REMOVAL OF PHARMACEUTICAL RESIDUES AND OTHER PERSISTENT ORGANICS FROM MUNICIPAL SEWAGE AND SURFACE WATERS APPLYING MEMBRANE FILTRATION

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ABSTRACT

In military out of area missions, it may be necessary to produce drinking water even from highly contaminated surface waters. These waters may contain a large number of organic, inorganic, and microbiological contaminants such as pharmaceutically active compounds (PhACs), pesticides, flame retardants, and heavy metals. The mobile drinking water purification units must be able to remove such contaminants as much as possible to meet the requirements of the maximum tolerance levels for drinking water set by the German and also by the European legislation. In the course of a research project, we investigate two drinking water purification units using the membrane filtration technique. Presently, these units undergo extensive long-term trials. If the testing of the new devices is positive regarding their functionality and their ability to remove all possible contaminants (also including radioactive compounds), they shall substitute the conventional devices using some chemicals and charcoal filtration for water purification. The new membrane purification units are much lower in costs and the drinking water that is generated has much higher acceptance regarding its organoleptic quality. In 2000, we tested a mobile drinking water purification unit, commercially available since 1999. This unit is able to generate up to 1600 liters of drinking water per hour. In a field study at the Teltowkanal, a canal carrying a high burden of municipal sewage effluents, the functionality and the efficiency of this device was tested to remove high amounts of algae, microbes, and organic and inorganic pollutants. The results from this fatigue test proved the ability of the water purification unit to reduce all contaminants to meet the maximum tolerance levels set by the drinking water regulation. Residues of PhACs have almost totally been removed from the surface water where these contaminants are found at individual concentrations up to the µg/L level. The prefiltration device was very effective in its ability to remove algae and solid particles, to protect the membranes from clogging, and to enable an almost maintenance-free operation. Some results from this study will be presented in this paper.

INTRODUCTION

<u>General Considerations</u>

In recent years, the occurrence and fate of PhACs in the aquatic environment was recognized as one of the emerging issues in environmental chemistry and a matter of public concern. Residues of PhACs have been found as contaminants in several investigations of sewage, surface, and ground water samples (Stan & Heberer, 1997; Halling-Sørensen et al., 1998; Heberer

& Stan, 1998; Ternes, 1998; Daughton & Ternes, 1999; Möhle et al., 1999; Wilken et al., 2000; Heberer, in press). Due to the high amounts of pharmaceuticals prescribed in human medical care, considerable amounts of persistent drug residues are passed through the municipal sewage plants which therefore act as point sources for the neighboring waters. Thus, drug residues are found at concentrations up to the µg/l-level in surface waters contaminated by municipal sewage effluents. Wherever contaminated surface water is used for ground water recharge in drinking water production, the polar drug contaminants can also cause problems to drinking water supplies by leaching into the ground water aquifers of the drinking water plants (Heberer et al., 1997 & 2001; Heberer, in press).

In general, there is a need to focus on reducing or if possible eliminating the release of organic compounds into surface water. This may be achieved by restricting their use or by using modern technologies such as new, highly effective sewage treatment technologies. The use of some organic contaminants may be prohibited to protect the aquatic environment but the main purpose of pharmaceuticals is given by their medical indication. Thus, the use of a particular compound can and should not be generally banned for environmental reasons. Nevertheless, restrictions for their use may be desirable and achievable in the future. This may be especially important with regard to possible effects such as resistances of bacteria against antibiotics. As far as the medical indication allows, drugs showing a negative environmental behavior in environmental risk assessment studies could and should be replaced by other compounds.

Many of the above mentioned aspects have already been discussed and will possibly be put into future legislation. In the meantime, it is necessary to assess the present situation. PhACs occur at concentrations up to the µg/L-level in surface water and in a few cases, at very low concentrations also in ground and drinking water. These low concentrations may, from a toxicological point of view, not be harmful to humans but their occurrence in ground or drinking water is also not desirable from a hygienic point of view (Heberer & Stan, 1998; UBA, 2000) or with regard to the precautionary principle. Thus, there is a need to develop and study new drinking water treatment technologies to remove such organic contaminants from drinking water.

<u>Aims of The Research Project Funded by the German Ministry of Defense</u>

In 1999, the German Ministry of Defense initiated a research project on this issue. This project has three defined objectives. The first one is the investigation of PhAC residues in ground water wells used for drinking water production by the German Army. Secondly, the occurrence and fate of PhAC residues in the effluents from an Army hospital in Berlin is currently under investigation. This is done both by calculations of the pharmaceutical loads deriving from prescription amounts and knowledge on pharmacokinetics and by confirmatory measurements of target compounds in the individual and combined hospital effluents. The fate of these residues will be investigated down to the sewage treatment plants and the receiving surface waters. A final goal is to provide an environmental risk assessment for the compounds discharged from this particular hospital and to compare and apply these results to other military and civil hospitals. In the third part of this project we investigate the effectiveness of new, mobile drinking water treatment techniques for the removal of PhACs, some other contaminants, and microbes.

