,69	Eicosane (CAS); n-Eicosane 1	63881	96	*alkane*
42	68,81	2,78	Docosane (CAS); n-Docosane; C22H4 1	86056	97	*alkane*
43	72,06	1,85	EICOSANE 1	63904	94	*alkane*
44	75,95	1,51	Eicosane (CAS); n-Eicosane 1	63881	95	*alkane*

PA-PZ (March 2007) F2 (medium polar fraction)

Peak no.	ret. time	area %	substance	CAS number	Quality	*comment*
1	11,63	2,67	1,2-DICHLOROBENZENE-D4	000000-00-0	91	*INTERNAL STANDARD*
2	17,5	0,58	Ethanol, 1-(2-butoxyethoxy)- (CAS)	054446-78-5	80	
3	18,14	2,96	Naphthalene-d8; Naphthalene-d8-	001146-65-2	91	*INTERNAL STANDARD*
4	19,31	4,69	Ethanol, 2-phenoxy- (CAS); 2-Phenox	000122-99-6	91	*used as bactericide*
7	23,72	1,8	Ethanol, 2-(2-butoxyethoxy)-, acetate	000124-17-4	90	
8	28,46	1,11	Phenol, 2,4-bis(1,1-dimethylethyl)- (	000096-76-4	94	*phenole*
9	29,24	3,9	Acenaphthene-d10	015067-26-2	83	*INTERNAL STANDARD*
11	31,93	0,73	1,2-Benzenedicarboxylic acid, diethyl 1	000084-66-2	95	*phthalate: DEP (plast.)*
16	39,13	4,19	Anthracene-D10	000000-00-0	93	*INTERNAL STANDARD*
17	39,57	3,56	BUTYL-2-ETHYLHEXYL PHTHALATE	000000-00-0	83	
18	42,21	1,96	1,2-Benzenedicarboxylic acid, bis(2-m 1	000084-69-5	90	*phthalate: DIBP (plast.)*
19	44,77	0,79	HENEICOSANE 1	000000-00-0	95	*alkane*
20	47,16	0,65	N-DOCOSANE 1	000629-97-0	95	*alkane*
21	49,44	0,3	Tricosane (CAS); n-Tricosane 1	000638-67-5	98	*alkane*
24	55,17	8,13	1,2-Benzenedicarboxylic acid, bis(2-e 2	000117-81-7	90	*phthalate: DEHP (plast.)*
25	55,77	0,39	Eicosane (CAS); n-Eicosane 1	000112-95-8	95	*alkane*
26	56,53	2,13	1,1-DICYANO-2-METHYL-4-(P-CYANOP	000000-00-0	78	
27	56,64	2,73	PERDEUTERO-CHRYSENE; Chrysene-d	001719-03-5	93	*INTERNAL STANDARD*
28	56,8	4,77	3-[(E)-2-Chloro-1-methyl-1-butenyl]-3	000000-00-0	90	
31	57,73	0,83	Eicosane (CAS); n-Eicosane 1	000112-95-8	95	
34	59,63	1,3	Eicosane (CAS); n-Eicosane 1	000112-95-8	93	
35	61,56	1,91	Eicosane (CAS); n-Eicosane 1	000112-95-8	97	
36	63,68	1,96	Eicosane (CAS); n-Eicosane 1	000112-95-8	97	
37	66,06	1,75	Eicosane (CAS); n-Eicosane 1	000112-95-8	95	
38	66,55	0,65	Perylene-d12 1	001520-96-3	95	*INTERNAL STANDARD*
39	68,81	1,44	Eicosane (CAS); n-Eicosane 1	000112-95-8	86	

PA-PZ (March 2007) F3 (polar fraction)

