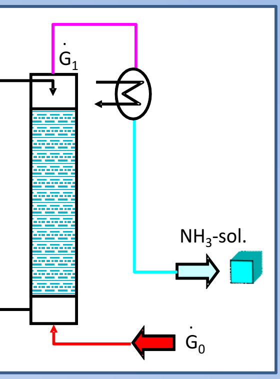
Felix Tettenborn **Separate urine collection and treatment**

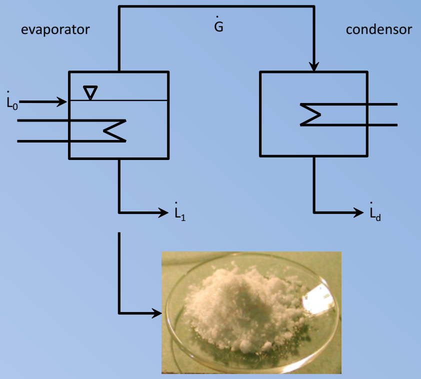
Hamburger Berichte zur Siedlungswasserwirtschaft

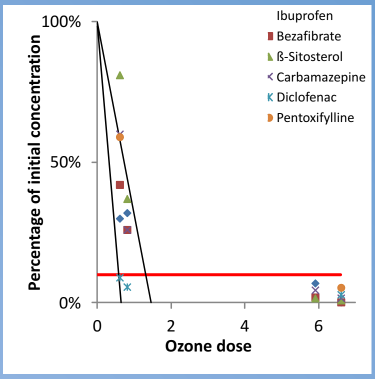
79

Felix Tettenborn

Aspects of systems for separate urine collection and treatment - Selected techniques and potential implementation in an urban context







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Aspects of systems for separate urine collection and treatment

Selected techniques and potential implementation in an urban context

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Abstract

In the world of sanitation new approaches for solutions for tomorrow are becoming more and more relevant. Since volume flow of urine is small but rich in nutrients compared to other wastewater flows, the separate collection and treatment of urine allows an effective resource recovery especially of the nutrients nitrogen and phosphorous. Additionally treatment efforts for other wastewater flows can be reduced and a large fraction of micropollutants e.g. pharmaceutical residues is extracted and can be reduced by separate collection and treatment of urine.

The current and future potential of separate collection and treatment of urine in an urban context was evaluated by an analysis of collectable volume and concentrations of urine from collection tanks of public urinals in the city of Hamburg, by modeling a transportation system, and by testing different treatment options for nutrient recovery and reduction of pharmaceutical residues in laboratory and pilot scale. The results of the analysis of collection, the modeling of transport, and the testing of treatment options were combined to evaluate the Hamburg example regarding the feasibility of a separate collection and treatment of urine system.

The findings of the analysis of collection tanks complement the existing data pool regarding real nutrient concentrations of source separated urine from public toilets.

For design of the transport model, consolidated findings from solid waste management could be transferred to a urine collection and transport system, due to similar specific volumes.

With the treatment methods, all objectives namely nutrient recovery, improvement of product handling, and reduction of pharmaceutical residues could be reached.

In a basic system an evaluation of the competitiveness of a system for separate collected and treated urine could be demonstrated for those with high fractions of higher concentrated urine.

Finally, the successful implementation of separate collection and treatment of urine in an urban context is dependent upon boundary conditions, in particular on objective of the existing or planned wastewater management concepts, on parameters such as costs and availability of competitive products, and on distances to agricultural areas with high nutrient demand. At appropriate boundary conditions, the separate collection and treatment of urine in densely populated areas is a promising detail in the context of new sustainable water and sanitation concepts.

Zusammenfassung

Im Bereich zukunftsfähiger und ressourcenorientierter Siedlungswasserwirtschaftskonzepte spielen integrierte und effektive Detaillösungen eine immer größere Rolle. Die getrennte Erfassung und Behandlung von Urin erlaubt in diesem Kontext eine effektive Abtrennung und Rückgewinnung von Nährstoffen (insbesondere N und P) aus einem verhältnismäßig kleinen, aber stark konzentrierten Volumenstrom. Durch die Abtrennung eines großen Nährstoffanteils wird der verfahrenstechnische Aufwand der Aufbereitung der restlichen Abwasserteilströme erheblich reduziert, die gewonnenen Nährstoffe können zu hochwertigen Düngemitteln aufbereitet in der Landwirtschaft eingesetzt werden. Zudem kann ein großer Anteil von Mikroschadstoffen, in Form von Arzneimittelrückständen, über eine Abtrennung und Behandlung von Urin erfasst und reduziert werden.

Um das aktuelle und zukünftige Potential der getrennten Erfassung und Behandlung von Urin in einem städtischen Kontext zu untersuchen, wurden Sammeltanks von öffentlichen Toilettenanlagen der Stadt Hamburg hinsichtlich erfassbarer Volumina und Konzentrationen untersucht, ein Transportmodell für Hamburg entworfen und verschiedene Behandlungsverfahren zur Nährstoffrückgewinnung und zur Reduktion von Arzneimittelrückständen im Labor- und Pilotmaßstab getestet. Die Ergebnisse der Elemente Erfassung, Transport und Behandlung wurden anschließend kombiniert, um am Beispiel Hamburgs den Einfluss unterschiedlicher Anschlussgrade und Sammlungstechniken auf die Wirtschaftlichkeit eines Gesamtsystems zur getrennten Erfassung und Behandlung von Urin zu evaluieren.

Die aus der Untersuchung der Sammeltanks der Stadt Hamburg gewonnenen Ergebnisse ergänzen den bisher nicht ausreichend belastbaren Datenbestand bezüglich realer Nährstoffkonzentrationen in getrennt erfasstem Urin aus öffentlichen Toilettenanlagen. Es zeigte sich, dass die Nährstoffkonzentrationen 45 % der in der Literatur angegebenen Durchschnittskonzentrationen von Urin liegen. Aufgrund der Konzentrationszusammensetzung konnte gefolgert werden, dass bei Phosphor ein Verlust von 10 % über Ausfällungsprozesse stattfindet und der Verlust von Stickstoff über Ammoniakausgasung vernachlässigbar ist. Verdünnungseffekte durch Fremdwassereintrag spielen eine untergeordnete Rolle. Das bedeutet, dass die niedrigen Konzentrationen im Substrat der Sammeltanks auf den Eintrag von niedrig konzentriertem Urin zurückzuführen sind.

Bei der Entwicklung des Transportmodells konnten, aufgrund ähnlicher spezifischer Volumina, Erkenntnisse aus dem Bereich der Abfallwirtschaft auf ein Urinsammel- und -abfuhrsystem übertragen werden. Der für die Abfuhr erforderliche Energieaufwand bewegte sich bei günstigen Randbedingungen in einem Bereich von unter 10 % der zur Erfassung und Behandlung erforderlichen Gesamtenergie.

Durch die Behandlungsverfahren konnten alle Ziele, namentlich Nährstoffrückgewinnung, Verbesserung des Produkthandlings und die Reduktion von Arzneimittelrückständen, erreicht werden. Durch eine Rektifikation im Pilotmaßstab konnte bei einer 97 %igen Abreicherung eine 12 %ige Ammoniaklösung als Produkt gewonnen werden. Nach theoretischen Berechnungen ist eine Rückgewinnung einer 20 % Ammoniaklösung möglich. Bei einer Eindampfung wurde ein 50 fach aufkonzentriertes Nährstoffprodukt erzielt, in dem Phosphor

vollständig enthalten blieb. Über eine anschließende Kristallisation konnten 90 % des Phosphors in kristalliner Form zurückgewonnen werden. Während über die Eindampfung ein erheblicher Teil der Arzneimittelrückstände reduziert werden konnte, hatte die kurze Aufenthaltszeit bei hohen Temperaturen im Rektifikationsreaktor keinen relevanten Einfluss auf Arzneimittelrückstände. Bei den ergänzenden Verfahren zur Reduktion von Arzneimittelrückständen zeigte sich, dass über die UVC-Bestrahlung im Labormaßstab ein rund 100 fach höherer Energieinput benötigt wird als bei einer Ozonierung. Der Energiebedarf für die Nährstoffrückgewinnungsverfahren bewegte sich im Rahmen des gemeinsamen Energiebedarfs, der für eine Nährstoffelimination in herkömmlichen Kläranlagen und die Herstellung von vergleichbaren Düngemitteln benötigt wird. Insgesamt konnte die technische Machbarkeit der durchgeführten Verfahren dargestellt und relevante Betriebsparameter zusammengestellt werden.

Über eine abschließende Beispielrechnung konnte gezeigt werden, dass bei einem höheren Anschlussgrad mit höher konzentrierten Fraktionen ein System für getrennt erfassten und behandelten Urin wettbewerbsfähig sein kann.

Die erfolgreiche Umsetzung einer getrennten Erfassung und Behandlung von Urin im urbanen Kontext ist von Randbedingungen abhängig, insbesondere von der Zielsetzung des vorhandenen oder geplanten Abwasserbehandlungskonzeptes sowie von Parametern wie Kosten und Knappheit von Konkurrenzprodukten und Entfernung landwirtschaftlich genutzter Bereiche mit großem Nährstoffbedarf. Es konnte dargestellt werden, dass bei entsprechenden Randbedingungen die getrennte Erfassung und Behandlung von Urin auch im dicht besiedelten Raum ein vielversprechender Baustein im Kontext einer nachhaltigen Siedlungswasserwirtschaft ist.

Content

List of figures

List of tables

List of abbreviations and symbols

AOP	Advanced oxidation process
AOX	Adsorbable Organohalogens
ave	Average
BSU	Behörde für Stadtentwicklung und Umwelt, Hamburg, Germany
BWB	Berliner Wasserbetriebe
C:N:P	Carbon-Nitrogen-Phosphorous ratio
cf	Concentration factor
CFU	Colony forming unit
COD	Chemical oxygen demand
Conc	Concentrate from evaporation process
Cond	Condensate from steam stripping/rectification process
cov	Coverage
DDD	Defined daily dose
df	Dilution factor
Dist	Distillate from evaporation process
d_{tour}	Distance per tour
d_{week}	Distance per week
eawag	Swiss Federal Institute of Aquatic Science and Technology
ecosan	Ecological Sanitation
equ	equivalent
F	Steam loading factor
f_d	Detour factor
$\dot{G}$	Molar flux of a gas during stripping/rectification process
GFK	Glass fiber reinforced plastic
GIS	Geographic information system
GTZ	Deutsche Gesellschaft für Technische Zusammenarbeit (GTZ) GmbH
He	Henry coefficient [bar mol/mol]
HSE	Hamburger Stadtentwässerung AöR, Part of Hamburg Wasser
HTU	Height of transfer unit
IWW	Rheinisch-Westfälisches Institut für Wasserforschung gGmbH
KMU	KMU Umweltschutz GmbH
KWB	Kompetenzzentrum Wasser Berlin
L	Molar liquid flux during stripping/rectification process
L1–L9	Code designation of location of urinals in Hamburg
LOD	Limit of detection
LOQ	Limit of quantification
MAP	Magnesiumammoniumphosphate
mol-%	Substance quantity in percentage
MP	Micro pollutants
na	not analyzed
nc	not calculable

n_{ctp}	Number of collection and treatment points
nd	no data available
N_{depl}	Nitrogen depleted substrate after stripping/rectification process
NH_3	Ammonia
NH_4-N	Ammoniacal nitrogen
norm	Normalized units
NOVAQUATIS	Cross-cutting Eawag research project concerned with urine source separation
n_{tours}	Number of tours
NTU	Number of transfer unit
n_{ves}	Number of collection vessels
p	Partial pressure
PhaR	Pharmaceutical residues
PO_4-P	Phosphate as phosphorous
priv	private
pub	public
Q	Quantity
S1 – S3	Scenario 1 to 3
SCST	Research and demonstration project
SuSanA	Sustainable Sanitation Alliance
t	Time
TC	Total carbon content
TN	Total nitrogen content
TOC	Total organic carbon
TP	Total phosphorous content
TS	Total solids, dried solid content (in German TR)
TUHH	Hamburg University of Technology
UD	Urine diversion
udt	Urine diversion toilet
UVC	UV-light below 240 nm
v	Velocity
ves	Collection vessel
WHO	World health organization
wlu	Waterless urinals
X_0	Initial concentration in liquid phase
X_1	Measured end concentration in liquid phase
X_N	Loading of depleted liquid after stripping/rectification process
X_t	Calculated end concentration in liquid phase
Y_0	Initial concentration of substance in gas phase
Y_1	Measured end concentration in gas phase
Y_t	Calculated end concentration in gas phase

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Prologue

The introduction of the water closet and sewer systems is often celebrated as one of the central blessings for the hygienic conditions in the paste in large cities like Hamburg, Germany, and London, England, where people suffered from diseases and miserable hygienic living conditions in the 19th century. The conditions were due to an enormous growth of population and at the same time a lack of wastewater infrastructure based on a lack of knowledge regarding hygienic threats connected with wastewater. The change in dealing with human excreta by implementing an infrastructure system allowing flushing them away with large quantities of water did yield tremendous improvements of the hygienic situation within the cities. However, it can be believed that this improvement was most likely also due to growing education and awareness of hygienic rules. Nevertheless, the implementation of large sewer systems did shape the picture of wastewater management up until now.

Whether we will witness a further change as experienced during the period of urbanization and industrialization remains uncertain. However, in respect to the growing importance of environmental protection and clean water worldwide, the next significantly different level of development in excreta management would probably be a waterless one. A clean and comfortable collection of undiluted matter (e.g. based on dry separation toilets with sealed collection bags for fecal matter and a separate urine collection) for minimal volume creation and most effective treatment possibilities targeting resource recovery thereafter, an efficient transport system requiring little costs and effort for establishing and maintenance, and treatment possibilities allowing to recover all substances with negligible emissions. Many elements of such a system are already developed; a small contribution is made by this thesis.

While the change towards the now established wastewater infrastructure was boosted by epidemics or in the case of Hamburg additionally by a huge conflagration in the year 1842 calling for new infrastructure on many different levels, we hopefully can achieve the next change without experiencing a disaster, or as *Londong (2008)* said “change without catastrophe”.

While the first solution’s theme was “flush it away”, the current one is “reuse, don't waste”.

A. Introduction

A.1. Motivation

The starting point of this thesis was the research project “Resource recovery and removal of pharmaceutical residues – Treatment of separately collected urine” within the research project of Kompetenzzentrum Wasser Berlin “Sanitation Concepts for Separate Treatment of Urine, Feces and Greywater “ (SCST). The aim was to investigate selected processes, mainly targeting ones with potential for an industrial style implementation, for the treatment of yellow water i.e. urine for resource recovery and the removal of pharmaceutical residues. The results of this part of the research project were documented in *Tettenborn et al.* (*2007*) and are further processed in this thesis.

While some questions targeting the treatment efficiency could be answered, new questions also arose. The concentrations of the investigated substrates varied from literature values and no satisfying data base was available for a realistic scenario calculation for an implementation of a urine separating system in the city of Hamburg, starting from a promising current situation with public urinals already available, where large amounts of urine are collected. Additionally the topics regarding collection potential and transport in this urban context were not sufficiently answered.

Therefore the missing links were worked on gaining an excellent data base for substrate quality from public urinals and draft results on the issue of transport. Additionally the research on the treatment options was continued, adding new information to the existing processes and their potential for implementation. Combining all of these results, a fictive case study was set up for the stepwise implementation of a urine separation system for a city like Hamburg with resource recovery on an industrial scale.

However, it has to be kept in mind after all that resource recovery from source separated urine is only one interesting and promising option out of many in the field of new sanitation systems, and should be considered as one part of a complete sanitation system.

Overall the research was the logical consequence of and was built on the work of *Otterpohl et al. (1997)*, *Behrendt et al. (2002)*, and *Niederste-Hollenberg (2003)*.

A.2. Aim and structure of this work

The urge and potential of reuse instead of waste elimination has reached the field of current wastewater management within the last two decades. New sanitation systems are often based on source separation to cope the challenge of resource recovery (*DWA 2008*). In this context of source separated wastewater streams, urine plays an important role because its comparably small volume and high nutrient concentrations allow for an efficient nutrient recovery.

Since the end of the 1990s several pilot projects have been established with a separate collection and usage of urine. Some in rural context with direct application of the collected urine in agriculture (*Bastian et al. 2005*; *Hanaeus et al. 1997*) others focusing on a subsequent treatment more on a decentralized level (*Bischof & Meuler 2004*; *Larsen & Gujer 1996*).

Within this work the relevant aspects for the realization of larger systems of separate urine collection with subsequent treatment on an industrial scale is investigated. The aspects range from collection potential over transport issues to selected treatment methods. Depending on the topic and the target questions of each individual aspect, a matching working procedure was applied. The results are merged in a concentrated form in this thesis.