<u>Use of Mobile Drinking Water Purification Units in Foreign Military Missions</u>

In the course of foreign military missions, a reliable and save drinking water supply for the soldiers and the other military personnel is one of the most important logistics and has to be guaranteed. Public water supply is often not available (not sufficient, damaged, or destroyed) or safe. Thus, it may be necessary to produce sufficient and clean drinking water even from highly contaminated surface water. Such waters may contain various organic, inorganic, and microbial pollutants such as PhACs, pesticides, industrial chemicals, heavy metals, or pathogen bacteria. Independent of the origin of the surface water, the mobile drinking water purification units have to reduce the concentrations of any contaminant as much as possible to supply hazardous-free drinking water to the costumers and to meet the requirements of the European drinking water regulations (EEC, 1980; 1998) or additional standards (e.g. STANAG 2136). The requirements for drinking water purification units designed for military use are even stronger than those for civil use as they also have to guarantee the removal of any kind of nuclear, biological, and chemical contaminations (NBC).

Conventional mobile purification units need many personnel for maintenance and large amounts of chemicals and active charcoal. Drinking water produced by conventional techniques meets all regulations but often lacks in acceptance among the costumers due to the way it is produced by using chemicals and charcoal powder and due to turbidity problems. With regard to their growing international duties, the Bundeswehr decided to replace the conventional purification units by modern ones using membrane filtration for purification. Before these units are used in military missions, they need to be tested over a long time-scale under extreme and "worst-case" conditions such as large amounts of various contaminants (including NBC), cold and hot temperatures, and high humidity.

In this paper the first results from our field investigations on the performance of a mobile drinking water purification unit at an urban canal highly polluted by municipal sewage effluents, are presented. This system which uses bag filter pre-filtration, reverse osmosis, UV-radiation (optional), and chlorination (optional) for water treatment is also commercially available.

DESCRIPTION OF THE MOBILE PURIFICATION UNIT, THE FIELD SITE AND THE ANALYTICAL METHODS

Description of the Mobile Water Purification Unit

The field study at the Teltowkanal was carried out with the mobile drinking water purification unit WTC 1600 GT (Figure 1), commercially available from Alfred Kärcher GmbH & Co., Winnenden, Germany. It has been designed to generate drinking water from surface, river, sea, and brackish water. The capacity of the unit has been calculated to produce a minimum of 1600 liters of drinking water per hour (only in single pass mode) meeting the requirements of the European and the German drinking water regulation (TVO).

The tested unit consisted of the following components: A power supply generator, a raw water supply pump (including a connection hose and a device for floating use), a pre-filtration unit (duplex bag filters with a particle separation <0.5 µm abs.), two water-cooled high pressure pumps, and a reverse osmosis unit using cross-flow technique (figure 2), a UV-disinfection unit, a post chlorination unit, a heating unit (to operate the system at very low temperatures), automatic cleaning device, measurement technology (e.g. for conductivity measurements), a control box (SPS control) to operate the unit automatically, and a one axis trailer.

Figure 1: Photography of the mobile drinking water purification unit Kärcher TWC 1600 GT at the field site near the Teltowkanal in September 2000.

Specifications:

Operation temperatures (air):	-32°C to +49°C, below 0°C the heating system is required
Storage temperatures:	-40°C to +72°C
Raw water temperature:	0 to +49°C
Humidity:	5 to 100%
Sea level:	0 to 3000 meters above sea level
Turbidity (NTU):	< 150
pH :	5-9
Mechanical resistance :	3.5 g
Raw water exploitation :	40-50%
Membrane elements :	Eight Filmtec SW 30 4040
Desalination rate :	< 99.7 % nominal
Raw water needed for operation :	about 4000L/h
Drinking water output (min.):	1600 liters per hour in single-pass mode (figure 3) 600 liters per hour in double-pass mode (figure 4)
Max. salt content:	55,000 ppm
Operating pressure :	max. 69 bar
Raw water pressure:	1.5-3 bar
Noise (generator):	max. 53 dB(A)
Sizes:	length: 2.8 meters; width: 1.6 meters; height: 1.05 meters
Weight (without trailer):	1550 kg