Peak no.	ret. time	area %	substance	CAS number	Quality	*comment*
9	11,41	0,15	1-Hexanol, 2-ethyl- (CAS); 2-Ethylh	000104-76-7	83	
13	12,1	15,4	Ethane, 1,2-bis(methylthio)- (CAS)	006628-18-8	83	
18	13,02	0,51	trans-1,2,3-Trimethylindoline; 1H-I	055049-68-8	83	
20	13,54	10,5	Cyclopentasiloxane, decamethyl- (CAS)	000541-02-6	76	
23	14,49	0,47	Benzoic acid, methyl ester (CAS); M	000093-58-3	83	
24	14,8	1,32	Octanoic acid, methyl ester (CAS)	000111-11-5	95	*FAME (caprylic acid)*
27	15,59	0,16	Pentanedioic acid, dimethyl ester (CA	001119-40-0	78	
33	16,81	1,27	Cyclotetrasiloxane, octamethyl- (CAS)	000556-67-2	86	
40	18,15	1,15	Naphthalene-d8; Naphthalene-d8-	001146-65-2	90	*INTERNAL STANDARD*
41	18,51	1,77	Nonanoic acid, methyl ester (CAS)	001731-84-6	97	
45	19,33	0,34	Ethanol, 2-phenoxy- (CAS); 2-Phenox	000122-99-6	91	*used as bactericide*
46	19,59	1,19	Cyclohexasiloxane, dodecamethyl- (CAS	000540-97-6	81	
57	22,13	0,88	Decanoic acid, methyl ester (CAS)	000110-42-9	96	*FAME (capric acid)*
73	25,62	0,11	methyl decanoate; METHYL CAPRINATE	000110-42-9	87	
83	27,25	3,3	PARA-T-BUTYL-BENZOIC ACID, METHY	026537-19-9	95	*PTBBA*
85	27,63	1,54	1,2-Benzenedicarboxylic acid, dimethy	000131-11-3	91	*DMF (i.e. insecticide)*
91	28,95	0,54	Dodecanoic acid, methyl ester (CAS)	000111-82-0	98	***FAME (lauric acid)***
92	29,23	1,03	Acenaphthene-d10	015067-26-2	87	*INTERNAL STANDARD*
95	29,62	0,45	1,3-Benzenedicarboxylic acid, dimethy	001459-93-4	93	
98	30,11	0,47	Nonanedioic acid, dimethyl ester (CAS	001732-10-1	78	*(Azelaic acid)*
117	35,1	0,5	METHYL TETRADECANOATE; METHY	000000-00-0	98	
127	36,74	0,07	2-Amino-5-benzyl-3-bromopyridine N-Ox	130277-04-2	83	
132	37,95	0,28	Pentadecanoic acid, methyl ester (CAS	007132-64-1	95	
136	39,11	1,2	Anthracene-D10	000000-00-0	95	*INTERNAL STANDARD*
137	39,53	0,14	1,2,4-Benzenetricarboxylic acid, trim	002459-10-1	78	
138	39,63	0,11	Nonadecane (CAS); n-Nonadecane	000629-92-5	90	*alkane*
139	39,95	0,04	3-Cyano-6,7-dihydro-2-methyl-4-(methy	130445-85-1	83	
140	40,1	0,13	11-Hexadecenoic acid, methyl ester (C	055000-42-5	87	
143	40,67	2,01	Hexadecanoic acid, methyl ester (CAS)	000112-39-0	98	***FAME (palmitic acid)***
151	42,2	0,41	1,2-Benzenedicarboxylic acid, dibutyl	000084-74-2	93	*phthalate: DBP (plast.)*
155	43,26	0,1	Heptadecanoic acid, methyl ester (CAS	001731-92-6	96	
162	45,18	0,29	8-Octadecenoic acid, methyl ester, (E	026528-50-7	99	
165	45,73	1	Octadecanoic acid, methyl ester (CAS)	000112-61-8	98	***FAME (stearic acid)***
167	46,74	0,11	5.beta.-Podocarpa-8,11,13-trien-16-oi	003745-36-6	89	
168	47,14	0,09	Docosane (CAS); n-Docosane; C22H4	000629-97-0	93	*alkane*
173	48,72	0,03	11H-Dibenzo[b,e][1,4]diazepin-11-one,	013450-70-9	92	
176	49,44	0,09	Tetradecane (CAS); n-Tetradecane	000629-59-4	86	*alkane*
180	50,36	0,11	Eicosanoic acid, methyl ester (CAS)	001120-28-1	95	
185	51,98	1,76	1-Phenanthrenecarboxylic acid, 1,2,3,	001740-19-8	99	
191	53,74	0,05	Nonadecane (CAS); n-Nonadecane	000629-92-5	90	*alkane*
192	53,96	0,08	6-Cyanomethyl-8-methylbenzo[b]naphtho	089817-21-0	80	
195	54,64	0,14	Docosanoic acid, methyl ester (CAS)	000929-77-1	90	
197	55,16	0,88	1,2-Benzenedicarboxylic acid, bis(2-e	000117-81-7	91	*phthalate: DEHP (plast.)*
199	55,77	0,08	HEXACOSANE	000000-00-0	87	*alkane*
200	56,51	0,64	1,1-DICYANO-2-METHYL-4-(P-CYANOP	000000-00-0	83	
201	56,62	0,69	PERDEUTERO-CHRYSENE	001719-03-5	94	*INTERNAL STANDARD*
202	56,79	1,25	1,1-DICYANO-2-METHYL-4-(P-CYANOP	000000-00-0	83	
203	56,98	0,36	1H-Indole, 2-methyl-3-phenyl- (CAS)	004757-69-1	86	
205	57,5	0,08	DEHYDROABIETIC ACID, METHYL EST	000000-00-0	89	
206	57,73	0,18	Heptacosane (CAS); n-Heptacosane	000593-49-7	95	*alkane*
210	58,59	0,19	Tetracosanoic acid, methyl ester (CAS	002442-49-1	94	
214	59,62	0,22	Nonacosane (CAS); n-Nonacosane; C	000630-03-5	91	*alkane*
219	61,54	0,31	Nonacosane (CAS); n-Nonacosane; C	000630-03-5	97	*alkane*
224	63,66	0,26	Heneicosane (CAS); n-Heneicosane	000629-94-7	95	*alkane*
227	66,04	0,15	Hentriacontane (CAS); Untriacontane	000630-04-6	92	
228	66,54	0,39	Perylene-d12	001520-96-3	93	*INTERNAL STANDARD*
231	68,81	0,12	Docosane (CAS); n-Docosane; C22H4	000629-97-0	95	*alkane*