The thesis is structured according to the logic order of the main elements of a urine separating system. For each element background, focus of investigation, working procedure, and results are discussed in an individual chapter. While the elements were investigated under different boundary conditions and in individual contexts, the results offer their individual potential and strengths, allowing to combine the gained knowledge and to find out about the general potential of one (out of many) urine separation system in an urban context.

The research work of investigation of the elements consisted in

- collection: survey of public urinals;
- transport: logistic model based on excel (scenario development according to available data for the city of Hamburg);
- treatment: design and operation of the treatment units, both in lab scale as well as in semi-technical scale as a pilot plant; and
- implementation of the gained results in a basic fictive case study to elucidate the potential of scaling up a system for separate urine collection and treatment.

In depth focus was applied on the investigation of selected treatment techniques of source separated urine for nutrient recovery and for the removal of pharmaceutical residues and on the survey of the current situation.

A.3. History and background

A.3.1. From the past to current sewer systems

Sanitation has been linked to human settlements throughout history. These signs of civilizations were discovered e.g. in the ruins of Eshnunna, an ancient Sumerian city in upper Mesopotamia, dated about 2500 B.C or even earlier in relicts from the Indus civilization (about 2550 B.C.) or the Minoan civilization (about 3400–1200 B.C.) (*Farnsworth Gray 1940*; *Foil et al. 1993*).

At least as old as these sanitation systems or most likely even older is the knowledge about the value of human excreta for different uses. Urine e.g. was used for washing and coloring garments, and tanning leather. For these purposes urine had to be collected separately. From the Roman Emperor Vespasian (Caesar Vespasianus Augustus 9–79AD) it is reported that he stabilized the financial situation of the roman empire amongst others by putting a tax on urine described in the well known description[1] of *Suetonius Tranquillus* (*2006*) from with the saying "pecunia non olet"[2] was probably derived. The urine was collected by the guild of fullers, the garment cleaners of Rome, using the ammonia in urine for cleaning purposes. The use of urine for tanning continued beyond medieval times. Next to the value of urine for handcraft processes, the fertilizing value of excreta was also well known. People would collect their 'night soil' which had a cash value as fertilizer for crops (*Gayman 1996*).

However, with the decline of the Roman Empire the knowledge about hygiene within the cities got lost leading to severe hygienic conditions and many epidemic outbreaks up to the middle of the 19th century when large sewer systems were started to be built to improve hygienic conditions within the cities (*Bechmann 1889*; *Gayman 1996*; *Grötz & Pelc 2008*; *Seeger 1999*). The main transportation process within these systems is based on gravity, requiring large amounts of flushing water (*Metcalf & Eddy 2003*). Nowadays, since the value of clean water becomes more and more obvious the procedure may be questioned.

A.3.2. Industrialization and the effects of the fertilizer industry

Nearly at the same time as the invention of modern sewer systems, chemists like Justus von Liebig started more and more to understand and describe the chemical fundamentals and complexity of fertilizing in agriculture (*Liebig 1840*; *1878*) and finally to develop synthetic fertilizers (*Liebig 1855*; *1859*) such as superphosphate which was until recently one of the most commonly used fertilizers, leading to an enormous increase of agricultural production between the end of the 19th and the beginning of the 20th century. While in the agricultural literature of the late 19th century the topic of mineral fertilizers was heavily and controversially discussed (*Liebig 1856*; *1865*; *Wolff 1856*; *Wolff 1858*) the value of urine for fertilizing purposes was still well known and often described, e.g. by *Fürst* (*1868*), *Neydeck* (*1854*), and *Wolff* (*1868*).

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[1] « Reprehendenti filio Tito, quod etiam urinae vectigal commentus esset, pecuniam ex prima pensione admovit ad nares, sciscitans num odore offenderetur; et illo negante: "Atqui," inquit, "ex lotio est." » Translation: « Titus complained of the tax which Vespasian had imposed on the contents of the city urinals. Vespasian handed him a coin which had been part of the first day's proceeds: "Does it smell bad?" he asked. And when Titus said "No" he went on: "Yet it comes from urine." » Source: *Suetonius Tranquillus* (*2006*)

[2] Money doesn't smell

However, the invention and the development of the Haber-Bosch process based on the findings of *Haber* (*1922*), *Mittasch & Frankenburger* (*1929*), *and Schlögl* (*2003*) a few decades later finally allowed the production of highly concentrated ammonia that could be further processed to highly potential fertilizers in large quantities These high concentrations were then economically viable to transport over long distances, which became necessary since distances from agricultural production fields and human settlements started to grow more and more apart during the increasing industrialization and urbanization. Because of its rapid enlargement, the Haber-Bosch process is seen as one of the exemplars of the chemical industry of the 20th century for world economy (*Travis 1993*). The yearly ammonia production by the Haber-Bosch process in large scale industry was meanwhile above 100 million tons with a total energy demand of about 5.4 EJ[3]. Nowadays average reactor capacities range from 1500–2000 tones ammonia per day (*Yara & IFA 2006*).

Large scale industrial processes and large sewer systems are normally built for time spans of at least one generation in which investment costs for construction should be amortized. The consequences are that nowadays not only is the water /wastewater management based on centralized structures, but the fertilizing industry is also based on large scale processes allowing the most cost and energy efficient production (*Lund et al. 2010*).

A.3.3. Need for alternatives

Phosphorous resources are not infinite (*Cordell et al. 2009*) and the production nitrogen fertilizers is highly depending on energy and thus energy prices.

At the same time, up until now two main options for nutrients contained in communal wastewater have been realized on a larger scale: the direct use of wastewater fractions (mainly sludge) in agriculture that has meanwhile ended in some European countries because of contamination of these fractions with heavy metals and other unwanted substances; and the elimination of the nutrients contained in the wastewater, which became necessary before releasing the treated wastewater to the aquatic environment to prevent eutrophication and the die off of surface water bodies. As a third and sustainable option it is now more and more discussed to extract and recover nutrients from wastewater in concentrated form for later use abroad.

Changing boundary conditions such as climate change, rapid urbanization, increasing world population, but on the other side decreasing population in some developed regions require new approaches and innovations regarding a more flexible water infrastructure which are contradicting conventional water infrastructure systems with very long service lives (*Hillenbrand & Hiessl 2006*). As alternative to large and inflexible centralized wastewater infrastructure systems techniques for decentralized structures are meanwhile well established and new operation models proved to be successful (*Hiessl et al. 2007*).

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[3] 1 EJ = 10^{18} J = 1.000.000.000.000.000.000 J

Since first approaches regarding innovations such as new sanitation systems (*DWA 2008*) are already made, *Londong et al.* (*2011*) see the changing boundary conditions as a chance for the implementation of new and innovative concepts.

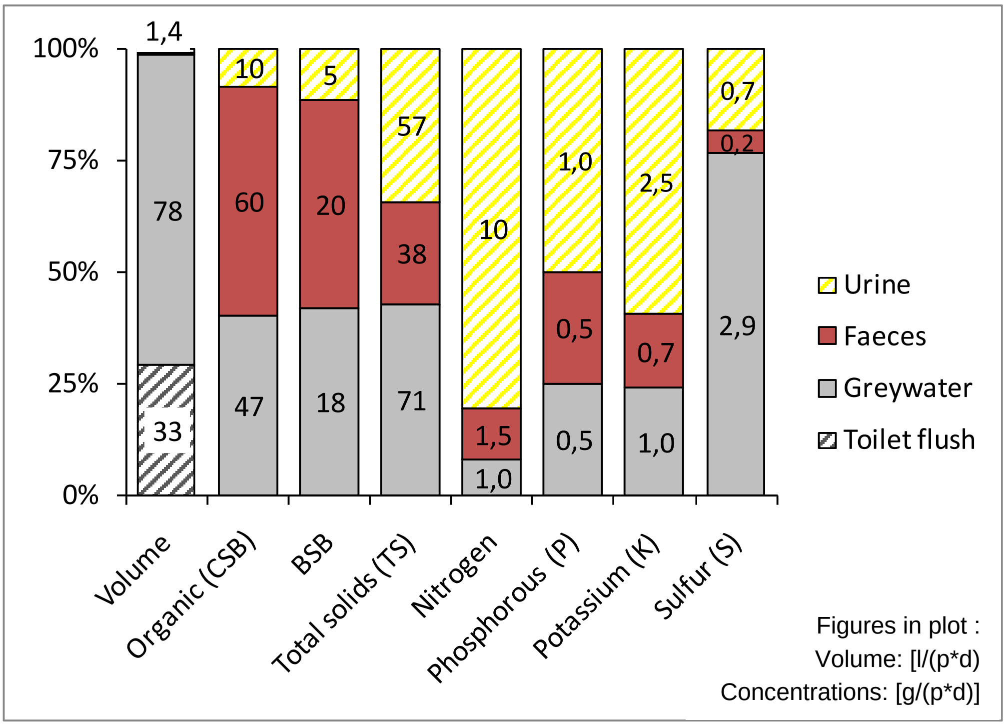
A.3.4. Alternatives with new sanitation approaches

In the middle of the 1970s the idea of a more resource oriented economy started to grow resulting in the first official initiative at EU-level, the council directive of 15 July 1975 on waste (*NORM-EU 1975*) claiming the prevention and the reduction of waste generation and its hazardousness, and the recycling/reuse of waste products. Subsequent directives in Germany were the Kreislaufwirtschafts- und Abfallgesetz (*NORM 1994*) claiming that hierarchy prevention comes before recycling which comes before elimination. This guiding principle was also implemented in more and more industrial processes bringing a new definition of values. According to (*DWA 2008*) in wastewater forming industries these were:

- multiple reuse of non preventable process- and washing waters
- closing loops
- substance recycling
- recovery

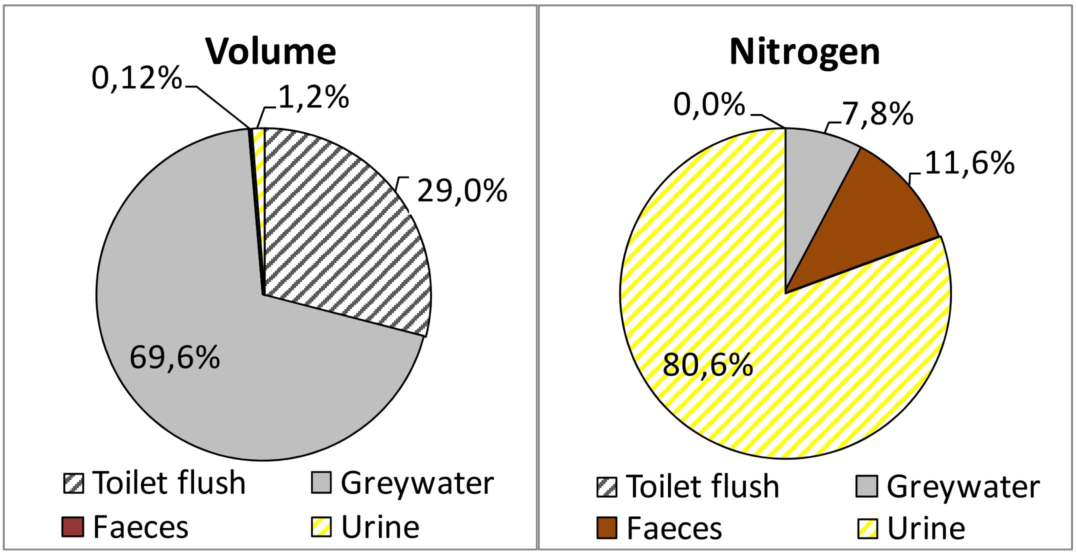
At the same time source control alternatives to the large and inflexible wastewater infrastructures with high resource demand were developed firstly with emphasis on sanitation without water (*Winblad & Kilama 1985*). The idea of source control and reuse was further developed by different groups mainly in Sweden (*Hanaeus et al. 1997*; *Jeffrey et al. 1997*), Switzerland (*Larsen & Gujer 1996*), and Germany (*Lange & Otterpohl 2000*; *Otterpohl et al. 1997*; *Otterpohl et al. 2003*). These concepts were based on the collection of separated wastewater streams as blackwater (toilet wastewater) and greywater (wastewater from sinks, showers, and washing machines) directly at the source (*Otterpohl 2002*). Further separation leads to the collection of urine respectively yellowwater (urine with flush water) and brownwater (feces with flush water) respectively feces. Consequently the fraction containing mainly the organics and most of the pathogens is separated from the nutrient rich but rather harmless solution (*Höglund 2001*; *Otterpohl 2001*).

The main fraction of nutrients in communal wastewater stem from human urine (*DWA 2008*; *Fittschen & Hahn 1998*). About 80 % of nitrogen, about 50 % of the total phosphorous load, and about 60 % of potassium of communal wastewater are due to the small volumetric fraction of 1.4 % of the total wastewater volume contributed by urine (*Ciba Geigy AG 1977*; *Larsen & Gujer 1996*; *Otterpohl 2002*) (see Figure 1). Because of the relatively high nutrient content, urine is still considered an excellent liquid plant fertiliser (*Ganrot et al. 2007b*; *Muskolus 2008*; *Simons & Clemens 2003*; *Vinnerås & Jönsson 2002*). The nutrients are well available to plants due to their occurrence as soluble ions (nitrogen as ammonium and nitrate; phosphorus mainly as ortho-phosphate ions; potassium ions; and sulphur as sulphate ions (*Schönning 2006*)).



Data from DWA (*2008*)

Figure 1: Volume and concentrations per person and day of the different source separated wastewater streams in Germany



Data from DWA (*2008*)

Figure 2: Fractions in household wastewater

In Figure 2 the relevance of a specific view on urine is pointed out with the example of nitrogen. In about 1.2 % of the total wastewater volume, about 80 % of the total nitrogen load are contained (concentrations of about 7 g N/l). On the other side as e.g. greywater is a highly diluted stream (in 70 % of the volume are e.g. about 8 % of nitrogen contained), in witch concentrations with about15 mg N/l (*DWA 2008*) are rather low (*Otterpohl et al. 1999*).

Knowing about the high nutrient fraction and the low volumetric fraction both compared to other wastewater streams, the abovementioned third option regarding the recovery of nutrients in wastewater can partially be realized within new sanitation concepts by urine separation and its separate use or treatment.

A.3.5. Obstacles and requirements of new sanitation systems

However, nutrient concentrations in urine are low compared to mineral fertilizers. Since current world food cultivation is located far away from the consumers and from fertilizer productions (*Muirheid 2010*), processes that concentrate nutrients are needed to allow an application where needed.

When applying liquid organic fertilizers from separation systems such as stored urine directly to agricultural fields, increased emission of ammonia can lead to increased acidification (+ 30-110 %) and eutrophication of terrestrial ecosystems as *Remy* (*2010*) has reported.

Additionally, in recent years more and more problematic substances such as pharmaceutical residues were detected in surface, ground, and drinking water (*Daughton 2001*). The main reason is the low ability of nowadays wastewater treatment to work as a barrier against these micropollutants (*Niederste-Hollenberg 2003*). At the same time a large fraction can be contained in urine. Around 70 % of pharmaceuticals are excreted via urine accounting for 50 % of the overall ecotoxicological risk (*Lienert et al. 2007a*; *Lienert et al. 2007b*). *Winker* (*2009*) did an in depth evaluation of literature regarding the potential risk of the usage of urine as fertilizer in agriculture. From these and from pot experiments it was found that some pharmaceuticals were detected in high amounts (~50 %) in the soil after a period of three months, significant amounts were detected in aerial plant parts (30 %), and traces in roots (0.2 % of applied amount) (*Winker 2009*).

While “plant matter production was not affected by the applied pharmaceutical concentrations” (*Winker 2009*), insufficient knowledge exists regarding pharmaceuticals ingested by humans through crops. Hence, if urine should be used as fertiliser, treatment methods have to be applied for an efficient removal of these substances to achieve systematic cleaning (*Larsen et al. 2004*).

A.4. Consequences for urine separating systems

Urine separation is one option to deal with the nutrient load in communal wastewater, to cope with the nutrient demand in agriculture, and to reduce a fraction of certain micropollutants.

Urine separation is already realized to some extent by the usage of urinals. Often waterless urinals are implemented for saving flushing water. In some cases the substrate is even collected separately in collection tanks instead of being directed into the sewer system.