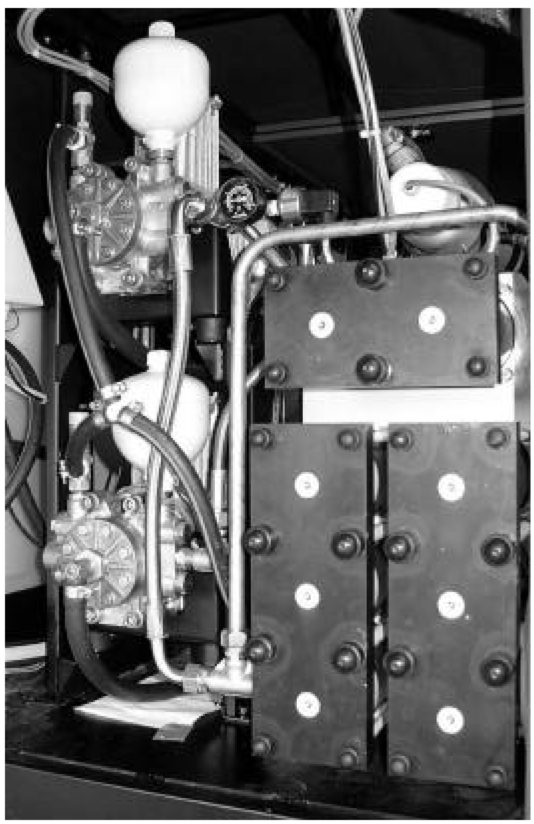

Figure 2: Photography of the reverse osmosis module of the mobile drinking water purification unit Kärcher TWC 1600 GT. High pressure pumps on the left and pressure pipes (eight 40'' membrane modules) of the reverse osmosis (right).

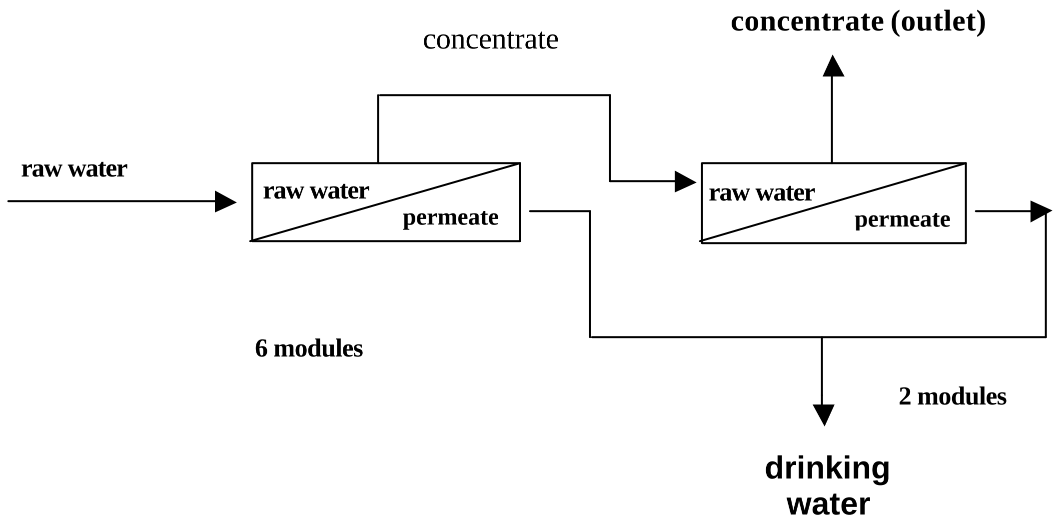

Figure 3: Flowscheme of the reverse osmosis unit operated in single pass-mode (generation of up to 1600 liters of drinking water per hour).

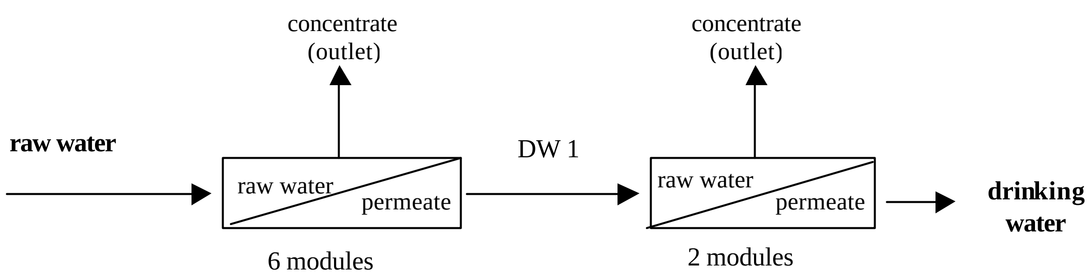

Figure 4: Flowscheme of the reverse osmosis unit operated in double pass-mode (generation of up to 600 liters of drinking water per hour).

As shown in Figure 3, the unit is ususally operated only in single-pass mode. The concentrate obtained from the first six membrane modules is again applied to the last two modules to increase the raw water extration yields. In case of nuclear cotaminations, the unit has to be run in double-pass mode, shown in Figure 4.