NI-SW (March 2007) F1 (non polar fraction)

Peak no.	ret. time	area %	substance	CAS number	Quality	*comment*
1	10,01	1,65	Trisulfide, dimethyl (CAS); 2,3,4-T	003658-80-8	90	
2	11,58	4,85	1,2-DICHLOROBENZENE-D4	000000-00-0	91	*INTERNAL STANDARD*
3	16,42	4,06	Camphor; Bicyclo[2.2.1]heptan-2-one, 1,7,	000464-49-3	98	***camphor***
4	17,55	5,57	Cyclohexanol, 5-methyl-2-(1-methylethyl)-	001490-04-6	91	***menthol (mint oils)***
5	18,15	7,15	Naphthalene-d8; Naphthalene-d8-	001146-65-2	91	*INTERNAL STANDARD*
6	26,93	2	2,6-di(t-butyl)-4-hydroxy-4-methyl-2,	000000-00-0	99	*alkane (BHTOH)*
7	29,24	4,44	Acenaphthene-d10	015067-26-2	78	*INTERNAL STANDARD*
9	31,92	3,47	1,2-Benzenedicarboxylic acid, diethyl	000084-66-2	95	*phthalate: DEP (plast.)*
	33,99	1,41	Heptadecane (CAS); n-Heptadecane	000629-78-7	95	*alkane*
10	39,12	7,46	Anthracene-d10-	001719-06-8	97	*INTERNAL STANDARD*
11	39,58	3,86	1,2-Benzenedicarboxylic acid, dibutyl	000084-74-2	72	*phthalate: DBP (plast.)*
13	43,38	1,52	SULFUR; Sulfur, precipitated	007704-34-9	76	***sulfur***
14	43,51	5,09	Sulfur, mol. (S8) (CAS); OCTA-SULFU	010544-50-0	86	***sulfur***
15	43,75	9,83	Sulfur, mol. (S8) (CAS); OCTA-SULFU	010544-50-0	90	***sulfur***
16	43,93	4,92	SULFUR; Sulfur, precipitated	007704-34-9	83	***sulfur***
17	56,65	4,76	PERDEUTERO-CHRYSENE; Chrysene-d	001719-03-5	98	*INTERNAL STANDARD*
18	61,55	1,77	Octadecane (CAS); n-Octadecane; O	000593-45-3	96	*alkane*
19	63,67	2,02	Eicosane (CAS); n-Eicosane	000112-95-8	98	*alkane*

NI-SW (March 2007) F2 (medium polar fraction)

Peak no.	ret. time	area %	substance	CAS number	Quality	*comment*
7	13,37	2,42	Phenol, 4-methyl- (CAS); p-Cresol	000106-44-5	91	*dissolvent, desinfectant*
9	13,88	0,12	Phenol, 4-methyl- (CAS); p-Cresol	000106-44-5	83	*dissolvent, desinfectant*
11	14,04	0,07	Phenol, 3-methyl- (CAS); m-Cresol	000108-39-4	76	
19	17,64	0,11	Menthol; Cyclohexanol, 5-methyl-2-(	000089-78-1	83	***menthol (mint oils)***
22	18,97	0,03	2,7-dioxa-4,9-divinylspiro[4.4]nonane	123538-83-0	90	
33	21,56	0,12	syn-tricyclo[4.2.1.1(2,5)]dec-3-en-9-	119478-28-3	96	
35	22,3	1,05	1H-Indole (CAS); Indole; Ketole	000120-72-9	87	***biomarker (human faeces)***
39	24,06	0,43	Pyridine, 3-(1-methyl-2-pyrrolidinyl)	000054-11-5	91	***nicotine***
43	25,33	0,33	Phenol, 4-chloro-3,5-dimethyl- (CAS)	000088-04-0	80	
56	29,52	0,66	2H-Indol-2-one, 1,3-dihydro- (CAS)	000059-48-3	90	
59	29,98	0,14	1H-Indole, 2,3-dihydro-4-methyl- (CAS	062108-16-1	81	
61	30,43	0,14	Benzene, 1-isocyanato-2-methyl-; Is	000614-68-6	83	
69	31,93	0,5	1,2-Benzenedicarboxylic acid, diethyl	000084-66-2	96	*phthalate: DEP (plast.)*
72	32,46	0,7	2,6-Dimethylphenyl isocyanate; Benz	028556-81-2	90	
73	32,54	0,42	2,6-Dimethylphenyl isocyanate; Benz	028556-81-2	86	
78	33,67	0,16	3-Ethyl-2,1-benzisoxazole	000000-00-0	90	
83	34,31	0,07	1a,9b-dihydro-4-methyl-1H-phenanthro[	111005-47-1	83	
106	39,18	0,35	DECADEUTEROPHENANTHRENE; Phe	001517-22-2	91	*INTERNAL STANDARD*
116	41,32	1,99	Caffeine; 1H-Purine-2,6-dione, 3,7-	000058-08-2	91	***caffeine (coffee, tea)***
120	42,2	0,57	1,2-Benzenedicarboxylic acid, dibutyl	000084-74-2	87	*phthalate: DBP (plast.)*
148	48,49	1,22	Phenol, 4,4'-(1-methylethylidene)bis-	014227-18-0	90	***Bisphenol A (BPA)***
259	62,11	0,19	Cyclotrisiloxane, hexamethyl- (CAS)	000541-05-9	80	*INTERNAL STANDARD*
322	72,2	0,45	Cyclotrisiloxane, hexamethyl- (CAS)	000541-05-9	80	*INTERNAL STANDARD*