A.4.1. Aims of treatment of source separated urine

The motivation and the main objectives aimed for by urine separation systems with subsequent treatment of the collected urine are[4]:

- removal of a larger nutrient fraction from household wastewater
    - nutrient reduction in wastewater as a first action to reduce nutrient emissions resulting in eutrophication and dead zones around densely populated regions with insufficient wastewater management
    - nutrient reduction at the wastewater treatment plant with the consequence of reduced energy demand and reduced required area
    - improved anaerobic treatment in new sanitation concepts since no subsequent nutrient removal would be required;
- nutrient recovery for use of nutrients in agriculture and/or industry
    - improved nutrient recovery
    - volume reduction (transport/storage)
    - production of fertilizing goods
    - production of market goods; and
- reduction of micropollutants (pharmaceutical residues)

Nutrient reduction in wastewater as a first action to reduce dead zones in densely populated costal regions with insufficient wastewater treatment

Larsen et al. (*2009*) state: “source separation could lead to sustainable solutions more directly than traditional approaches”. Especially in areas with so far insufficient wastewater management, new sanitation systems based on source separation seem to be a very promising solution. The implementation of source separation with reuse has been proven in rural areas in different pilot projects (*Blume 2009*; *Deegener et al. 2005*; *Holmer & Itchon 2008*). However, the need for sustainable wastewater management solutions in more densely populated areas is not lower.

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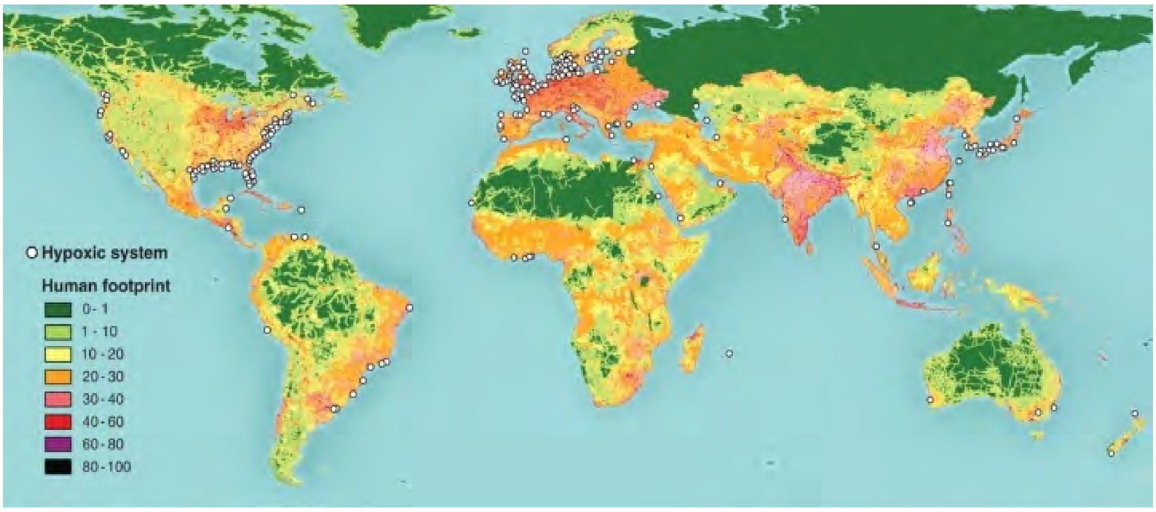
[4] based on a compilation of *Peter- Fröhlich* (*2010*)

Especially in densely populated coastal areas with insufficient wastewater treatment, nutrient loads from wastewater are leading to eutrophication and thus contributing to the increasing phenomenon of dead zones (consequence of hypoxic areas) (*Diaz & Rosenberg 2008*). A distribution of hypoxic systems leading potentially to dead is presented in Figure 3. The zones are relating to densely populated areas e.g. large cities in coastal regions. These dead zones become more and more a threat to fishing, which often is of major importance to the regional economy.

As a first step to protect the costal zones from eutrophication and hypoxic "dead zones", *Larsen et al.* (*2007*) are calling urine source separation the "technology of choice", allowing a nitrogen separation of theoretically up to 80 %, more than the average nitrogen removal by most denitrifying processes on treatment plants.



Source: Diaz & Rosenberg (2008)

Figure 3: Global distribution of scientifically reported dead zones (hypoxic systems).

Nutrient reduction at conventional wastewater treatment plants

Wastewater treatment plants are normally amongst the largest communal energy consumers. Within common wastewater treatments plants, aeration steps for the activated sludge treatment for degradation of organic material and especially for nutrient removal are dominating the energy demand. By removing human urine from the rest of the household wastewater, nutrient elimination at a wastewater treatment plant could be reduced by about 80 % for the nitrogen elimination, and by about 50 % for the phosphorous removal. (*Larsen & Udert 1999*) stated for this situation a C:N:P ratio of 75:5:1 which would be ideal for activated sludge treatment (*NORM 2000*). Consequently energy and space for nitrification and denitrification steps could be spared.

Niederste-Hollenberg (*2003*) and *Wilsenach & Loosdrecht* (*2003*) calculated for different scenarios that a collection of 60 % of urine would be sufficient to allow for an activated sludge process without additional nitrification and denitrification step to reach the required effluent limits.

Maurer et al. (*2003*) calculated the energy demand for denitrification at a wastewater treatment plant with 45 MJ/kg nitrogen and 49 MJ/kg phosphorous for P-precipitation. Accordingly about 60–80 % of the total energy demand for the nitrification process could be saved when collecting urine separately.

For enhancing the nitrogen elimination from wastewater *Sackewitz & Maier* (*1999*) described an air stripping process of sludge liquor containing up to 25 % of the daily nitrogen load of the waste water treatment plant. The process was conducted at plants in Göttingen and Cuxhaven, Germany. Ammonia concentrations of the treated wastewater stream were between 0.8 and 1.2 g NH_4-N l^{-1}, thus below the concentrations of separate collected urine. According to *Sackewitz & Maier* (*1999*) the process is more economically efficient the higher the concentrations in the treated stream with an ideally high pH-value.

Improved anaerobic treatment (no subsequent nutrient removal required)

While anaerobic treatment, because of its positive energy balance and its relatively small space requirements, is meanwhile an often favoured option for wastewater treatment in new sanitation concepts (*Hillenbrand 2009*; *Mohr 2005*), nutrient loads of the wastewater require additional treatment steps.

By combining these systems with urine separation efforts, for a subsequent treatment of anaerobic digested wastewater could be reduced tremendously and realized e.g. by simple polishing steps. *Wriege-Bechtold et al.* (*2010*) reported positive results from mesophilic digestion tests of brownwater.

Improved nutrient recovery

For processing a small but concentrated volume rather than a large volume with lower concentrations, smaller treatment units can be used and less energy per recovered substance should be needed. Nutrient concentrations in human urine are roughly two log units higher than in communal wastewater, the volume makes up 1–2 % compared to communal wastewater.

However, treatment efficiency often increases with plant size. Therefore at the moment the advantage might be on the side of larger treatment steps implemented in conventional wastewater treatment.

Nutrient recovery for usage in agriculture

According to *Clemens et al.* (*2008*) about 17–25 % of the demand of the main relevant nutrients nitrogen, potassium, and phosphorous in agriculture could be replaced in Germany by the use of nutrients contained in urine.

The nutrient content of the urine is similar to that of crops harvested from the fields, thus equivalent to the amount of nutrients required for recharging the nutrient content of the soil. The amount of nutrients from one person is sufficient to fertilize an area of about 200–400 m^2 (*Jönsson et al. 2004*).

However, densely populated areas with a large potential for urine collection and agricultural areas with a large demand in nutrients are in a globalized world mostly far apart from each other. Thus any concentration of the nutrients contained in urine will reduce transportation costs.

By supplementing mineral fertilizer, significant amounts of energy could be saved. *Maurer et al.* (*2003*) calculated the energy demand for production of N-fertilizer to be 45 MJ/kg nitrogen and 29 MJ/kg phosphorous.

Nutrient recovery for usage in industry

Besides the use of products gained from urine treatment in agriculture, there might be a potential for products from urine treatment in industry. Many industrial processes are based on nitrogen or phosphorous resources. In the combustion industry for example ammonia or urea are used in flue gas treatment to reach limits of *BIMSCHV* (*NORM 1990*). Especially in combustion processes with temperatures above 650 °C where nitrogen monoxide is formed (*Schultes 1996*), dry treatment processes based on SNCR-/ SCR have to be applied. In these processes ammonia (NH_3) or urea ($CO(NH_2)_2$) is injected into the flue gas to reduce nitrogen oxides to molecular nitrogen (N_2). In the automobile industry the effect of NO_x-reduction is as well applied as in combustion industries. AdBlue® is a product of BASF for a SCR-process in automobiles. The procedure is the same as in flue gas treatment (*BASF 2008*). Purity requirements are described in *DIN 70070* (*NORM 2005*).

Urea used to be used for defrosting airfields and airplanes. However, this seems to be uncommon nowadays (*Wille et al. 1998*). Further, urea is used in the pharmaceutical or cosmetic industry as an antifungal agent and as a moisture carrier. However, high purity is needed.

Pure phosphorous is used in the plastics industry, for producing detergents or in pesticides. For industrial production of pure phosphorous, mainly apatite rocks such as hydroxyapatite (HAP) are used. Because of existing treatment infrastructures and the large quantities of treated material, the phosphorous industry is mainly interested in resources similar to phosphorous rocks. Phosphorous from urine e.g. in the form of struvite could become interesting due to technological innovations and due to decreasing phosphate resources (*Udert 2002*).

The idea of using wastewater as a nitrogen source for a pure ammonia product is not a new one. *Nitsche & Nitsche* (*1995*) suggested recovering 25 % ammonia water from steam stripping of communal wastewater instead of eliminating the nitrogen in the wastewater treatment plant.

At the Höchst industrial park, urea containing wastewater is concentrated in a two stage evaporation process and used directly for NO_x-removal in the flue gas treatment of the adjacent sewage sludge combustion plant (*Steyer 2005*). The heat energy of the sewage sludge combustion plant is used for the production of additives for flue gas treatment.

A.4.2. Frame, boundary conditions within the present thesis

Any urine separation system needs to be set in a context of a complete wastewater infrastructure in which also the other wastewater streams are dealt with. This could either be: a conventional wastewater infrastructure with sewers and centralized treatment plants with reduced need for nutrient removal; a complete new wastewater infrastructure based on separate treatment and collection of each, brownwater and greywater; or a combined system of areas with conventional systems and areas with new sanitation systems. Many options can be thought of. Focus of this thesis is on the module urine collection, transport, and treatment, only.

A.4.3. Focus questions of investigation

With the above mentioned considerations in mind, the following questions were investigated in the present thesis:

- general system description including collection, transport, treatment and possible whereabouts of compounded products;
- discussion of substrate and its nutrient content in a given context (public urinals in Hamburg);
- deriving a solution for collection and transport and discussing necessary aspects of logistics behind these systems (model of a truck-based transport system in Hamburg); and
- finding and designing options for industrial style treatment processes valid for urine treatment. Finding operation and yield parameters of these selected treatment options for recovery of nutrients and removal of micropollutants (partially conducted in pilot sale).

Especially the field of medium to large scale industrial style processes for urine treatment based on semi-centralized systems has not been evaluated sufficiently so far.

B. Collection: public urinals – substrate quality and characteristics

B.1. Introduction

Concentrations of urine vary from person to person and throughout the day (*Ciba Geigy AG 1977*) due to personal metabolism, habits, disease, and/or metabolic status. For xample morning urine is said to be higher in concentration than the over all day average. Consequently the location of collection will influence concentrations. Collection at home (in private households) should yield higher concentrated urine, collection at schools and office buildings (work place) would lead to lower concentrations depending mainly on habits regarding quantity of liquid intake. It can be assumed that concentrations of urine collected in public locations would also mainly depend on user habits.

For collection of human urine, separation toilets or urinals can be used. Both exist as flushing or non-flushing, waterless versions. While separation toilets are implemented so far only in several pilot projects, waterless urinals for males are well established (*Ruchti et al. 2005*). A good overview of different types can be found in *DWA* (*2008*).

Especially in public toilets for males, all types of urinals are common in both versions, flushing and waterless. The driver for waterless systems is mostly of economical nature because of water savings.

In new sanitation concepts, where one of the main aims of collecting and treating urine yields towards nutrient recovery, dilution effects are unwanted. Thus focus is laid here on non-flushing or low-flushing systems.

Separation toilets can be found in many pilot projects e.g. in Germany: Hans Huber AG, Berching (*Bischof & Meuler 2004*); SCST Stahnsdorf, Berlin (*Peter-Fröhlich et al. 2004*); GTZ main office building, Eschborn, Frankfurt (*Ulrich & Olt 2009*); in Austria: Solarsiedlung Linz (*Hochedlinger et al. 2008*; *Steinmüller 2006*); in Switzerland: Forum Chriesbach eawag, Dübendorf (*Larsen & Lienert 2007*; *Larsen et al. 2001*); in Sweden: (*Hellström & Johansson 1999*); and in the Netherlands: STOWA[5] (*Wilsenach 2005*). Amongst others separation efficiency and occurring problems were investigated. These results and observations from two separation systems in Germany were used in this work.

While urinals for women are not common, a few are available. *Möllring* (*2008*) gives a good overview of different types in public areas and a detailed description about history and boundary conditions. Most of the urinals for women are operated with large flushing volumes. Waterless urinals for women especially for public areas are not common.

Urinals for males are installed mainly in public places and non-private ('community') lavatories, e.g. in office-buildings, schools, universities, restaurants, shopping centers, hotels,

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[5] http://themas.stowa.nl/themas/projects

airports, etc. In many cases waterless urinals are used for environmental issues (water saving) and economic reasons (savings on the water bill). While in most cases, the urine is not collected separately but discharged into the conventional sewerage system in Hamburg, experiences with the operation of public urinals equipped with collecting tanks were gained as described by *Grönwall* (*2005*). For the present thesis user numbers were observed as well as nutrient concentrations analyzed by *Goldammer* (*2011*).

B.2. Focus of investigation

To get detailed information on the potential of urine separation in a city like Hamburg, the current status of a larger urine collection system was observed.

User frequency of public toilets, collectable material per time, and characteristics of collected substrate from public toilets were investigated.

B.3. Material and methods

B.3.1. Study site Hamburg

According to *(Grönwall 2008)*, Hamburg is one of the few cities in Germany with a sustainable concept approach for public toilets. The aim was to reduce costs for the city by increased quality of public toilets. The locations are areas with high traffic volume, places of interest for tourists, and social hot spots. As far as possible, handicapped accessibility was realized. Next to about 170 toilets, twelve waterless urinals with a collection tank are installed (*Grönwall 2005*; *Winker & Grönwall 2010*). The distribution of urinals with a collection tank over Hamburg is shown in Figure 4 and a detailed description of the locations and surrounding conditions is given in Appendix table 1.

From the twelve public urinals nine (L1-L9) were investigated more thoroughly (Table 1). Filling level was measured over a period of three months from January 2009 until March 2009. To get an estimation of maximum loads, user censuses were conducted.

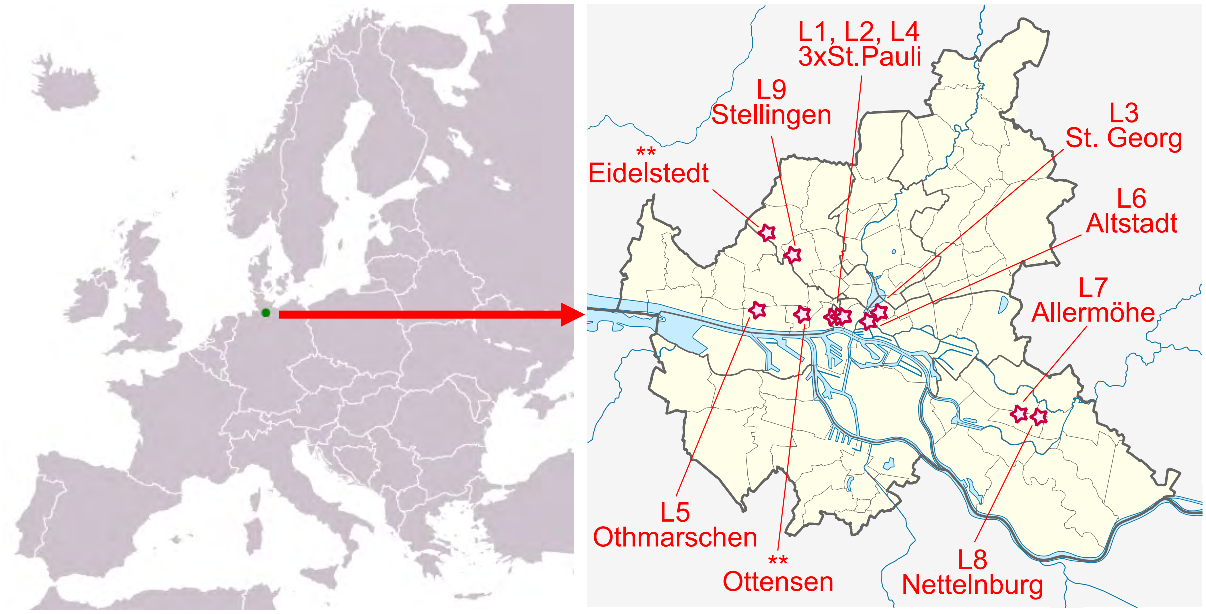
Over the period of three months, samples were taken two to three times a week from nine locations. Additionally, one serviced public urinal was investigated (L6). The samples were analyzed in respect of N, P, pH, and conductivity. Additionally total carbon, potassium, calcium, sodium, sulfate-, and chloride ions were measured in grab samples.