Description of the Field Site Where the Study Was Carried Out in September 2000

The performance of the drinking water purification unit was tested for more than one year at different (extreme) environmental conditions using all possible kinds of contaminants (including NBC). The purpose of our field trial carried out in September 2000 was to check the performance of the mobile drinking water purification unit under realistic but also under "worst-case" conditions. We wanted to test various contaminants but our main target compounds were the PhACs that have never been tested before.

What was needed for the field trial was a "natural" surface water containing municipal sewage discharges, contaminants from other sources, and many algae and particulates to test the practical performance including blocking of the pre-filtration unit. Thus, the Teltowkanal, a shallow canal located in the southern districts of Berlin, (Figure 5) was selected for the field study because it carries the highest loads of sewage discharges of all Berlin water ways. Additionally, it also contains many algae and much particulate organic matter that is distributed in the surface water by the shipping traffic. The Teltowkanal was build between 1901 and 1906 (SenStadtUm, 1987) and used as drainage for rainwater and industrial wastewater from districts formerly located outside of Berlin. It was also used as a shipping canal for industrial supply and as a short cut for the shipping routes between the rivers Oder and Elbe. The Teltowkanal has a total length of approximately 35 kilometers and connects the rivers Dahme and Havel (SenStadtUm, 1987). Today, it is characterized by high proportions of sewage effluents being discharged into the canal by the municipal sewage treatment plants (STPs) in Stahnsdorf and by Berlin's two largest STPs in Waßmannsdorf and Ruhleben (only from April to October). In several sections of the canal, the municipal sewage effluents account for up to 40 percent of the average surface water flow (SENSUT, 2000), but under extreme conditions (dry periods with low surface water flows) the proportions of municipal sewage may also reach up to 84 percent (SENSUT, 2000). As far as contaminations from municipal sewage discharges are concerned, the surface water of the Teltowkanal represents some kind of "worst-case scenario." As shown in Figure 5, the field study was carried out downstream from the sewers of the STPs Waßmannsdorf and Ruhleben to guarantee a high degree of contamination by municipal sewage effluents in the raw water.

<u>Parameters and Analytical Methods</u>

All samples were analyzed for various organic contaminants including pesticides, polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), and volatile compounds (chlorinated VOCs and BTX) using standardized test methods (DIN, German industrial standard methods). Standard methods were also applied for the analysis of some heavy metals, kations, anions, and microbes. Additionally, the samples were analyzed for 23 environmentally important organic contaminants including some PhACs and two flame retardants. Until now, no standard methods are available for these contaminants. Thus, we used a multi-method described below.