NI-SW (March 2007) F3 (polar fraction) (1/2)

Peak no.	ret. time	area %	substance	CAS number	Quality	*comment*
11	11,32	0,09	Heptanoic acid, methyl ester (CAS)	000106-73-0	91	
14	11,83	1,49	Benzene, 1-methoxy-4-methyl- (CAS)	000104-93-8	95	
15	12,17	0,39	Ethane, 1,2-bis(methylthio)- (CAS)	006628-18-8	80	
18	12,78	0,06	Methyl 3,4-Dimethylhexanoate	000000-00-0	86	
23	13,4	0,08	Phenol, 4-methyl- (CAS); p-Cresol	000106-44-5	93	*Cresol*
31	14,9	0,27	Octanoic acid, methyl ester (CAS)	000111-11-5	95	*FAME (caprylic acid)*
33	15,26	0,15	Benzene, 1-chloro-4-methoxy- (CAS)	000623-12-1	91	
34	15,46	0,06	3,5-Dimethylanisole; 2,5-Dimethylan	000874-63-5	81	
35	15,59	0,04	Benzene, 1-chloro-4-methoxy- (CAS)	000623-12-1	92	
36	15,68	0,04	Pentanedioic acid, dimethyl ester (CA	001119-40-0	86	
43	17,01	0,23	Methyl 4-methyloctanoate; Octanoic	015870-07-2	80	
44	17,46	1,66	Benzeneacetic acid, methyl ester (CA	000101-41-7	90	***PPA (nat. + art. flavour)***
48	18,59	0,69	Nonanoic acid, methyl ester (CAS)	001731-84-6	93	
53	19,43	0,13	Ethanol, 2-phenoxy- (CAS); 2-Phenox	000122-99-6	87	*used as bactericide*
64	21,18	0,33	Benzenepropanoic acid, methyl ester (	000103-25-3	93	
67	21,63	0,12	1-METHYLINDOLE	000000-00-0	90	
68	21,79	0,17	CIS-THUJAN-10-OIC ACID METHYL EST	067246-65-5	86	
71	22,23	0,25	Decanoic acid, methyl ester (CAS)	000110-42-9	95	*FAME (capric acid)*
73	22,43	0,13	2,6-Octadienoic acid, 3,7-dimethyl-,	002349-14-6	87	
77	22,92	0,18	1-chlor-2,2,3,3-tetramethyl-1-(1-prop	080631-33-0	91	
82	23,56	0,15	Tridecanoic acid, 3-methyl-, methyl e	002412-84-2	78	
83	23,67	0,3	Benzene, 1,3,5-trichloro-2-methoxy- (	000087-40-1	96	
85	23,87	0,11	Phenol, 4-(methoxymethyl)-2,6-dimethy	005048-02-2	83	
86	24,1	0,66	methyl anthranilate	000134-20-3	94	***MA (etheric oil;flavour)***
88	24,31	0,12	Benzoic acid, 3,4-dimethyl-, methyl e	038404-42-1	76	
90	24,8	0,15	3-Methoxy-4b-methyl-cis-4b-,5,6,7,8,8	094272-70-5	89	
91	24,96	0,66	Benzoic acid, 4-(1-methylethyl)-, met	020185-55-1	97	
92	25,04	0,47	Benzenebutanoic acid, methyl ester (C	002046-17-5	94	
94	25,35	0,27	Benzoic acid, 4-methoxy-, methyl este	000121-98-2	80	