In addition to the waterless urinals in Hamburg, samples of the SCST demonstration site Stahnsdorf were analyzed for the contamination of pharmaceutical residues. A site description and main parameters are given in the appendix in chapter I.1.1.

Table 1: Name of the location of the urinals and abbreviation for the following discussion

Abbreviation	Location name of the public urinal
L1	Beim Trichter, St. Pauli
L2	Hamburger Berg, St. Pauuli
L3	Hansaplatz, St. Georg
L4	Reeperbahn No.7, St. Pauli
L5	S-Bahn station Othmarschen
L6	Rotunde, Central station, Altstadt
L7	S- Bahn station Allermöhe
L8	S- Bahn station Nettelnburg
L9	S- Bahn station Stellingen
**	Eidelstedt; Ottensen; St. Pauli

** not further investigated



Based on maps from NordNordWest andFoundert, Wikimedia Commons

Figure 4: Locations of public waterless urinals with collection tank, in Hamburg (for name and comments see Appendix table 1)

B.3.2. Technical set-up of urinals

The design of the urinals is displayed in Figure 5. The urinals are composed of a special marble powder mixture, and the drain is fitted with membrane odor traps. The effluent of the urinals is fitted with a slider and can be drained off into underground tanks made of a sandwich construction with glass fiber reinforced plastic and polymer-concrete layers, or for daily cleaning purposes into a nearby sewer system in witch the floor drain and an overflow of the collection tanks is directed as well.

Three different collection systems respectively tanks are applied:

- longish tank with unsealed cover (first urinal L3) with a total volume of 2 m^3;

- globular tanks with a total collection volume of 2.7 m^3 used at seven of the investigated nine locations; and
- collection system at serviced public toilet, where waterless urinals of the male restrooms are connected to a collection tank with a total volume of 640 liters (L6).

The longish tank was the first collection tank, put in place in 2003. The next collection tanks were ball-shaped modified oil tanks with two layers of glass fiber reinforced plastic (Figure 6). The one serviced pubic urinal has an individually made collection system.

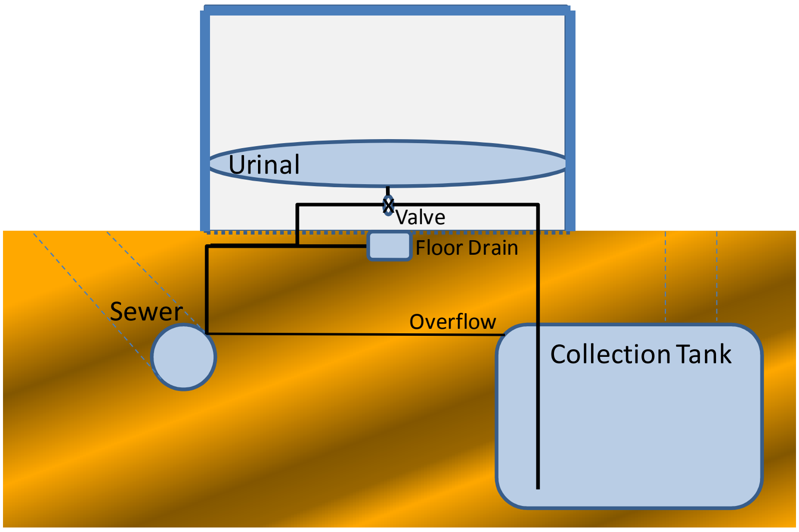


Figure 5: Scheme of urinal according to *Grönwall* (*2008*)

source: *Grönwall* (*2008*)

Figure 6: Modified oil tanks made from glass fiber reinforced plastic of Haase GFK-Technik GmbH

B.3.3. Measuring of urine quantities over time

Over the period of nearly three months (21.01.2009 to 30.03.2009) eight of the eleven urinals and one public toilet with on-site cleaning staff were sampled on a regular frequency between twice a week and every two weeks depending on their location and amount of usage. Six of the nine tanks were emptied before start of the investigation. Three had a filling volume of 600 to 900 liters corresponding to 1/4 to 1/3 of the maximum volume. The collection tanks were not emptied during the sampling period. Two of them were full after 17 days, two were not filled during the sampling period, although these were the ones that were not emptied before sampling (for details see Appendix table 1).

The quantity was detected by measuring the filling level at each location with subsequent calculation of the average daily inflow based on a rating table.

While some tanks were fitted with a level indicator, a plumb line was used in the other cases.

The accuracy was based on measuring method and tank geometry in a range of about 2 % of the tank volume.

B.3.4. Usage census

At the 30.01.2009 a census was conducted at one location (L9). At the time of the census a soccer game and a rock concert took place. The observation location was a van parked nearby to the urinal. The counting was conducted with manual counters over the period of 7 hours.

B.3.5. Sampling

A total of 88 samples were taken over the period of three months from nine different locations. 500 ml samples were taken with a sampling cylinder and a pumping device from the homogenized substrate. Because of tank geometry and filling heights from very low to completely full, homogenization might have been insufficient and sampling depth varied. Samples were taking during the regular operation of the urinals. Therefore, the samples consisted of a mixture of stored urine and fresh urine.

While the valve was supposed to exclude detergents from entering the collection tanks, it occurred that that valve was not closed before cleaning. At the same time it happened at other locations, that the valve was not opened after cleaning, so losses of urine into the sewer could happen. A dilution of the content of the collection tank by high ground water or entering surface water seems to be unlikely but cannot be excluded.

B.3.6. Analytics

Main parameters

The samples were analyzed in respect to pH, conductivity, ammonium/ammonia nitrogen expressed as NH_4-N and phosphate expressed as PO_4-P. A detailed characterization of the analytical procedure can be found in the appendix in chapter I.1.2.

Further analytics

Additionally 21 samples and three sediment samples were analyzed for total carbon, potassium, calcium, sodium, sulfate-, and chloride ions. Details can be found in the appendix in chapter I.1.2.

Pathogens

A total of twelve samples were tested regarding different biological parameters. Ten of these samples were tested directly after sampling, and two were tested after a storage time of two months. For the testing regarding microbiological parameters Merckoplate Brolacin-Agar (Bromthymolblau-Lactose-Cystin-Agar) (*Merck 2003*), and Chromocult Coliform Agar (*Merck 2009*) were used as ready-made nutrient solutions for detection of the following bacteria:

- Brolacin-Agar
    - Escherichia coli, Citrobacter u.a
    - Enterobacter, Klebsiella u.a.
    - Proteus, Serratia
    - Pseudomonas
    - Streptococcus agalactiae
    - Staphylococcus aureus, Staphylococcus epidermidis
- Chromocult Coliform Agar
    - Escherichia coli
    - Citrobacter freundii
    - Salmonella enteritidis
    - Enterococcus faecalis

In each case 500 µg were applied on each culture medium. The petri dishes were left for breeding in an incubator for 24 h at 36.5 °C. After counting with use of a magnifier and a 1 cm^2 grid, the counted numbers were converted to colony forming units.

Pharmaceutical residues

During the testing period in winter 2009 no samples were tested for micropollutants. However, during the SCST-Project (Peter-Fröhlich et al. 2007a) extensive testing for 21 pharmaceuticals in urine was conducted, three times at the location L3, and three times at the separation system of BWB Stahnsdorf, between March 2005 and Mai 2006. Levels of detection in the urine matrix are given in the appendix in Appendix table 2.

All analytics regarding pharmaceutical residues were conducted by IWW (Rheinischwestfälisches Institut für Wasser Beratungs- und Entwicklungsgesellschaft mbH) using HPLCMS (Butzen et al. 2005).

Statistics

Runaway values were detected according to *Grubbs (1969)* and *NORM* (*1985*) and removed if runaway was proven.

B.4. Results

B.4.1. Volume quantities and user frequency

Over three months from January to March the average daily increase of 9 investigated locations varied depending on each location between 26 and 128 l/d, resulting in an average of 1.9 m^3 per month and urinal location (see Table 2 and Appendix table 1).

At two locations (L2, L6) the collection tank was filled to its maximum within 17 days. Two other locations (L1, L2) were filled within 30–40 days, and two locations (L4, L5) were filled within nearly 60 days. One location (L7) did not reach its maximum level during the sampling period of three months. At one location (L8) a 1 m^3 sample was taken on day 14, thus the maximum level was also not reached any more during the sampling period. At the location L9 the collection tank was filled within 70 days. Filling levels over time are given in Figure 7.

Since the time frame of the investigations was spread over the three winter months, it can be assumed that the average amounts per month and per location over the year will be much higher possibly in a range of 25–35 % higher which would mean an average of 2.4–2.6 m^3 per month and location.

Table 2: Substrate volume increase per time of investigated urinals during sampling period

Location	Average [l d^{-1}]	Median [l d^{-1}]	Min [l d^{-1}]	Max [l d^{-1}]
L1	94	87	33	203
L2	71	54	36	165
L3	128	109	92	203
L4	46	42	29	78
L5	36	34	15	59
L6	27	32	15	35
L7	29	29	22	35
L8	26	21	11	44
L9	67	41	15	169

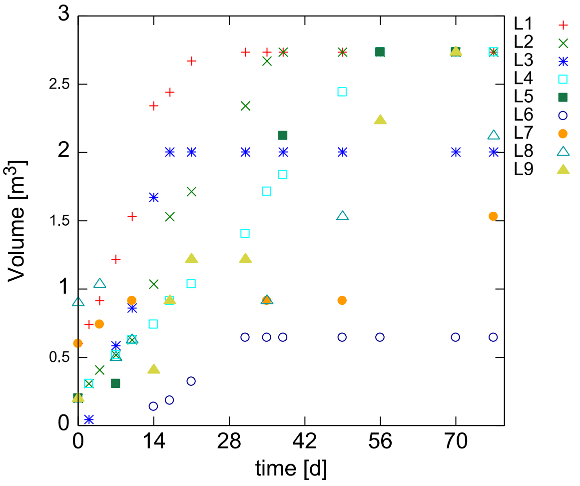


Figure 7: Filling levels of the nine locations. The collection tanks were not emptied during the sampling period except one exception (L9).

During the sampling period a user census was conducted. A total of 475 people were counted. The number of users varied between 5 and 140 per hour. The volume difference within the tank from the beginning of the census until the end of the census was about 150 l resulting in an average ‘contribution’ of 0.32 liters per person.

During the starting period of the sampling, the slider for cleaning purposes of five of nine locations was open, directing the urine into the sewer instead of the collection tank. After contact with the maintenance staff, this did not happen any more. However, it is possible, that the slider was thereafter not opened any more, not even for cleaning purposes, which would result in water and detergents entering the collection tanks.

B.4.2. Substrate quality

Main parameters: N, P, pH, conductivity

At each location a total between 6 and 14 samples were drawn over the sampling period. To allow a distribution-comparison of different parameters the average concentrations of parameter X of each individual location was divided by the overall average of all locations (X_{oa}) of this parameter resulting in a normalized concentration of parameter X (Eq 1)

Eq 1

$$X_n = {X_{ia}}/{X_{oa}}$$

X_n: normalized concentration of parameter X

X_{ia}: average concentration of parameter X of the individual location ‘i’

X_{oa}: overall average concentration of parameter X of all locations

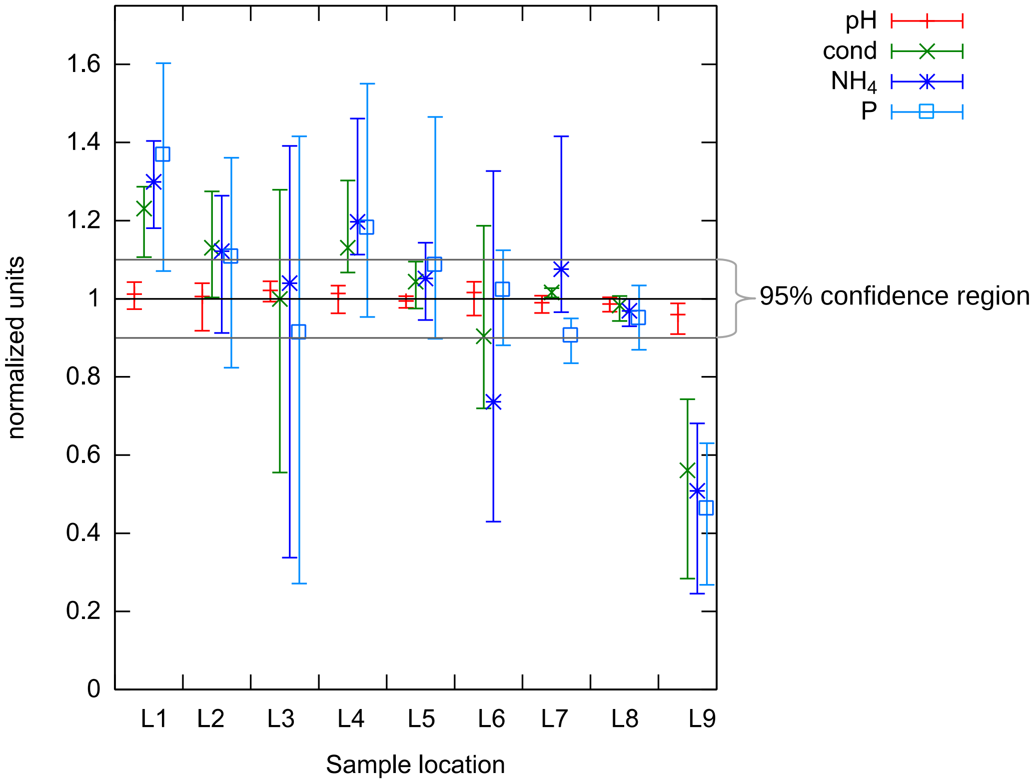


Figure 8: Overview of the normalized main parameters of the individual location with dispersion bar representing the minimum and maximum values

Values greater than 1 represent a higher location average of parameter X than the overall average, values smaller than 1 represent a smaller local average than the overall average. The normalized values (X_n) are presented in Figure 8 together with a dispersion bar representing the minimum and maximum values of each individual location. Detailed plots of pH, conductivity, ammonia concentrations, and phosphorous concentrations over the sampling period are given in Figure 9 to Figure 12.

During the filling process of the collection tanks the low concentrations especially of ammonia increased at the locations with low values in the beginning, resulting in a large range of dispersion. The low ammonium concentrations of two locations (L6, L9) especially in the beginning of the sampling period could be due to several reasons:

In some of the emptied tanks at the beginning of the sampling period it could be that the freshly contributed urine was not completely hydrolyzed. Lower pH values of two locations could be an indicator for this (Figure 9). This would result in lower NH_4-N values.

Low substrate volumes within the collection tanks in the beginning would result in a large interface area to volume ratio which could enhance ammonia losses due to outgassing (evaporation). The effect of nitrogen losses due to outgassing in collection systems should normally be negligible (*Udert 2002*). However, if air exchange can happen, losses can be higher. While the collection tank at L3 was filled within a short period during the sampling and the effect of low concentrations decreased, the effect of low NH_4-N concentrations remained at one location (L9) and disappeared only very slowly at another location (L6). Low values of L9 could be explained by a dilution effect, since also P-concentrations were very low.

Also dilution effects could have occurred having larger effects in emptier tanks.

For calculating a mean value for ammonia concentrations in the collection tanks, the two locations with abnormally low NH_4-N concentrations were excluded. Thus, a median for the ammonia concentration of 3.76 g/l was calculated from the remaining 72 values (Figure 10) with a 95 % confidence interval of 3.63 to 3.91 g/l.

Similar to low ammonia concentrations at location L9, the values for phosphorous concentration at this location (L9) were also significantly lower than the others (Figure 11). The low conductivity of this location (Figure 12) would support the theory of dilution effects. These dilution effects could either be due to users habits, since the location is highly frequented during special events such as soccer games or rock concerts in the nearby two arenas or by external infiltration of water.