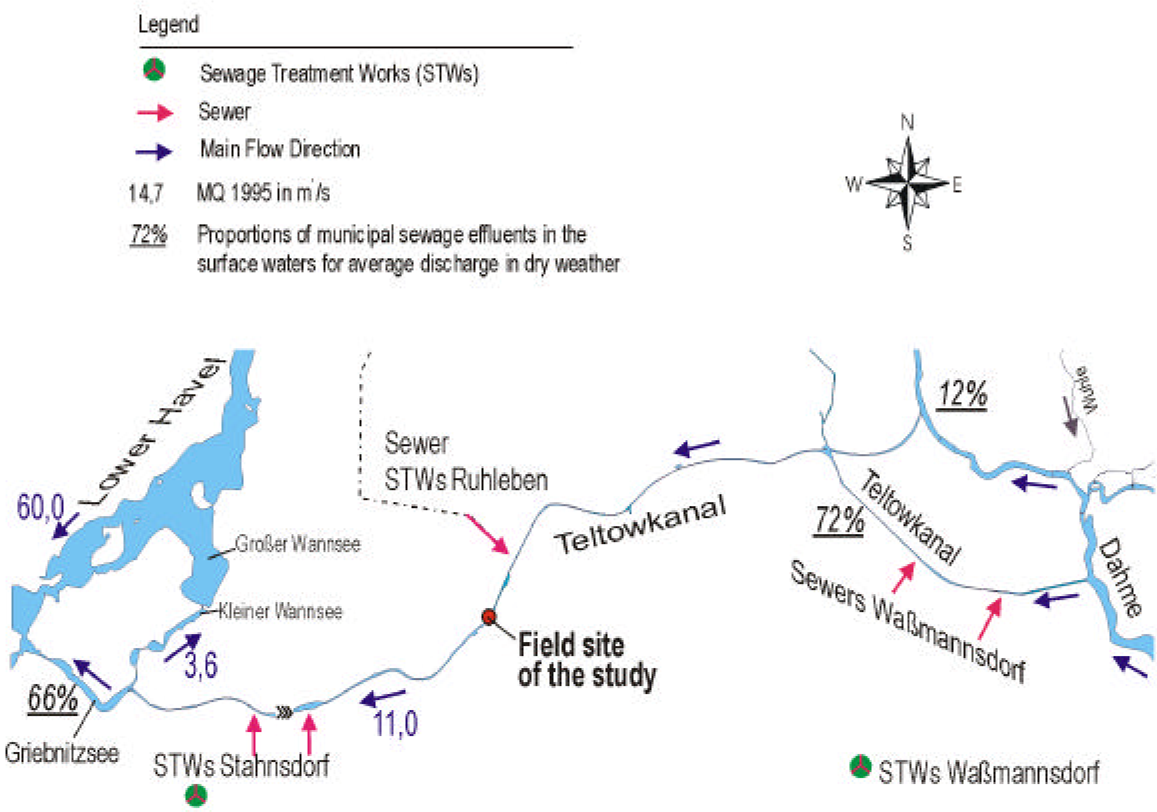

Figure 5: Map showing the field site at the Teltowkanal in Berlin, Germany, were the study was carried out in September 2000.

Analytical method for the determination of some PhACs, flame retardants, and other polar contaminants: All samples were filtered using 0.45 μm cellulose nitrate membrane filters and stored at 4°C prior to analysis. The pharmaceutical residues were analyzed using a multi-method described by Heberer et al. (1998) and Reddersen & Heberer (in prep.). For trace analysis, the samples were concentrated from (up to) one liter down to a final sample volume of 0.1 µl (concentration factor: up to 10,000). The method applies solid-phase extraction (SPE) using non-endcapped reversed-phase octadecyl adsorbent for the extraction of the analytes from the water samples. The extracted residues were then derivatized using pentafluorobenzyl bromide as derivatization reagent. The remaining residues were dissolved and analyzed applying gas-chromatography-mass spectrometry (GC-MS) using selected ion monitoring (SIM). The recoveries of the analytes were between 70 and 110 percent. For quality control, a suitable surrogate standard (2-(4-chorophenoxy)butyric acid) was added to the samples before sample preparation. The detection limits for the pharmaceuticals are between 1 and 10 ng/L, depending on the individual compounds, the sample volume and the sample matrix (Reddersen & Heberer, in prep.).

RESULTS OF THE FIELD TRIAL

Introduction

The WTC 1600 GT water purification unit was tested on September 5 and 6, 2000, at the Teltowkanal in Berlin, Germany. Its performance was run in single-pass (09/05/00) and double-pass mode (09/06/00) without additional purification such as UV disinfection or chlorination. This resulted in a total of more than 5000 individual values obtained and evaluated in terms of the field trial. Although the surface water of the Teltowkanal contained much organic matter such as algae and solid particles, the pre-filtration and the whole system worked with high reliability. The operational life of the bag filters used for pre-filtration varied between 1.5 and 2.5 hours depending on the shipping traffic in the canal. Whenever a bag filter was blocked, the system switched automatically (by pressure control) to the second filter without interrupting the purification process. In the following sections, selected results showing the performance of the purification unit in single-pass mode are presented.

Organic Parameters Including PhACs, Pesticides and Some Polar Organics

Several PhACs, pesticides, flame retardants, and other polar contaminants were detected in the surface water of the Teltowkanal at maximum individual concentrations up to 2.1 µg/L (detected for tris-(chloroisopropyl)-phosphate). As shown for some PhACs in Figure 6, temporal fluctuations of the concentrations were observed for several compounds. Due to the high proportions of municipal sewage effluents the fluctuating loads for the individual PhACs are still reflected by their varying surface water concentrations. Other compounds such as clofibric acid (metabolite of blood-lipid regulating compounds) were detected at almost constant concentrations (between 140 and 160 ng/L, Figure 6) over the whole sampling period (from 8 a.m. to 6 p.m.). This effect may be explained by the individual application practice and by differences in excretion.

In the finished water of the drinking water purification unit, none of the investigated PhACs or any other organic contaminant was detected at significant concentrations, neither when the system was operated in the single-pass mode nor when it was operated in the double-pass mode. The concentrations of all contaminants detected in the surface water of the Teltowkanal were reduced to concentrations below the analytical limits of detection (Table 1). Figures 7 and 8 show the varying concentrations of two PhACs in the raw water from the Teltowkanal and the efficacy of their removal in single-pass mode by the tested drinking water purification unit. The removal rates, which were calculated from the average raw water concentrations and the average finished water concentrations, are also shown in Table 1. In those cases, where the analytes were not detected in the permeate samples, the minimum removal rate was calculated from the individual limit of detection.