98	26,04	0,16	4-(1-cyclohexenyl)-2-trimethylsilyl-1	000000-00-0	83	
101	26,44	0,19	METHYL N-METHYLANTHRANILATE	000000-00-0	90	
105	27,34	0,35	PARA-T-BUTYL-BENZOIC ACID, METHY	026537-19-9	95	*PTBBA*
108	27,73	0,43	1,2-Benzenedicarboxylic acid, dimethy	000131-11-3	97	*DMF (i.e. insecticide)*
117	28,91	0,14	DIHYDRO-NEOCLOVENE-(II)	000000-00-0	90	
118	29,02	0,38	Dodecanoic acid, methyl ester (CAS)	000111-82-0	98	***FAME (lauric acid)***
122	29,67	0,34	1,4-Benzenedicarboxylic acid, dimethy	000120-61-6	97	*manufact. of polyesters*
124	30,05	0,81	Benzeneacetic acid, .alpha.-methyl-4-	061566-34-5	96	***Ibuprofen (pain killer)***
125	30,2	0,56	Nonanedioic acid, dimethyl ester (CAS	001732-10-1	91	*(Azelaic acid)*
128	30,78	0,07	2(1H)-Naphthalenone, octahydro-4a-met	054594-42-2	83	
136	32,23	0,46	7-TERT-BUTYL-4-METHYL-5-NITROBEN	072900-75-5	83	
138	32,55	0,44	Acetic acid, (2,4-dichlorophenoxy)-, methyl	001928-38-7	76	***2,4-D (herbicide)***
139	32,91	0,12	r-1,c-5,c-6-trimethylspiro[bicyclo[4.	100692-74-8	83	
142	33,19	0,12	2H-1,4-Benzothiazin-3(4H)-one (CAS)	005325-20-2	83	
144	33,45	0,38	Benzothiazole, 2-(methylthio)- (CAS)	000615-22-5	95	
146	33,75	0,12	2,3-Dimethoxy-1-phenyl-5,5-dimethylcy	077787-40-7	83	
149	34,46	0,23	1-fluoro-2-(t-pentyl)-1H-phosphirene	131974-65-7	83	
151	34,69	0,2	1-Naphthalenecarboxylic acid, methyl	002459-24-7	93	
155	35,18	0,61	Tetradecanoic acid, methyl ester	000124-10-7	98	***FAME (myristic acid)***
157	35,72	0,17	2(1H)-Quinolinone, 1-methyl- (CAS)	000606-43-9	95	
162	36,57	0,25	Acetic acid, (2,4,5-trichlorophenoxy)	001928-37-6	86	***2,4,5-T (herbicide)***
164	36,96	0,78	Pentadecanoic acid, methyl ester	007132-64-1	97	
165	37,22	0,51	Tetradecanoic acid, 12-methyl-, methy	005129-66-8	96	
166	37,3	0,12	3-(2-isopropylphenoxy)pyridazine	000000-00-0	90	
170	38,02	0,44	Pentadecanoic acid, methyl ester (CAS	007132-64-1	98	
175	38,76	0,25	1-(1-METHOXYCARBONYLETHYL)-4-(2-	000000-00-0	93	
177	39,07	0,28	DIMETHYL ESTER OF 3-OXO-TETRAD	000000-00-0	91	
178	39,2	0,35	DECADEUTEROPHENANTHRENE	001517-22-2	94	*INTERNAL STANDARD*
181	39,72	0,59	Hexadecanoic acid, methyl ester (CAS)	000112-39-0	90	***FAME (palmitic acid)***