When excluding this location, a median of 280 mg/l of phosphorous can be calculated from the remaining 74 values with a 95 % confidence interval of 265 to 294 mg/l.

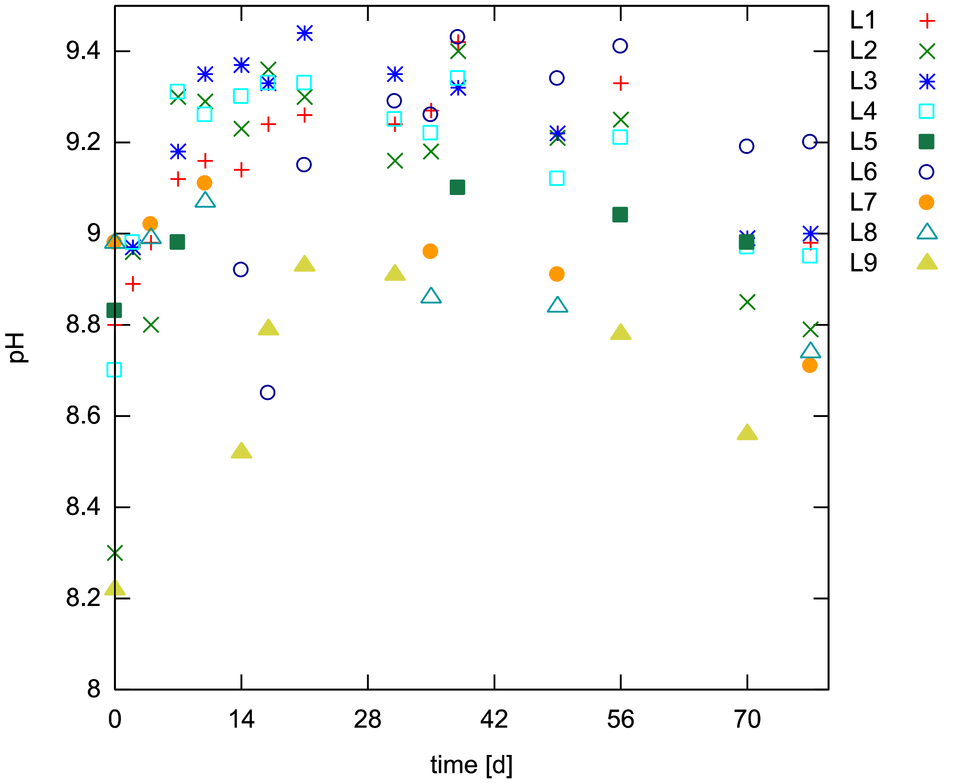


Figure 9: pH values in the small range between pH 8 and pH 9.5 over the sampling period

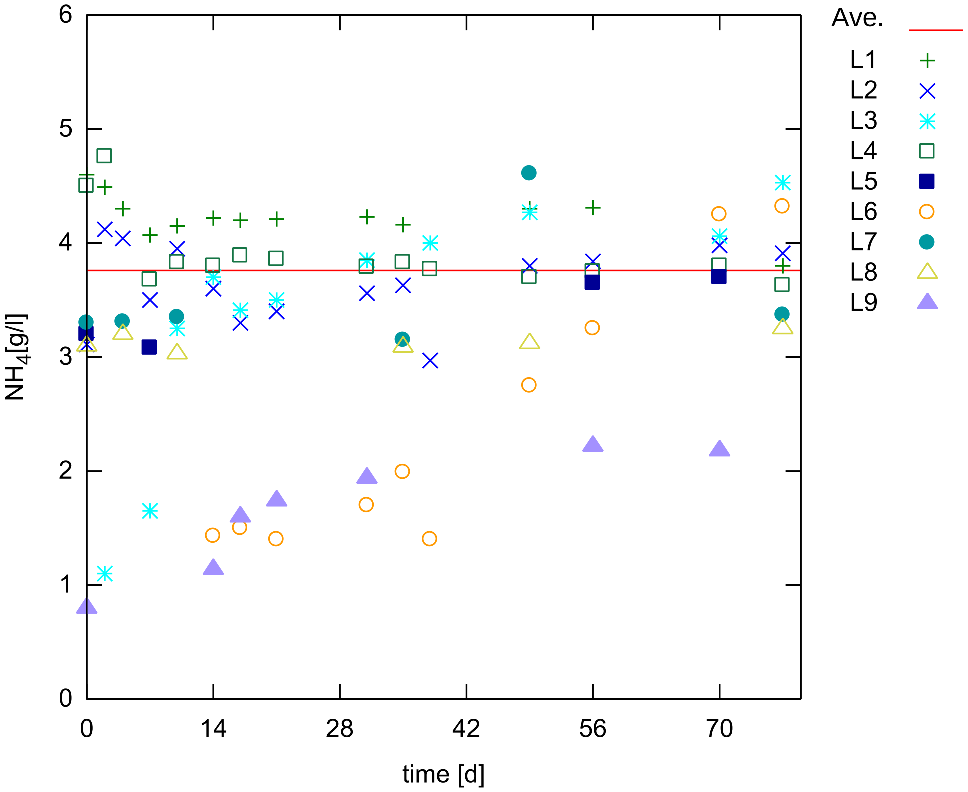


Figure 10: Ammonia concentrations in collection tanks at nine different locations during the sampling period of three months. Average (Ave.) = 3.76 g/l

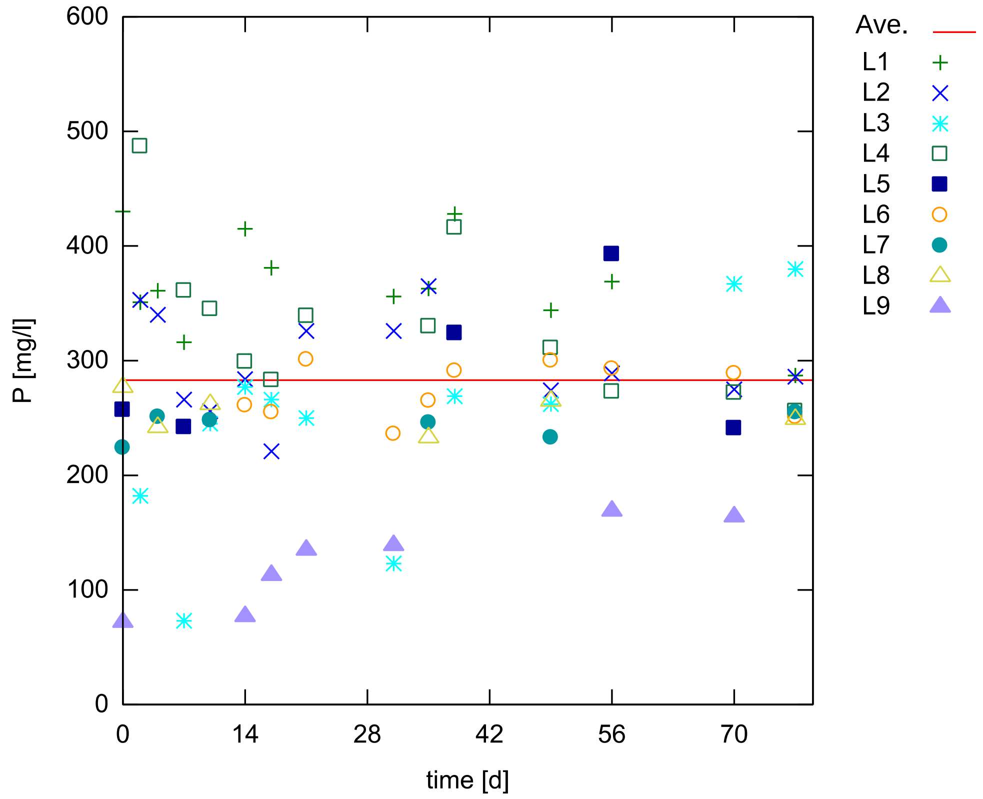


Figure 11: Phosphorous concentrations in collection tanks at nine different locations during the sampling period of three months. Average (Ave.) = 283 mg/l

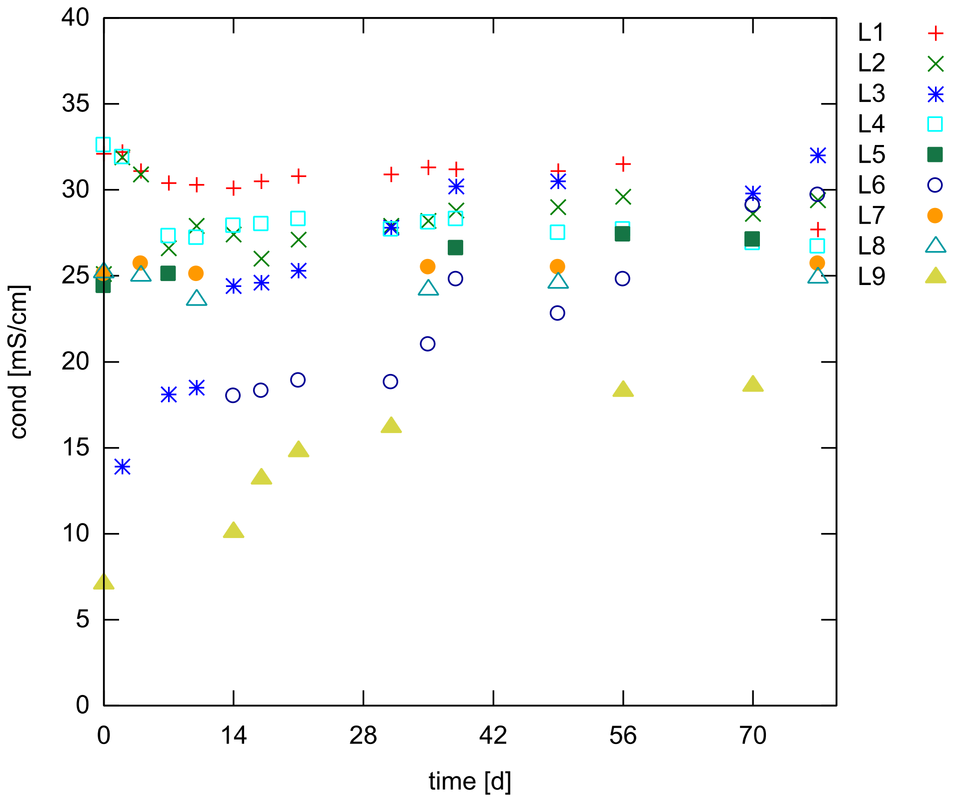


Figure 12: Conductivity of urine samples of nine public urinals during a three month sampling period

Since conductivity of a fluid is an expression of its ion concentration, correlations exist between the dominating ion concentrations and its conductivity. At the same time concentration ratios of different components are usually in a similar range. Therefore conductivity is an excellent and simple analytical method for observing the general concentration of urine or its dilution. *Jähring et al.* (*1977*) e.g. could show the correlation between Na^+ concentration and osmolarity versus Na^+ concentration and conductivity in fresh urine. Because osmolarity is affected by amounts of glucose in urine which can vary, conductivity served as a better parameter for renal concentration power.

In urine stored in collection tanks, ammonium is one of the dominating parameters. Therefore a strong correlation of NH_4-N and conductivity can be observed (Figure 13).

The median conductivity was 27 mS/cm. Values were varying between 7 and 33 mS/cm.

The linear regression of the correlation between ammonium und conductivity can be described by Eq 2 with an R^2 of 0.85.

Eq 2 $$NH_4 = 0.1667 * \mathrm{Conductivi\ ty} - 0.927$$

Also the concentrations of phosphorous can be given roughly by measuring the conductivity (see Figure 14), since usually all content parameters of urine are in a similar ratio. From Figure 14 a resulting transformation factor that is described in Eq 3. The measured concentrations correlated with an R^2 of 0.62.

Eq 3 $$P = 12.728 * \mathrm{Conductivity} - 43.88$$

Due to uncontrolled precipitation processes within the collection tanks, phosphorous concentrations can vary leading to deviations of the nitrogen-phosphorous ratio and thus to a lower correlation with conductivity.

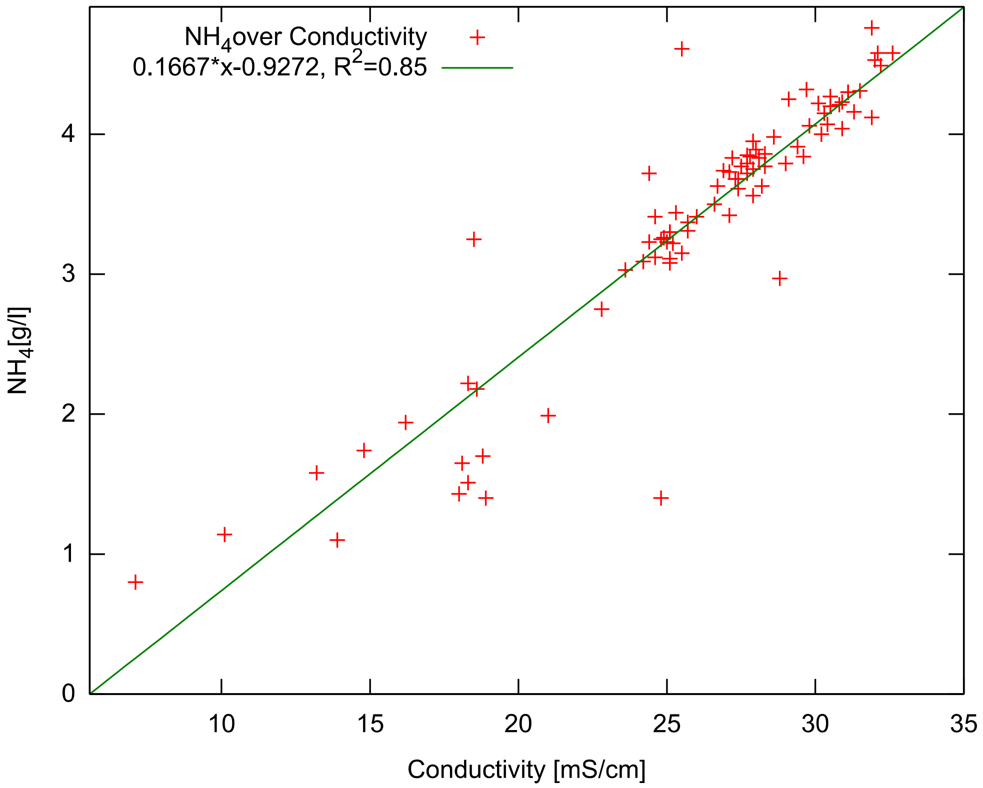


Figure 13: Correlation of NH_4-N and conductivity

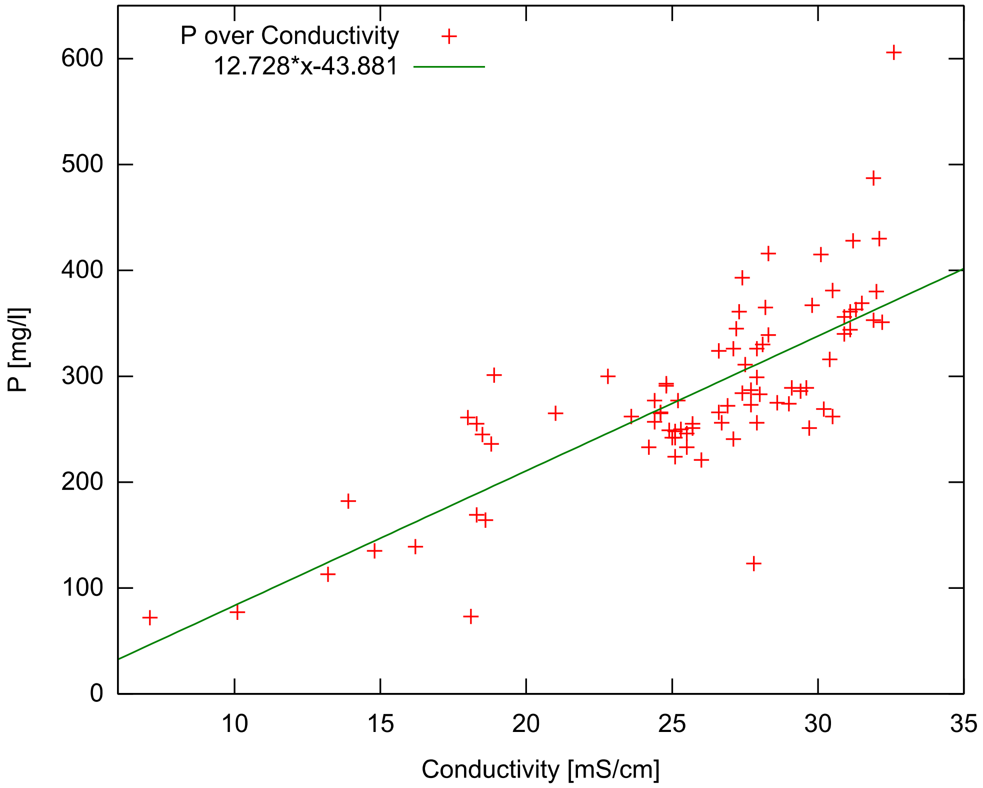


Figure 14: Correlation of P and conductivity (R^2 = 0.62)

Low ammonium concentrations and to some extent low phosphate concentrations could be explained in most cases by low filling levels. Except from one example, filling level and concentrations were correlating.