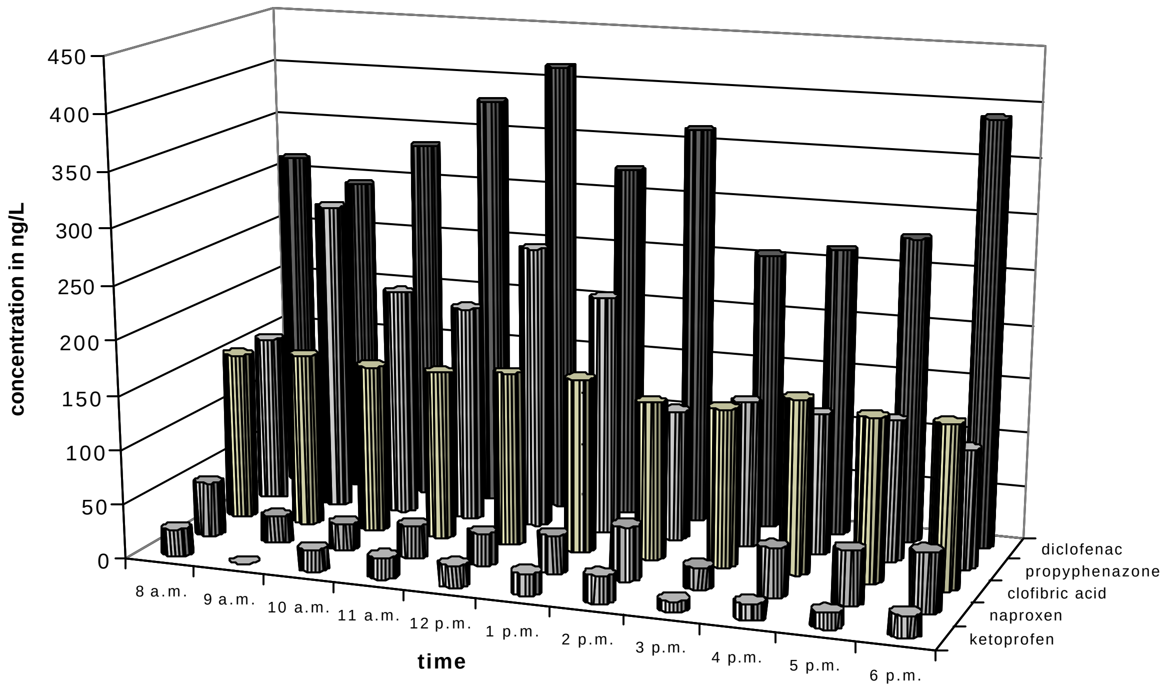

Figure 6: Temporal fluctuations of the concentrations measured for different drug residues at the selected location in the Teltowkanal. Repeated sampling on September 5, 2000.