NI-SW (March 2007) F3 (polar fraction) (2/2)

Peak no.	ret. time	area %	substance	CAS number	Qua-lity	*comment*
182	40,01	0,12	2(1H)-Naphthalenone, 3,4,4a,5,6,7,8,8	024795-35-5	90	
183	40,18	0,31	11-Hexadecenoic acid, methyl ester (C	055000-42-5	98	
185	40,39	1,3	9-Hexadecenoic acid, methyl ester, (Z	001120-25-8	99	***FAME (palmitoleicacid)***
188	40,79	1,36	Hexadecanoic acid, methyl ester (CAS)	000112-39-0	98	***FAME (palmitic acid)***
192	41,44	0,78	Caffeine	000058-08-2	95	***caffeine (coffee, tea)***
194	41,76	0,77	Tridecanedioic acid, dimethyl ester (	001472-87-3	90	*(brassylic acid)*
196	42,07	0,54	Methyl 3-[3-(methoxycarbonyl)-4-methy	072719-12-1	86	
199	42,6	0,16	Hexadecanoic acid, 9-methyl-, methyl	000000-00-0	78	
201	43,01	0,32	2,5-Furandione, 3-(dodecenyl)dihydro-	025377-73-5	93	
203	43,34	0,39	Hexadecanoic acid, 14-methyl-, methyl	002490-49-5	90	
207	44,14	0,65	Methyl 2-ethylhexyl phthalate	000000-00-0	78	
213	44,96	0,43	12a-Methoxy-5H,12aH-[2]benzopyrano-(4	058963-54-5	83	
214	45,19	0,47	9,12-Octadecadienoic acid, methyl est	002566-97-4	99	
215	45,28	0,55	9-Octadecenoic acid (Z)-, methyl ester	000112-62-9	99	***FAME (oleic acid)***
216	45,41	0,53	8-Octadecenoic acid, methyl ester (CA	002345-29-1	99	
218	45,71	0,81	Benzene, 1,1'-(1-methylethylidene)bis	001568-83-8	90	*industrial chemical*
219	45,83	0,73	Octadecanoic acid, methyl ester (CAS)	000112-61-8	99	***FAME (stearic acid)***
220	46,11	0,17	.alpha.-Caryophyllene alcohol; 4,8-	004586-22-5	89	
224	46,69	0,42	Benzoic acid, 2-[(2,3-dimethylphenyl)	001222-42-0	97	
228	47,69	0,22	6-(Isopropoxycarbonyl)-5,8-dimethoxy-	000000-00-0	87	
233	48,68	0,21	2-Dodecen-1-yl(-)succinic anhydride	019780-11-1	90	
239	49,61	0,22	1H-Indene, 5-butyl-6-hexyloctahydro-	055044-36-5	91	
244	50,45	0,4	METHYL EICOSANOATE	000000-00-0	98	
246	50,76	0,25	2-(2-Hydroxyphenyl)benzimidazole; P	002963-66-8	86	
247	50,96	0,22	Culmorin	000000-00-0	80	
254	52,08	0,55	1-Phenanthrenecarboxylic acid, 1,2,3,	001235-74-1	97	
257	52,57	0,17	Longifolenaldehyde; 1,2,4-Methenoaz	019890-84-7	80	
260	53,15	0,3	Pyrrolidine, 1-(6-phenyl-1-cyclohexen	026974-24-3	91	
261	53,33	0,33	2(1H)-Naphthalenone, octahydro-4a-met	054594-42-2	84	
263	53,67	0,34	methyl pentadecanoate / anteiso chain	000000-00-0	80	
264	53,82	0,37	Eicosane, 2,6,10,14,18-pentamethyl- (	051794-16-2	90	*alkane*
266	54,1	0,27	5,8-epoxy-5,8-dihydroetinoic acid	129520-10-1	90	
268	54,38	0,26	4-naphthylcytosine; 4-naphthylamino	127970-28-9	83	*biomarker*
270	54,74	0,4	Docosanoic acid, methyl ester (CAS)	000929-77-1	95	
278	55,85	0,21	Eicosane (CAS); n-Eicosane	000112-95-8	95	*alkane*
282	56,71	0,47	PERDEUTERO-CHRYSENE; Chrysene-d	001719-03-5	98	*INTERNAL STANDARD*
285	57,25	0,2	23,24-BISNORCHOLA-5,17(20)-DIEN-3.	072654-92-3	84	
286	57,63	0,3	DEHYDROABIETIC ACID, METHYL EST	000000-00-0	99	
287	57,82	0,3	14-.BETA.-H-PREGNA; 14-.BETA.-PREG	000000-00-0	96	*biomarker*
289	58,16	0,39	Phenanthrene, 3,6-dimethoxy-9-(4-methox	133628-01-0	91	*PAH*
291	58,69	0,42	Tetracosanoic acid, methyl ester (CAS	002442-49-1	96	
293	59,05	0,31	Penduletin; 4',5-Dihydroxy-3,6,7-tr	000569-80-2	90	
296	59,71	0,16	Octadecane (CAS); n-Octadecane; O	000593-45-3	87	*alkane*
303	60,87	0,23	1-(ETHOXY-2,2,2-D3)-9,10-ANTHRAQU	027715-49-7	78	
307	61,47	0,26	Cholest-3-ene, (5.alpha.)- (CAS); 5	028338-69-4	99	*biomarker*
308	61,64	0,43	Nonadecane (CAS); n-Nonadecane	000629-92-5	78	*alkane*
310	62,14	0,22	(+)-Aromadendrene; 1H-Cycloprop[e]a	000489-39-4	90	
311	62,26	0,29	6,7-Bis(trimethylsilyl)-2,3-naphthale	080964-24-5	90	
323	64,24	0,33	Cholesta-3,5-diene (CAS); Cholester	000747-90-0	89	*biomarker*
325	64,62	0,43	bis-(octylphenyl)-amine	000000-00-0	93	
326	64,84	0,72	12-Methoxy-8,12-abietadien-6,11,14-tr	072505-94-3	90	
329	66,13	0,54	2-[(4-Methoxyphenyl)amino]-3,6-dioxo-	092544-18-8	78	
331	66,64	0,23	Perylene-d12	001520-96-3	92	*INTERNAL STANDARD*
340	69,04	0,48	Dihydrocholesterol; Cholestan-3-ol,	000080-97-7	99	*biomarker*
345	70,73	0,31	Cholest-5-en-3-ol (3.beta.)- (CAS)	000057-88-5	99	***biomarker(Cholesterol)***
	71,19	1,27	Cannabidiol; 1,3-Benzenediol, 2-[3	013956-29-1	52	***CBD (cannabis)***