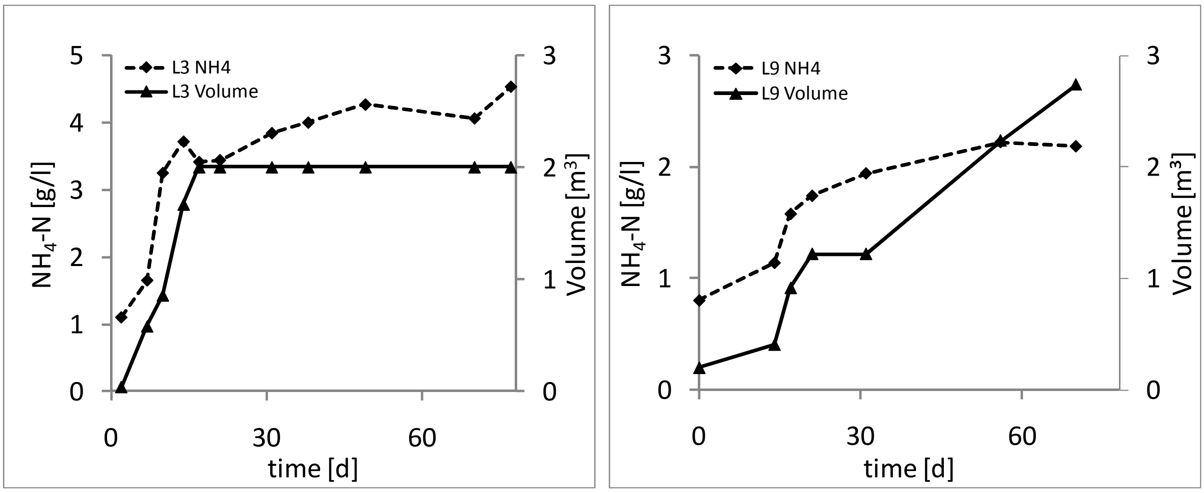


Figure 15: Filling level (volume) and ammonium concentration at two different locations over time

At two locations with slow volume increase (L6 and L9), concentrations of ammonium were significantly below average. Additionally, concentrations of ammonium were lower at one location in the very first filling phase (L3). An example of both situations is displayed in Figure 15. The low ammonia concentrations in these cases can be explained by a high surface to volume ratio, supporting out-gassing of ammonia. At locations with high volume increase and during high filling volumes concentrations were mainly above average.

Varying phosphate concentrations occurred especially at low to medium filling levels. Here, dilution effects with water from the daily cleaning session that sometimes was not hindered from entering the collection tanks could have happened. Dilution effects from rainwater that might have entered the collection tanks and affected concentrations especially at low filling levels can not be excluded.

Additional chemical properties and summarizing chemical parameters

Between 10 and 17 grab samples were analyzed for total carbon, chloride, phosphate, sulfate, potassium, magnesium, calcium, and sodium. An overview of the results is given in Figure 16. Average, median, minimum, and maximum values of all measured chemical parameters are given in Table 3.

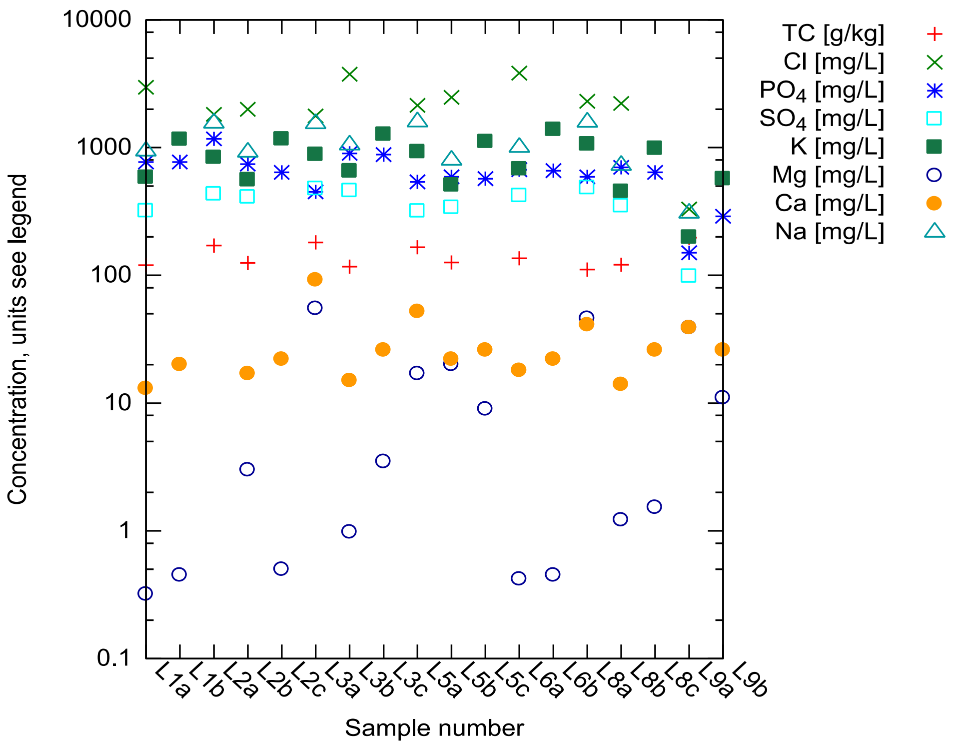


Figure 16: Chemical properties of urine samples (NH$_4$-N and TP excluded). Sample number Lix indicates location i and sampling time x

As presented in Figure 9, pH varied little between 8.2 and 9.4 with a median pH of 9.14 leading to a conclusion of a totally hydrolyzed substrate. In this pH range precipitation processes occur, leading to sediment that is easily dispersed during sampling. This is explaining to some extent the variations of calcium and magnesium concentrations which were generally low (median 22 respectively 3 mg/l) with minimum and maximum values of calcium: 13–92 mg/l respectively magnesium 0.32–55 mg/l.

Sulfur, chloride, sodium and potassium were in an expected range varying in a range of 20 to 40 % of their median values.

Table 3: Average values, median, min, and max of urine samples of all sampled public toilets in Hamburg

	pH	cond. [mS/cm]	NH$_4$-N [mg/L]	TP [mg/L]	PO$_4^{3-}$-P [mg/L]	SO$_4^{2-}$-S [mg/L]	Cl$^-$ [mg/L]	Na$^+$ [mg/L]	K$^+$ [mg/L]	Mg^{++} [mg/L]	Ca^{++} [mg/L]
Number of samples	88	88	77	74	17	10	10	11	17	17	17
Average	9.08	27.93	3700	292	222	134	2523	1094	871	12	29
Median	9.14	27.25	3760	280	215	138	2255	1007	882	3	22
Min	8.22	7.10	1100	73	95	106	1770	307	454	0.32	13
Max	9.44	32.60	4763	430	382	162	3820	1595	1385	55	92
Standard deviation	0.22	3.35	1016	64	64	22	747	426	292	18	19

Microorganisms

Ten samples of the collection tanks were tested for six different microorganism cultures. Two additional samples were stored for two months and tested for the different microorganism cultures thereafter.

Entrobacter were found in onla one of ten samples, Escherichia coli and Pseudomonas were found in two of ten samples, Proteus were found in three of ten samples, Staphylococcus were found in five of ten samples, and Streptococcus were found in seven of ten samples. The average number of colony forming units (CFU) ranged from three CFU/ml for Escherichia coli up to 57 CFU/ml for Staphylococcus (Figure 17 and Table 4).

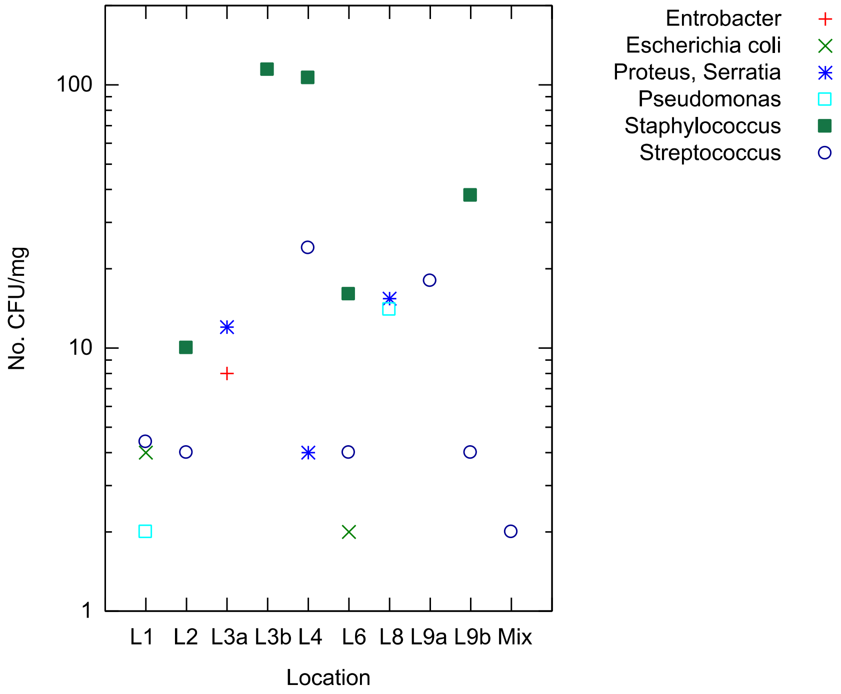


Figure 17: Numbers of colony forming units (CFU) per milliliter of substrate at different locations

Table 4: Detected colony forming units per ml of different microorganism parameters

Type	No of investigated samples	No of positive tested samples	Min. – Max. of positive samples [CFU/0.5ml]	Ave. in positive samples [CFU/ml]
Entrobacter, Klebsiella a.o.	10	1	4	8
Escherichia coli, Citrobacter a.o.	10	2	1–2	3
Proteus, Serratia a.o.	10	3	2–7	10
Pseudomonas	10	2	1–7	8
Staphylococcus	10	5	5–57	57
Streptococcus	10	7	1–12	9

In the two samples stored for two months before testing for microorganism parameters in one case Proteus, Serratia only was detected (58 CFU/ml).

Micropollutants

Eight of the tested 21 pharmaceuticals were found in the urine at the location L3 and at the separation system of BWB Stahnsdorf between March 2005 and Mai 2006. Concentrations are given in Table 5 and presented in Figure 18.

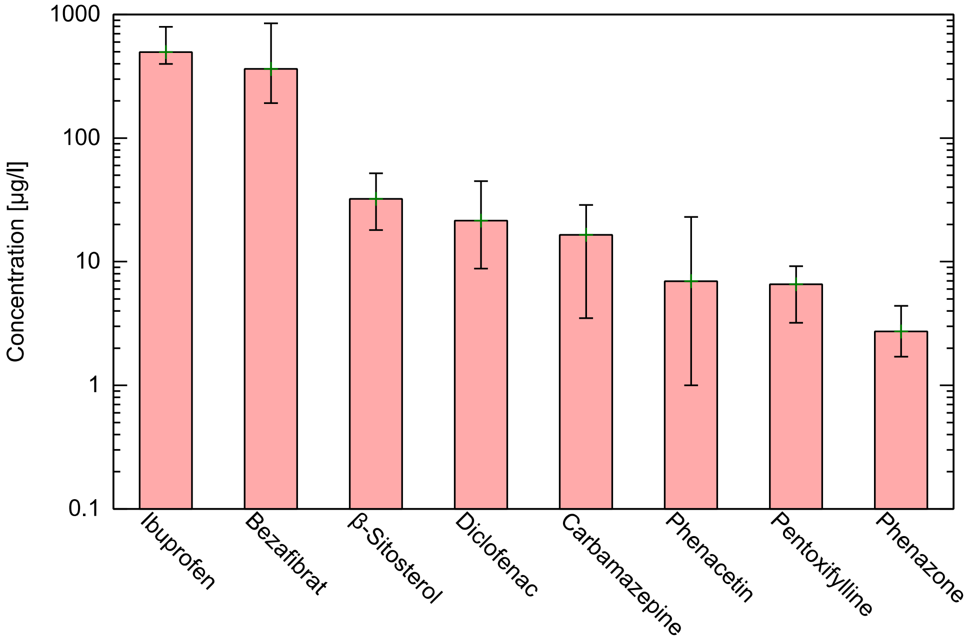


Figure 18: Average concentrations and the minimum and maximum concentrations of pharmaceutical residues found in urine

Table 5: Concentrations of pharmaceutical residues found in urine from a public urinal in Hamburg (L3) and from the separation system Stahnsdorf, Berlin

	No of investigated samples	**No of positive tested samples**	***Average*** [µg/l]	***Min*** [µg/l]	***Max*** [µg/l]	***95 % Confid*** [µg/l]
Ibuprofen	6	6	496	398	794	121
Bezafibrat	6	6	362	192	846	211
ß-Sitosterol	6	6	32	18	52	10
Diclofenac	6	6	21	9	45	10
Carbamazepine	6	6	17	4	29	7
Phenacetin	6	4	7	1	23	8
Pentoxifylline	6	5	7	3	9	2
Phenazone	6	3	3			

Ibuprofen and Bezafibrate were the pharmaceuticals with the highest concentrations by far (nearly all samples above 200 µg/l, and up to 850 µg/l). β-Sitosterol, Diclofenac and Carbamazepine were detected in ranges between 10 and 50 µg/l. All of these were found in all samples. Phenacetin (detected in four of six samples), Pentoxifylline (detected in five of six samples), and phenazone (detected in three of six samples) were mainly detected in the range just above the detection limit between 1 and 10 µg/l. All other pharmaceuticals were below the level of detection and are presented in Table 6.

Table 6: Substances below detection limit resp. not detected in urine samples

values in µg/l	**LOD**
Fenoprofen	<1.0
Indometacin	<1.0
Ketoprofen	<1.0
Clofibric acid	<1.0
Fenofibrat	<1.0
Estrone	<10
17α-Ethinylestradiol	<50

values in µg/l	**LOD**
Acetylsalicylic acid	no
Gemfibrozil	no
Estradiol	no
Estriol	no
Mestranol	no
16α-Hydroxyestron	no

B.4.3. Limitations

Because of geometry and size of the tanks, precise measurement of filling height, and thus of the contained volume, was not possible in all cases. The tank contents could not be homogenized in all cases before sampling. It can not be excluded that phosphorous containing detergents ended up in the tanks, since the valve for closing the tank and detouring the washing agents into the sewer system was not always used by the caretakers. It could be that surface runoff or high groundwater entered some of the tanks.

B.5. Data comparison

Nutrients and relevant ions

When comparing the average values of the nine public locations from 2009 (Ave L1-L9) with average values of one public location and one location at a separating system in Berlin, Stahnsdorf, including an office building and several apartment houses, higher values can be observed in both locations analyzed in 2005 (Table 7). Interesting to observe is the fact that from the two sites investigated in 2005, concentrations of urine of the separating system involving an office building and apartment houses are lower than the ones of the public urinal. However, it is likely that two different effects influenced the concentrations: at public urinals low concentrations can be expected due to the fact that only the fraction of low concentrated urine is collected. It can be assumed that mainly persons with a higher drinking input tend to use public toilets. In the case of the separating system including an office building and apartment houses male-functions of the separating toilets were reported leading potentially to flushing water entering the collection tanks.

Table 7: Comparison of measured data from 2009 with data from 2005

	pub* 2005	off & prv** 2005	Ave L1-L9 2009/ pub 2005	Ave L1-L9 2009/ off & prv 2005
pH	8.90	8.80	102 %	103 %
Conductivity [mS/cm]	~ 20	33.2	136 %	82 %
TN [mg/l]	4300	4200	87 %	90 %
TP [mg/l]	408	380	69 %	74 %
K^+ [mg/l]	1360	1200	65 %	74 %
Mg^{2+} [mg/l]	~ 0.2	8.40	nc	36 %
SO_4^{2-}-S [mg/l]	830	404	16 %	33 %
Na^+ [mg/l]	2085	na	48 %	nc
Cl^- [mg/l]	3430	na	66 %	nc

* pub: public urinal, Hamburg L3; ** off&priv: office building and apartment houses; in both cases mean values of three samples; data from (*Tettenborn et al. 2007*)
na: not analyzed; nc: not calculated

Comparing the presented concentrations within the urine of the public urinals in Hamburg with an extensive survey conducted by *Meinzinger & Oldenburg* (*2009*) and with other typical values for fresh urine (Table 8) it can be noted that the measured values are significantly lower than the literature values (see Table 9 and Appendix table 3).