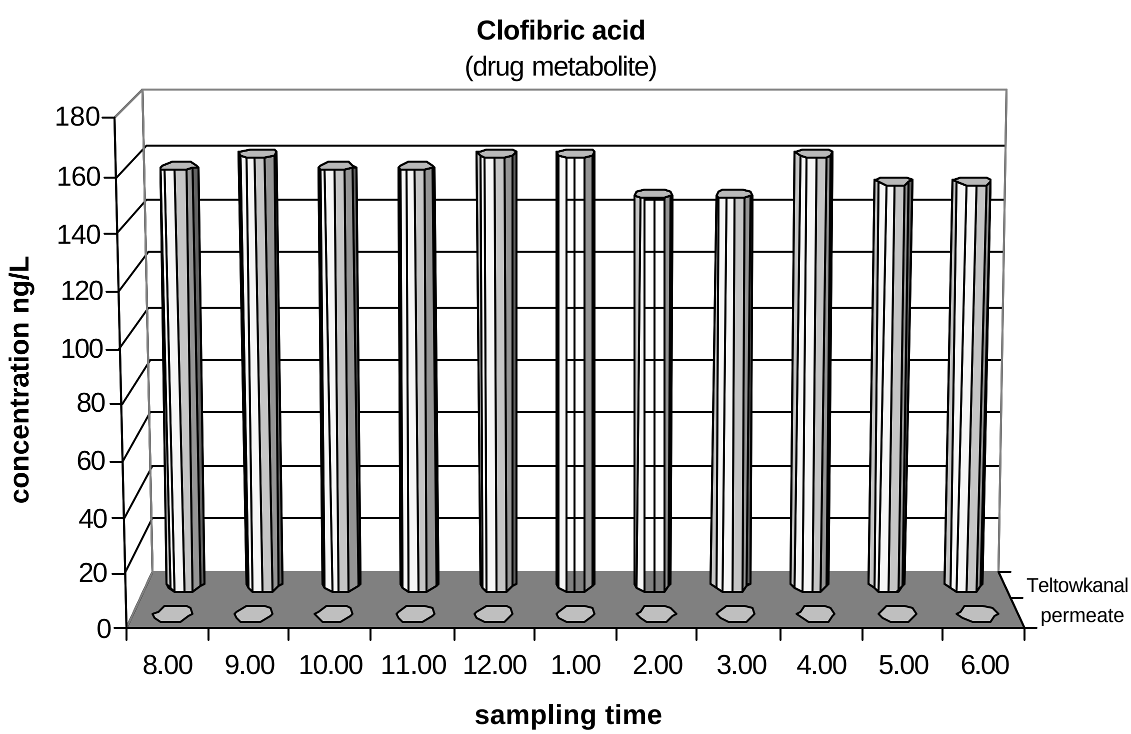

Figure 7: Concentrations of clofibric acid in the raw water from the Teltowkanal and in the permeate from the drinking water purification unit. Results from 09/05/00, when the system was operated in single-pass mode.

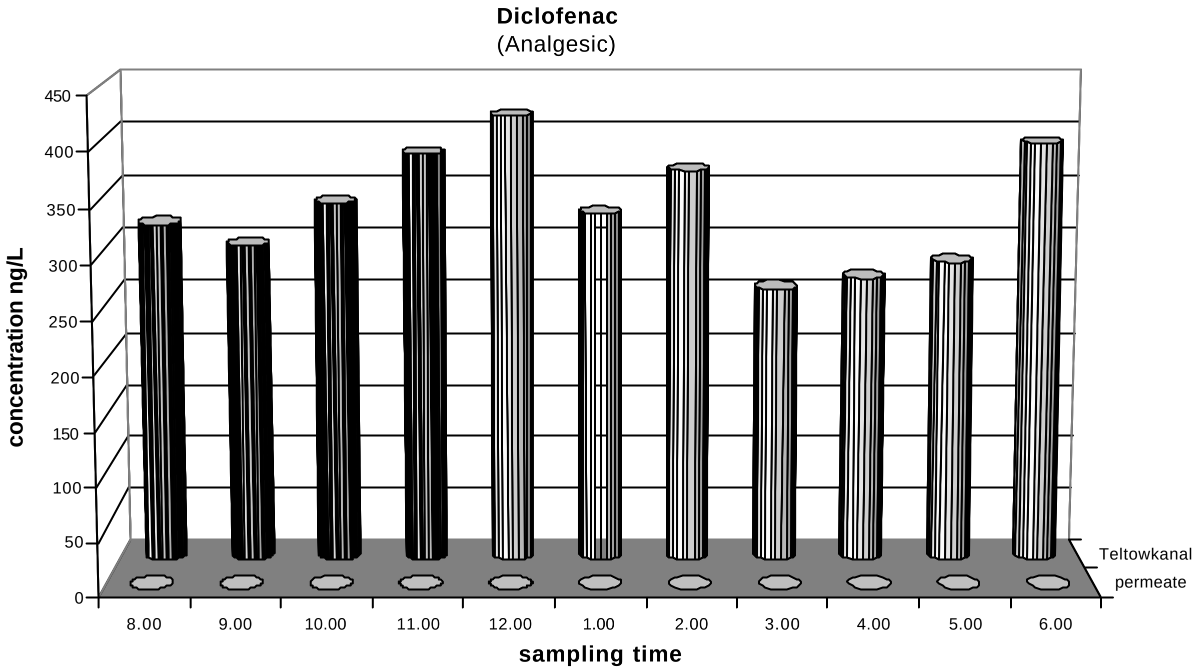

Figure 8: Concentrations of diclofenac in the raw water from the Teltowkanal and in the permeate from the drinking water purification unit. Results from 09/05/00 when the system was operated in single-pass mode.

Table 1: Average concentrations of selected contaminants in the raw water from the Teltowkanal and in the permeate after water purification using single-pass mode (09/05/00). Removal rates were calculated from the average raw water concentrations and the average permeate concentrations; and when analytes were not detected in the permeate samples, from the individual limit of detection.
[n.d.: not detected (below the limit of detection).]

Parameter	Average raw water concentration	Average permeate concentration	Removal rate in %
Anonymous*	290 ng/L	n.d.	> 99.7
Caffeine	430 ng/L	n.d.	> 99.8
Carbamazepine	330 ng/L	n.d.	> 99.7
Clofibric acid	155 ng/L	n.d.	> 99.4
Diclofenac	329ng/L	n.d.	> 99.7
Naproxen	38 ng/L	n.d.	> 95.0
N-(phenylsulfonyl)sarcosine	1143 ng/L	n.d.	> 99.9
Propyphenazone	170 ng/L	n.d.	> 99.4
Diurone	100 ng/L	n.d.	> 99.8
Mecoprop	93 ng/L	n.d.	> 98.9
Tris-(chloroethyl)phosphate	360 ng/L	< 10 ng/L	> 97.2
Tris-(2-chloroisopropyl)phosphate	945 ng/L	< 10 ng/L	> 98.9

* This compound has been identified as a metabolite of a pharmaceutical substance (Reddersen et al., in prep.). The identity of this compound cannot be publicized because of potential legal ramifications (Reddersen et al., in prep.).