NI-PZ (March 2007) F1 (non polar fraction)

Peak no.	ret. time	area %	substance	CAS number	Qua-lity	*comment*
1	11,99	3,55	1,4-Dichlorobenzene-d4	003855-82-1	91	*INTERNAL STANDARD*
2	12,12	1,73	1,2-DICHLOROBENZENE-D4	000000-00-0	95	*INTERNAL STANDARD*
3	15,29	0,46	4,5--epoxy-1-isopropyl-4-methyl-1-cyc	000000-00-0	78	
4	17,36	1,48	2-Dodecene, (Z)- (CAS)	007206-26-0	96	
5	18,57	5,88	Naphthalene-d8; Naphthalene-d8-	001146-65-2	91	*INTERNAL STANDARD*
8	24,64	0,41	1-Tetradecene (CAS); n-Tetradec-1-e	001120-36-1	91	*alkane*
9	27,36	2,3	2,6-di(t-butyl)-4-hydroxy-4-methyl-2,	5 000000-00-	9	
10	28,26	1,05	Benzene, 1,2,3,4-tetramethyl-5-(1-met	061142-67-4	83	
11	28,9	4,7	Phenol, 2,4-bis(1,1-dimethylethyl)- (	000096-76-4	95	*phenole*
12	29,06	0,61	Phenol, 2,6-bis(1,1-dimethylethyl)-4-	000128-37-0	95	
18	32,39	2,69	1,2-Benzenedicarboxylic acid, diethyl	000084-66-2	78	*phthalate: DEP (plast.)*
20	34,44	4,76	HEPTADECANE	000000-00-0	97	*alkane*
26	36,39	0,44	Phenol, 2-methyl-5-(1-methylethyl)- (	000499-75-2	87	
33	39,65	9,48	Anthracene-d10-	001719-06-8	95	*INTERNAL STANDARD*
34	40,05	10,5	1,2-Benzenedicarboxylic acid, bis(2-m	000084-69-5	83	*phthalate: DIBP (plast.)*
35	40,21	0,81	Anthracene-d10-	001719-06-8	80	*INTERNAL STANDARD*
38	42,68	8,6	1,2-Benzenedicarboxylic acid, dibutyl	000084-74-2	94	*phthalate: DBP (plast.)*
39	43,07	0,66	Phenol, 2,6-bis(1,1-dimethylethyl)-4-	000128-37-0	90	
41	47,65	0,34	2-(1'-METHYLINDOL-3'-YL)ETHENE-1,1-	065037-75-4	89	
42	47,75	0,66	1,4b,5,6,10,10a-Hexahydro-4b.beta.,8,	000000-00-0	78	
44	54,33	0,57	Eicosane (CAS); n-Eicosane	000112-95-8	78	*alkane*
46	57,33	4,13	PERDEUTERO-CHRYSENE; Chrysene-d	001719-03-5	89	*INTERNAL STANDARD*

NI-PZ (March 2007) F2 (medium polar fraction)

Peak no.	ret. time	area %	substance	CAS number	Qua-lity	*comment*
1	12,21	0,52	1,4-Dichlorobenzene-d4	003855-82-1	91	*INTERNAL STANDARD*
	18,55	0,4	Naphthalene-d8; Naphthalene-d8-	001146-65-2	80	*INTERNAL STANDARD*
4	29,04	0,4	7-Methoxy-2,2-dimethyl-2H-1-benzothio	086778-10-1	78	
	29,92	1,41	Acenaphthene-d10	015067-26-2	87	*INTERNAL STANDARD*
11	39,86	2,95	Anthracene-d10-	001719-06-8	93	*INTERNAL STANDARD*
15	42,82	5,69	1,2-Benzenedicarboxylic acid, dibutyl 1	000084-74-2	94	*phthalate: DBP (plast.)*
21	57,51	1,23	PERDEUTERO-CHRYSENE; Chrysene-d	001719-03-5	96	*INTERNAL STANDARD*
23	58,41	1,16	Eicosane (CAS); n-Eicosane 1	000112-95-8	94	*alkane*
27	62,35	7,88	Eicosane (CAS); n-Eicosane 1	000112-95-8	97	*alkane*
28	64,57	1,96	Heneicosane (CAS); n-Heneicosane 1	000629-94-7	95	*alkane*
30	67,09	2,59	Docosane (CAS); n-Docosane; C22H4 1	000629-97-0	93	*alkane*
33	70,02	3,79	Eicosane (CAS); n-Eicosane 1	000112-95-8	96	*alkane*
35	73,49	8,88	EICOSANE 1	000000-00-0	93	*alkane*
37	77,65	7,78	Eicosane (CAS); n-Eicosane 1	000112-95-8	97	*alkane*
38	82,68	4,12	3-Methylheneicosane; Heneicosane, 3 1	006418-47-9	93	
39	88,73	3,59	Eicosane (CAS); n-Eicosane 1	000112-95-8	93	*alkane*