Table 8: Typical concentrations of urine

Meinzinger & Oldenburg **(*2009*)**

	Data quality	Median [g/l]	Min [g/l]	Max [g/l]
COD	fair	5.0	4.0	11.0
N	fair	8.3	1.8	17.5
P	fair	0.8	0.2	3.7
K	fair	1.9	0.7	3.3

	Ciba Geigy AG **(*1977*)**	***Roempp*** **(*1997*)**	**Rink 1964***
	Average [mg/l]	Average [mg/l]	Range [mg/l]
Cl^-	3 800	5 930	4 400–6 600
Na^+	4 200	3 930	2 933–4 400
Ca^{2+}	170	330	7.35–220
Mg^{2+}	100	270	126–209

* extracted from *von Wolffersdorff* (*2004*)

Table 9: Comparison of measured median values and literature values

	Measured Median / Literature values
N	45 %
P	35 %
K	46 %

	Measured Median / Literature values
Cl	44 %
Na	26 %
Ca	9 %
Mg	2 %

While nitrogen can be lost in urine storage systems by evaporation of ammonia, and phosphorous can be lost from the solution by precipitation processes, potassium, sodium, and especially chloride should remain in solution and can give therefore valuable information about the original concentration of the average urine collected within the system. Assuming an average literature value for chloride around 5.2 g/l, and an average literature value for sodium around 3.9 g/l, in the samples of the public toilets 44 % of chloride of the average literature values, and only 26 % of sodium compared with the average literature values were detected. Potassium and also nitrogen are in a similar range as chloride. Therefore major losses by ammonia evaporation most likely did not occur. The ratio of measured phosphorous concentrations to literature values is with 35 % a bit lower than the ratio of nitrogen, potassium, and chloride. The ratio for magnesium and calcium values is significantly lower than for the other elements. Therefore it can be assumed that first precipitation processes already occurred within the collection tanks. About 15–20 % of the phosphorous had probably already precipitated.

From the roughly 45 % lower concentrations it can be assumed, that also concentrations of pharmaceutical residues would be higher in source separated urine collected at other locations.

While the presented measured data were gained from public urinals only, concentrations of urine in households and at working places can be found in literature. However, also here detected concentrations are rather low. *Jönsson et al.* (*1997*) e.g. is reporting for households with urine diverting toilets in Sweden phosphorous and nitrogen concentrations in the range of 40–48 % of the compiled median of *Meinzinger & Oldenburg* (*2009*), while *Rossi et al.* (*2009*) is reporting of households involving waterless urinals and urine diverting toilets nitrogen and phosphorous concentrations of 30–55 % of the compiled median of *Meinzinger & Oldenburg* (*2009*), and of working places nitrogen concentrations of 55–78 % of the compiled median of *Meinzinger & Oldenburg* (*2009*).

Microorganisms

According to *Schönning et al.* (*2002*) most bacterial contamination of urine is due to misplaced feces. Source separated urine from urine diversion toilets is on average contaminated by 9.1 mg feces per liter, leading to coliform contamination with mean values of 260 CFU/ml (median 21 CFU/ml), clostridia spores were found in concentrations, ranging up to 2 000 CFU/ml, Faecal streptococci occurred in high concentrations with partially more than 100 000 CFU/ml (*Schönning et al. 2005*).

Although in the present study only urine from waterless urinals was investigated, targeting microorganisms contamination from misuse can not be excluded, since during the sampling period the results of misuse was observed. Nevertheless detected concentrations were rather low.

Micropollutants

Micropollutants in for of pharmaceutical residues in urine storage tanks were also investigated in the research project Lambertsmühle (*Niederste-Hollenberg 2003*; *Strompen et al. 2003*) (Table 10).

Table 10: Concentrations of pharmaceutical residues found in urine in the research project Lambertsmühle.

values in [μg/l]	***Influent to storage***	***Storagetank***
Ibuprofen	1.2–720	1.6–161
ß-Sitosterol	5–410	8–92
Diclofenac	0.6–53	0.6–34
Carbamazepine	1.6–4.8	0.8–6.5
Clofibric acid	2.9–9.1	5.3–21
Estrone	10–259	Ave. ~ 50
Fenoprofen	nd	0.4–4.8
Bezafibrat	nd	nd
Phenacetin	nd	nd
Pentoxifylline	nd	nd
Phenazone	nd	nd
Indometacin	nd	nd
Ketoprofen	nd	nd
17α-Ethinylestradiol	nd	nd
Acetylsalicylic acid	nd	nd
Gemfibrozil	nd	nd
Estradiol	nd	nd
Estriol	nd	nd
Mestranol	nd	nd

nd for not detected

Source: *Niederste-Hollenberg* (*2003*)

Concentrations of ibuprofen and diclofenac were more or less in an equivalent range or slightly higher compared to the values of Lambertsmühle. Concentrations of carbamazepine and bezafibrate were significantly higher in collection tanks of the public urinals. Phenacetin, pentoxifylline, and phenazone were detected in more than 50 % of the samples from the collection tanks of the public urinals, however, concentrations were rather low. Clofibric acid and estrone could not be detected in the collection tanks of the public urinals but significant concentrations were found in the system of Lambertsmühle. This is no wonder as estrone this takes no wonder since it is a natural female hormone that should not occur in significant amounts in tanks collecting urine from urinals for males.

Values obtained within the research project Lambertsmühle varied up to two log units and more, while the values obtained from the public urinal were varying in average by a factor of 3 to 4 with one exception. This is most likely due to the much larger number of contributors in the public collection system. Therefore the values obtained from the public collection system can be used as further improvement for average concentrations of pharmaceutical residues in urine collection systems.

B.6. Intermediate discussion

After these consistency checks, the measured concentrations in the collection tanks of the public urinals in Hamburg can be used for designing new sanitation concepts involving large urine collection tanks with public access, in northern Europe. Nutrient concentrations should not be expected to exceed the concentrations measured within the public toilets in Hamburg, given in Table 3.

However, concentrations of the collected urine will depend on the collected fraction (differs from location: public toilets, toilets at business locations, toilets from private homes) and on the type of collection system (waterless urinals – no dilution by entering flushing water but used only by a certain quantity of male population; urine diverting toilets: separation grade, and possible dilution by entering flushing water).

Currently waterless urinals are the easiest way to collect urine undiluted. Although their distribution is limited mainly to public and semi-public areas (such as work etc.) and their usage is limited so far to only half of the population many large institutions are already equipped with waterless urinals just for water saving issues. During rehabilitation measures additional piping and collection tanks could be implemented for new constructions complete separation systems could be implemented right away.

Separation toilets are available but have their limitations in separation grade and dilution rate. Toilet manufactures seem to be skeptic regarding their future market potential (*Kannengießer 2010*).

It is believed that the collection of source separated urine at public places via urinals is the one easiest to be realized for collecting large volumes as the example of the city of Hamburg could show. However, concentrations were rather low. Despite the reported low concentrations found in some collection systems of living houses and working places[6] it is believed, that at non-public locations higher concentrations can be yield as long as dilution by flushing or cleaning water, or losses of ammonia and phosphorous can be reduced.

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[6] e.g. SCST Stahnsdorf, Berlin (*Peter-Fröhlich et al. 2004*; *Tettenborn et al. 2007*); Switzerland Forum Chriesbach eawag, Dübendorf (*Larsen & Lienert 2007*; *Larsen et al. 2001*)), Sweden (*Hellström & Johansson 1999*)

C. Transport and logistics

C.1. Introduction

In large scale UD-systems the transport of the separately collected urine would become an important topic, since a specific infrastructure needs to be developed. The location of collection (grade of decentralism) and the location of treatment (semi-central, central) are influencing the transport logistics and are depending to some extend on population density, quantities, and infrastructure. The two main options for the transport itself are piping systems (gravity or pressure) and wheel based pick-up systems with collection tanks and pick up trucks. More detailed descriptions of the different systems can be found in *Arnold et al.* (*2004*), *Bilitewski et al.* (*1997*), and *Hosang & Bischof* (*1998*).

Meinzinger (*2010*) compared transport scenarios for different source separation approaches in the urban context of Hamburg, Germany, and Arba Minch, Ethiopia in a material and energy flow analysis. The separated transport of source separated wastewater was one of the factors leading to an increase in the overall energy demand of a system. This increase could be balanced by energy savings e.g. due to nutrient recovery from urine or due to production of biogas, both enabled by source separation.

Costs for different transport scenarios for new sanitation concepts were compared by *Oldenburg* (*2007*). He found that: “The multiple sewer systems resulting from the separation of urine, brown or grey water are responsible for higher investment costs, which control the total project costs”. The costs for connection pipes and the sewerage for a separate urine transport in a 3-piping system were found to be at least about twice as high as the conventional solution. From the investigated scenarios the one with urine collection at household level and pick-up from thereon was in terms of total project costs and compared to the other new sanitation scenarios also in terms of investment costs the one with the lowest costs.

A different approach was investigated by *Rauch et al.* (*2003*) in which urine would be collected separately at household level and released into the sewer system “according to a centrally coordinated strategy”. However, “the aim was not the permanent separation of urine for recycling but the control of the temporal profile of the waste stream”, to level “out the nitrogen dynamics at the wastewater treatment plant”, and to avoid “combined sewer overflow containing urine”. However, a reuse as it is described in the framing scenario given in chapter A.4.2 would not be possible.

Because of the small volumes of urine generated per person and the general aim for high flexibility in scale that would also allow a stepwise integration of collection points spread over the city as currently realized in the case of public urinals, a combination of short distance piping systems to collection tanks and a pick up and transport from thereon with trucks or through infrastructure channels is the option that is looked at closer in the following chapter.

C.2. Background

C.2.1. Logistics

Logistical concepts are needed for efficient and resource saving pick up and transportation, especially in cases where different concepts might coexist as it might happen in the future: waste and different wastewater streams, different collection and transport systems, all combined at different transition stages.

The logistical aspects involve the handling of goods and services including storage, transport, and commissioning of the different products at different stages (*Tchobanoglous 1993*). The different steps can be divided in four main fields:

- Procurement logistics: how can the raw material find its way to the production site?
- Production logistics: how can the production/treatment be handled?
- Distributional logistics: how can the finished goods arrive to the costumer?
- Disposal logistics: how can waste be collected and finally disposed of or recycled?

Seeing source separated urine as resource rather than as waste, the evaluation on how separated urine can be transported to larger treatment facilities can be seen as procurement logistics which are the topic of this chapter. Production logistics are part of chapter D and E. Distributional logistics was the topic of *Tettenborn et al.* (*2009*), who evaluated the nutrient demand in Ghana and in the region of Beijing, China, that could be covered by nutrients from urine separation. For Beijing, different truck based transport options with transfer stations were discussed. Finally, disposal logistics are covered to a limited extend in chapter E, when discussing how the remaining side products from urine treatment can further be treated.

Collection and transport logistics can be distinguished depending on how the collection, transport, and distribution of resources and products are organized. Products from source separating systems can be transported directly to a treatment facility and distributed from thereon, or transport and distribution can be spread over transfer stations. Transfer stations can help to increase efficiency of long distance transport (*Bilitewski et al. 1997*), and can also be used for an intermediate treatment step or for storage.

C.2.2. Piping of urine

Some experiences of urine pipes were gained e.g. in the SCST-project of KompetenzZentrum Wasser Berlin (*Peter-Fröhlich et al. 2007a*) and in the NOVAQUATIS-project of the Swiss eawag (*Larsen & Lienert 2007*). In both projects transport distances did not exceed 1 km and in both cases severe incrustations did not occur.

Within the NOVAQUATIS-project precipitation processes in the piping system were observed and investigated. While some precipitation formation in traps and pipes occurred which could cause blockages (*Udert et al. 2003a*), most of the precipitation forming phosphate minerals were incorporated in the urine sludge at the bottom of the collection tank (*Udert et al. 2003b*) and could be pumped with the urine solution (*Udert et al. 2006*).

Regarding the collection systems in Hamburg, *Grönwall* (*2008*) reported heavy incrustations in the very first collection systems. After changing the design of odor traps combined with frequent maintenance, incrustation problems vanished.

While the investigated urine piping systems were based on gravity piping, experiences from vacuum systems in airplanes can be consigned to potential urine vacuum piping systems. In airplane vacuum systems, toilets with low flushing volumes are installed to keep the overall weight of flushing water to a minimum. It can be assumed that the largest fraction of toilet usages is due to urination. Incrustations occur quite frequently. Analysis of incrustations showed very comparable characteristics to incrustations in gravity based urine piping systems (*Tettenborn et al. 2008*). It can be expected that due to thin film formation on the inner walls of the vacuum pipes during the vacuum process, drying and evaporation processes will lead to an increased formation of incrustations. While in aviation high-tech materials and cleaning processes are used to cope with this situation, the implementation of vacuum systems for urine transport seems non-recommendable unless absolutely needed.

Although incrustations seem to be controllable in smaller piping systems, they could state a thread for long distance piping systems.

C.2.3. Wheel based pick up systems

Wheel based pick up systems are known from waste management systems. Here, the following factors are considered when setting up a logistic transportation system (*Bousonville 2002*):

- characterization of waste (density, weight, fractions, humidity),
- geographical preconditions (location, altitude, street system),
- storage container requirements – this includes volumes, temperature(climate), and substance specific considerations, and
- location of the pick up (in house, in common storage vessels).

Additionally in respect to the waste pick-up, the following constraints for service were defined by *Gallenkemper & Doedens (1994), MacFarland* (*1997*) and *Bilitewski et al. (1997)*:

- grade of separation,
- type of container system (in house, in the ground, fixed, movable),
- frequency of the collection,
- full service or partial service (auxiliary activities by the inhabitants),
- number of personnel required,
- quantity to be hauled for present and future loads,
- truck maintenance downtime,
- type and conditions of the roads,
- percent of time when the vehicles will be in productive use,
- residential lifestyle, and
- zoning laws and ordinance

The most relevant of these aspects are considered in the following chapter.

C.3. Focus of investigation

How should collection of source separated urine be organized? The possibilities of a truck based transport system were investigated on the example of Hamburg. Main questions were: How many collection points and what size of collection vessels would be suitable for a city like Hamburg?

For this a collection logistic for source separated urine in an urban context on a city scale was set up to provide reliable information about the pick-up system in an industrial environment, including costs and environmental loads associated.

Also the energy demand for transport via vehicles was derived from the model.

C.4. Material and methods

C.4.1. Study site Hamburg

The chosen site of Hamburg has a population of about 1.78 million inhabitants (*Statistikamt Nord 2010*), with a density of 2 300 p/km^2 which is comparable to other larger cities in industrialized countries (*Statistics 2010*). The distribution of the density over the different districts is presented in Appendix table 4 and Appendix table 5. The total number of buildings for living is about 232 000 consisting of roughly one third decentralized and family houses and two thirds multi family houses. The number of houses and the type of houses is typical for a European city with a high number of single family houses and only a small number of high rise buildings, in contrast to the United States of America or Asia. The total street length in the city of Hamburg is nearly 4 600 km (Appendix table 5).

C.4.2. Scenario set-up

Roughly based on the situation in Hamburg, three scenarios of a vehicle based transport system were modeled and evaluated with Microsoft excel. For each scenario the coverage was varied. The coverage is representing the number of persons involved, ranging from 0.1 % of the total population of the city of Hamburg involving about 1 800 persons, up to an idealistic full coverage of 100 % involving all 1.78 million persons living in Hamburg at the end of 2009. The range of coverage is displayed in Appendix table 8.

The three scenarios are completely fictitious and are describing extreme situations. The scenarios differed in the number, size, and distribution of collection vessels. To learn about the influence of vessel size, a total of five different vessel sizes were used within the three scenarios.

- In the first scenario (S1) a pickup mainly at household level with very small collection vessel sizes and only very few large collection vessels for coverage of highly dense populated areas is described. It is assumed that more than 75 % of the covered population would have their own individual collection vessel of 50 l that would be emptied on a weekly or fortnightly basis. The remaining 25 % of the covered population would be equipped with 200 l vessels (18.75 % of the covered population) and for a few central places 10 m^3 vessels would be used (for substrate collection of 6.25 % of the covered population).