Microbiology

As shown in Table 2, large amounts of bacteria including several species of faecal bacteria were detected in the surface water of the Teltowkanal (raw water). In the permeate the number of bacteria was almost totally reduced by the drinking water purification unit. Cross-contaminations during sampling or transport may be possible at a low extent. This may explain the low number of positive counts in the permeate samples.

Inorganic Parameters

As already shown for the organic contaminants, only small amounts of heavy metals, cations or anions were detected in the permeate from the drinking water purification unit, regardless of operating the system in single- or double-pass mode. The concentrations of all contaminants detected in the surface water of the Teltowkanal were reduced significantly often to concentrations below the analytical limits of detection (Table 3). The removal rates, which were calculated from the average raw water concentrations and the average permeate concentrations, are also shown in Table 3. In those cases, where of the analytes were not detected in the permeate samples, the minimum removal rate was calculated from the individual limit of detection.

Table 2: Selected results from the microbial investigations

single pass mode		8.00 a.m.	10.00 a.m.	12.00 p.m.	2.00 p.m.	4.00 p.m.	6.00 p.m
colony count 20 $^{+}$/. 2°C	canal	> 1000	> 1000	> 1000	> 1000	> 1000	> 1000
colony count 20 $^{+}$/- 2°C	permeate	3	2	3	0	4	2
colony count 36 $^{+}$/. 1°C	canal	> 1000	> 1000	> 1000	> 1000	> 1000	> 1000
colony count 36 $^{+}$/. 1°C	permeate	3	3	6	0	4	1
E.coli / specific Enterobacteriaceae	canal	E.coli/ coliforms	E.coli/ coliforms	E.coli/ coliforms	E.coli/ coliforms	E.coli/ coliforms	E.coli/ coliforms
E.coli / specific Enterobacteriaceae	permeate	negative	negative	negative	negative	negative	negative
Streptococcus faecalis / S. faecium	canal	positive	positive	positive	positive	positive	positive
Streptococcus faecalis / S. faecium	permeate	negative	negative	negative	negative	negative	negative
sulfite-reducing anaerobic growing bacteria	canal	positive	positive	positive	positive	positive	positive
sulfite-reducing anaerobic growing bacteria	permeate	negative	negative	negative	negative	negative	negative

Table 3: Average concentrations of selected inorganic parameters in the raw water from the Teltowkanal and in the permeate after water purification using single-pass mode (09/05/00). Removal rates were calculated from the average raw water concentrations and the average permeate concentrations and, when analytes were not detected in the permeate samples, from the individual limit of detection.

Parameter	Average raw water concentration	Average permeate concentration	Removal rate in %
Aluminium	0.39 µg/L	< 0.04 µg/L	> 87.2
Ammonia	0.34 mg/L	< 0.05 mg/L	> 85.3
Borate	0.24 mg/L	< 0.05 mg/L	> 79.5
Iron	0.77 µg/L	< 0.05 µg/L	> 93.5
Nitrate	18.84 mg/L	< 1.0 mg/L	> 94.7
Nitrite	0.39 mg/L	< 0.01 mg/L	> 97.4
Phosphate	0.96 mg/L	< 0.2 mg/L	> 79.2

CONCLUSIONS

The field trial of the mobile drinking water purification unit WTC 1600 GT showed the high efficiency and reliability of modern purification units applying membrane filtration. All contaminants detected in the surface water of the heavily polluted Teltowkanal, such as PhACs, pesticides, flame retardants, heavy metals, anions, and cations, were effectively removed by the system. For the spectrum of compounds investigated, the application of the double-pass mode was not found to be necessary. Double-pass mode operation is, however, inevitable to remove nuclear contaminations. Although the surface water of the Teltowkanal contained much organic matter such as algae and solid particles, the pre-filtration and the whole system worked with high reliability. The operational life of the bag filters used for pre-filtration varied between 1.5 and 2.5 hours depending on the shipping traffic in the canal. The generated drinking water meets all requirements set by the German and European drinking water regulations and by some other regulations such as NATO STANAG 2136.

OUTLOOK

This year in September, the performance of a prototype of another purification unit will be tested under "worst-case" conditions in another field trial. This newly designed system also applies membrane filtration (ultrafiltration and reverse osmosis) and is specified to produce as much as 5 m^3 per hour of drinking water in single-pass mode and 3.5 m^3 per hour in double-pass mode. In our investigations we will place this unit directly near the sewer of the municipal sewage treatment plants in Ruhleben (Berlin, Germany).

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