NI-PZ (March 2007) F3 (polar fraction)

Peak no.	ret. time	area %	substance	CAS number	Qua-lity	*comment*
1	9,21	0,23	Phosphoric acid, trimethyl ester (CAS	000512-56-1	87	
10	11,35	0,73	4-methylthio-2-butanone	000000-00-0	90	
17	12,67	4,64	Ethane, 1,2-bis(methylthio)- (CAS)	006628-18-8	80	
23	14,15	3,56	2-Cyclohexen-1-one, 3-methyl- (CAS)	001193-18-6	90	*MCH (pesticide)*
27	15,39	0,75	Octanoic acid, methyl ester (CAS)	000111-11-5	95	*FAME (caprylic acid)*
28	15,58	0,59	Dimethyl trans-1,2-Cyclopropanedicarb	000000-00-0	78	
35	18,35	0,11	DIMETHYL ESTER OF 2-BUTEN-1,2-DIC	013314-92-6	93	
36	18,78	0,84	Naphthalene-d8; Naphthalene-d8-	001146-65-2	90	*INTERNAL STANDARD*
37	19,09	0,85	Nonanoic acid, methyl ester (CAS)	001731-84-6	97	
39	19,94	0,36	Ethanol, 2-phenoxy- (CAS); 2-Phenox	000122-99-6	90	*used as bactericide*
40	20,19	0,46	Hexanedioic acid, dimethyl ester (CAS	000627-93-0	91	
43	21,76	0,61	DIMETHYL ESTER OF THIODIACETIC A	000000-00-0	91	
47	22,73	0,45	Decanoic acid, methyl ester (CAS)	000110-42-9	97	*FAME (capric acid)*
60	27,14	0,8	1,6-Dimethyl-2-cyano-3-ethyl-3-piperi	073657-69-9	90	
65	27,89	2,28	PARA-T-BUTYL-BENZOIC ACID, METHY	026537-19-9	95	*PTBBA*
66	28,25	1,56	1,2-Benzenedicarboxylic acid, dimethy	000131-11-3	97	*DMF (i.e. insecticide)*
71	29,55	0,17	Dodecanoic acid, methyl ester (CAS)	000111-82-0	93	***FAME (lauric acid)***
73	29,91	1,33	Z-jasmone	000000-00-0	91	
75	30,26	0,31	1,3-Benzenedicarboxylic acid, dimethy	001459-93-4	87	*MORFLEX 1129*
84	32,75	0,26	1-methyl-6-(1-oxoethyl)-3-oxo-4-prop-	125506-07-2	83	
97	35,71	0,38	Tetradecanoic acid, methyl ester (CAS	000124-10-7	94	*FAME (myristic acid)*
107	38,57	0,41	Pentadecanoic acid, methyl ester (CAS	007132-64-1	89	
112	39,81	1,19	Anthracene-d10-	001719-06-8	91	*INTERNAL STANDARD*
115	40,62	0,13	2,3-Dihydro-5,5-dimethyl-8-methoxy-5H	136545-79-4	78	
118	41,3	1,23	Hexadecanoic acid, methyl ester (CAS)	000112-39-0	97	***FAME (palmitic acid)***
120	41,78	0,61	endo-3,4-isopropylidenedioxy-exo-2-ph	130177-69-4	83	
133	44,39	0,71	11-methyl-6H-pyrido[4,3-b]carbazol-5-	108320-78-1	83	
141	45,82	0,27	7-Octadecenoic acid, methyl ester (CA	057396-98-2	87	
143	46,28	0,54	9-oxo-6-methoxy-1,4a(S)-dimethyl-2,3,	023526-56-9	78	
144	46,37	0,64	Octadecanoic acid, methyl ester (CAS)	000112-61-8	98	***FAME (stearic acid)***
146	47,26	0,14	Benzoic acid, 2-[(2,3-dimethylphenyl)	001222-42-0	86	
149	48,15	0,28	N-(3',3'-dimethylindolin-2'-on-1'-yl)	000000-00-0	76	
164	53,27	0,54	CIS-3-PROPOXY-B-METHYL-B-NITROST	000000-00-0	83	
174	55,35	0,69	CIS-3-PROPOXY-B-METHYL-B-NITROST	000000-00-0	86	
178	57,27	0,37	1H-Indole, 2-methyl-3-phenyl- (CAS)	004757-69-1	78	
179	57,38	0,65	PERDEUTERO-CHRYSENE; Chrysene-d	001719-03-5	91	*INTERNAL STANDARD*
180	57,53	0,7	1H-Indole, 2-methyl-3-phenyl- (CAS)	004757-69-1	78	
197	64,56	0,31	Tetracosane (CAS); n-Tetracosane	000646-31-1	92	
202	67,08	0,27	Pentacosane (CAS); n-Pentacosane	000629-99-2	83	
204	67,68	0,51	Perylene-d12	001520-96-3	91	*INTERNAL STANDARD*