- In the second scenario (S2) a pickup at neighborhood level is described. The same population fractions were used as in scenario 1 but the two smaller vessel sizes were modified to 0.5 m^3 for 75 % of the covered population and 2.5 m^3 for 18.75 % of the covered population. The large vessel size of 10 m^3 remained for 6.25%.
- In the third scenario (S3) a pickup at community level with an emphasis on a large number (for 85 % of the covered population) of 10m^3 collection vessels is described. The substrate of 12 % of the remaining population would be collected in 2.5 m^3 vessels, and a small fraction of 3 % would be fitted with 0.5 m^3 vessels.

The detailed distribution of the different collection vessels in the three scenarios is presented in Table 11.

Table 11: Assumed distribution of the different sizes of collection vessels in the three scenarios for 100 % coverage

Scenario	Description	Total number of collection* vessels	Small size containers [m³]	Ffraction of total vessel number *	Medium size containers [m³]	Ffraction of total vessel number*	Large size containers [m³]	Ffraction of total vessel number*	Average number of connected persons per vessel
S1	Individual household collection vessels	262 778	0.05	94 %	0.2	5.9 %	10	0.04 %	6.8
S2	Small neighborhood collection vessels	26 063	0.5	95 %	2.5	4.7 %	10	0.4 %	68
S3	Large community collection vessels	3 181	0.5	31 %	2.5	25 %	10	44 %	560

* fraction is based on the total number of vessels and not volume based
(depending on number of persons connected to one type of vessel size)

C.4.3. Work flow and assumptions

It was assumed that the number of tours per time would mainly be influenced by the individual volume to be transported per scenario and coverage. For each scenario and coverage the overall tour distance (d) was assumed to be an average distance from the main collection and treatment point to the individual collection points at household, neighborhood, or community level and back to the main collection and treatment point. A comparison with *Meinzinger* (*2010*) who developed a complex transport model based on detailed GIS-data for source separated wastewater streams proved this assumption to be correct.

The city of Hamburg has dimensions of more or less 30 km by 40 km [7]. Therefore the average tour distance for on centralized collection and treatment point was assumed to be half of the 40 km length of a complete tour.

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[7] Derived from Google maps 2010: Varendorfer Stadtweg in “Naturpark Harburger Berge” to Puckaffer Weg in “Duvenstedter Brook“

Depending on the coverage, up to four main collection and treatment points were considered (as shown in Appendix table 6) to be evenly distributed over the area of the city of Hamburg. The increase of the number of main collection and treatment points (n_{ctp}) would lead to a decrease of average tour distance by the factor $\sqrt{n_{ctp}}$ listed also in Appendix table 6.

Due to the fact that during a pickup at household level smaller streets would have to be covered, it was assumed that in scenario 1 the total driving distance was larger than in scenarios 2 and 3. This was compensated by a detour factor (f_d) of 1.4 for scenario 1, and 1.1 for scenario 2, multiplied by the driving distance for collection.

The total volume to be transported per coverage and scenario was calculated from the numbers of people involved using a safety margin of 10 % (Eq 4).

Eq 4
$$\mathbf{Q_{td} = 1.25\,l/(p * d) * 1.1 * n_p}$$

Q_{td} : Volume to be transported per day per scenario and coverage
n_p : number of persons involved per scenario and coverage

For simplification and better comparison in all scenarios, a maximum pickup frequency of once a week during five working days per week was used for further calculations (Eq 5).

Eq 5
$$\mathbf{\frac{N_{Tours}}{day} = Q_{td} * \left(\frac{7days/week}{5days/workingweek}\right) * (16\ m^3\ capacity\ per\ tour)^{-1}}$$

The distance per week was calculated by Eq 6.

Eq 6
$$\mathbf{d_{week} = \frac{N_{Tours}}{week} * d_{tour} * \frac{f_d}{\sqrt{n_{ctp}}}}$$

d_{week}: distance per week
N_{Tours}: Number of tours
d_{tour}: distance per tour
f_d: detour factor
n_{ctp}: number of main collection and treatment points

To evaluate manpower requirements and the number of required suction vehicles, additional assumptions were made for average speed, routing time, time for connection, and time for suction according Appendix table 7.

The required total working time per week per scenario and coverage was calculated by adding driving time, routing time per tour, connection time, and suction time (Eq 7).

Eq 7
$$\mathbf{t_t = d_{week} * v_{ave} + t_r * \frac{N_{Tours}}{week} + \sum[(t_c(ves) + t_s(ves)) * n_{ves}]}$$

t_t: total working time per week
v_{ave}: average velocity
t_r: routing time per tour
t_c: connection time per connected collection vessel
t_s: suction time per connected collection vessel
n_{ves}: number of vessels

From the total required working time needed per week for collection of the whole volume per coverage, the number of required vehicles and staff members was derived assuming a five day working week and an average time of 35 h per person. Similar to semi-automated waste-collection one person was assumed to operate one vehicle.

From the number of collection vessels and the number of required collection vehicles a very rough estimation of investment costs per scenario and coverage was made. From labor effort and required fuel a very rough estimation of operation costs per year was also made. Both are presented in the appendix in chapter I.2.

From the consumed amount of fuel per scenario and coverage the required energy for transport was derived.

C.5. Results and discussion

The average tour distance within the three scenarios was depending on the number of collection and treatment points and on the detour factors used in scenarios where pickup involved using smaller streets. Based on the simplified assumptions, the average tour distance per scenario and coverage ranged from 56 km in scenario 1 with very little coverage down to 20 km in scenario 3 with very high coverage. Details are presented in Table 12. This is in a comparable range of that found by *Meinzinger* (*2010*), who derived an average distance of 17.8 km for one way in scenarios with one centralized collection and treatment point (compare to coverage 0.5–25 % in Table 12), and 9.2 km for one way in scenarios with four collection and treatment points (compare to coverage 81–100 % in Table 12).

Table 12: Average tour distance per scenario and coverage

Coverage [%]	Average tour distance [km]		
	S1*	S2*	S3
0.5–25	56	44	40
26–50	40	31	28
51–80	32	25	23
81–100	28	22	20

The demand in vehicles and personnel was estimated based on the total time consumed for the collection trips per scenario and coverage. Due to the higher numbers of vessels to be emptied in scenario 1, the total time consumed for collection was the highest, resulting in a maximum team and vehicles demand of 57 for 100 % coverage. 42 teams and vehicles would be needed for collection at 100 % coverage in scenario 2, 37 teams and vehicles in scenario 3.

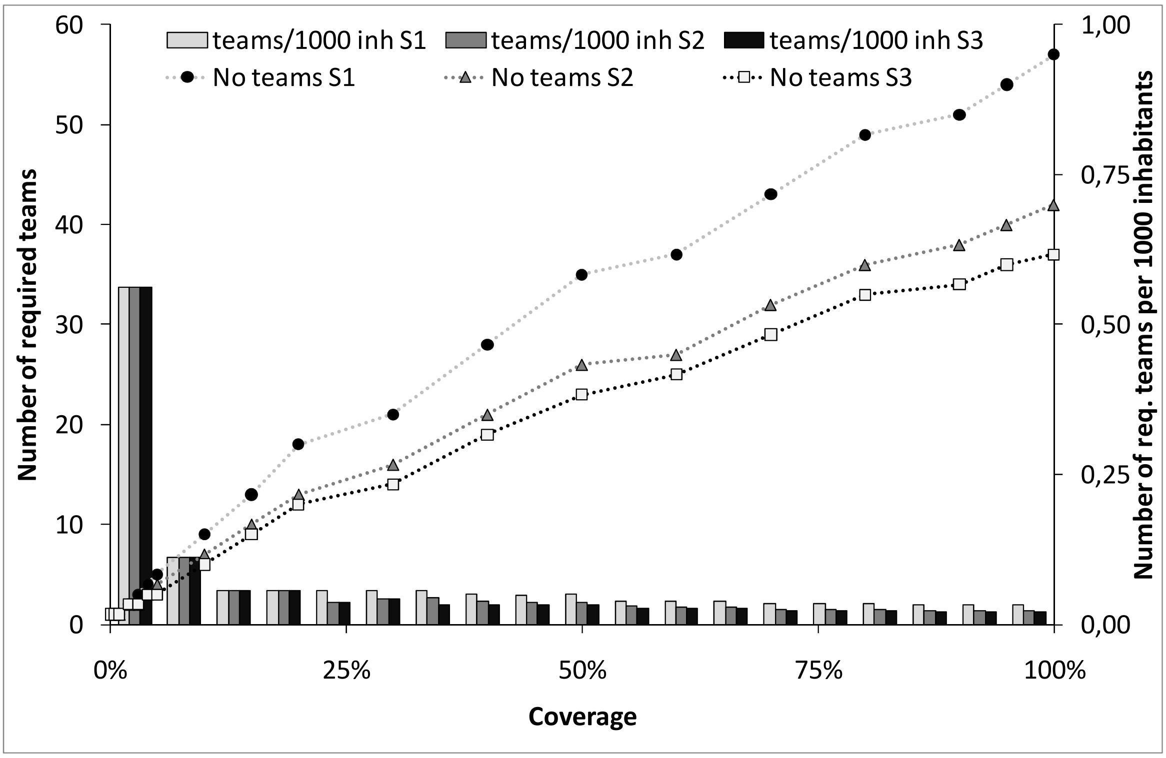

Figure 19: Number of required teams (lines) and number of required teams per 1 000 inhabitants per scenario and coverage (bars)

As presented in Figure 19, the personal and vehicle demand per 1000 inhabitants ranged from 0.56 at 0.1 % coverage to 0.02 at 100 % coverage, more or less the same for all three scenarios.

With very rough cost estimation the following general conclusions could be derived:

At the stage of 0.1 % coverage, the initial investment costs (including vessels and vehicles) were in a similar range in all three scenarios due to the fact that the costs for collection vehicles were dominating the costs for collection vessels by a factor of 20 in scenario 1 and 30 in scenario 3. With increasing coverage, the ratio between costs for required vehicles and required vessels was more balanced in a range between 1 and 3. A very similar picture can be derived for the ratio between personnel and fuel costs. While personnel costs are dominating at very low coverage, the ratio gets more balanced with increasing coverage.

Due to the high number of collection vessels in scenario 1, investment costs would be significantly higher and also operational costs, manly due to higher time consumption for collection and consequently a higher demand in personnel.

In the simplified scenarios the energy consumption for the transport was directly linked to the average transport distance per tour and per transported volume. In the three scenarios the number of main collection and treatment points was increased from 1 for low coverage of 0.1–20 % up to 4 collection and treatment points for coverage of more than 80 % (Appendix table 6). The differences between the three scenarios are mainly due to the detour factor used because of the fact that in a scenario with pickup from household level smaller streets have to be serviced compared to a scenario with large community tanks that can be mainly accessed via larger roads. In general for smaller systems with up to 10 000 people, the involved energy demand would be around 11 to 16 kWh/m³. By tour optimization and increase of the number of main collection and treatment points, the energy demand for collection and transportation on a vehicle basis in an urban context similar to Hamburg could be lowered to 5.7 kWh/m³ transported substrate, as derived here in scenario 3 for coverage of more than 80 % (Figure 20). The results for high coverage are in a comparable range of *Meinzinger* (*2010*) who calculated an energy demand of 3.6 kWh/m³ for transport in a urine separation scenario with 100% separation and 100% coverage.

Equivalent to the energy demand per transported volume presented in Figure 20, the production of CO_2 is linked directly to the transport distance and consumed fuel. For a realistic scenario in a city like Hamburg it would be in a range of 1.3 to 3.6 kg CO_2/m³ transported volume depending on scenario and coverage (conversation factors listed in the appendix in Appendix table 9).

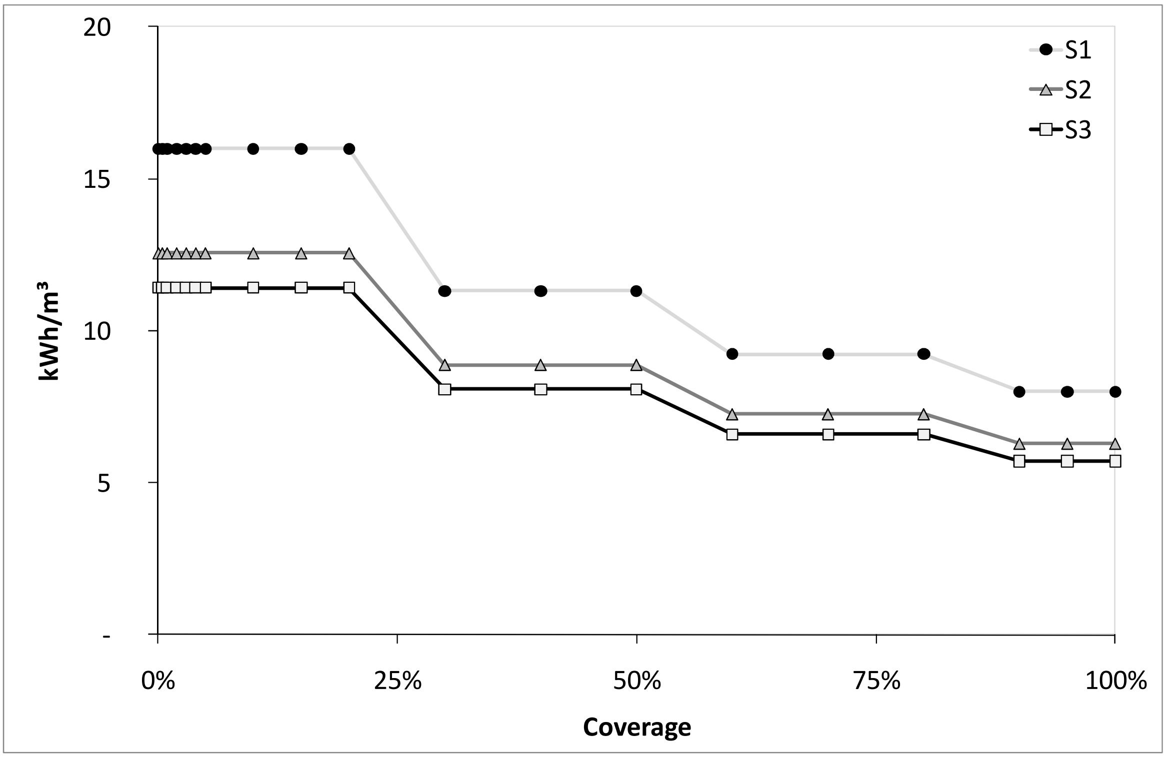

Figure 20: Energy consumption per transported volume

D. Treatment

D.1. Introduction

The aim of treating source separated urine is the safe and easily applicable discharge or even better use of it. The uncontrolled release of large quantities of nutrients contained in human urine into the environment could lead to eutrophication in surface water bodies and dead zones in maritime coastal regions, or after formation of urea to ammonia and nitrification to nitrate contamination of groundwater. The issue of micropollutants, such as pharmaceutical residues, detected more and more in the environment and released in large quantities via human urine, is still – to some extent controversially – discussed (*Winker et al. 2008a*; *Winker et al. 2008b*). Because of uncertainties about effects of pharmaceutical residues, methods for their removal have to be developed.

At the same time there is substantial demand for substances such as ammonia and phosphate e.g. for fertilizers in agriculture, as well as for raw materials in industry. Therefore the recovery of the contained nutrients is of high relevance for ‘closing the loop’ (*Otterpohl & Oldenburg 2007*; *Tettenborn et al. 2009*) and could be economically interesting for industrial purposes. In terms of nutrient recovery it is important to see the correlation of the nutrient content of the urine (its concentration) and its value: The higher the concentration and the smaller the volume containing this concentration, the lower are transport and storage costs for the same nutrient amount. Thus, any concentration process of source separated urine is highly appreciated, especially when applying urine separation in an urban context.

Many treatment options for nutrient recovery and for the removal of micropollutants have been investigated within the last ten years:

- Storage has been tested as a low tech process for sanitation purposes. Over time and at sufficient temperatures bacteria are inactivated (*Schönning et al. 2005*; *Vinnerås et al. 2008*) so the substrate would be safe for agricultural use.
- Precipitation of phosphate from urine is a very effective process for phosphorous removal from urine and recovery as valuable MAP-precipitate. Details were investigated by several institutions and published inter alia by *Ban & Dave* (*2004*), *Basakcilardan-Kabakci et al.* (*2007a*), *Ganrot et al.*. (*2007a*), *Kabdasli et al.* (*2006*), *Liu et al.* (*2008*), *Ronteltap et al.* (*2007*), *Tilley et al.* (*2008*), and *Tünay et al.* (*2009*).
- Also ammonia adsorption and ion exchange was investigated thoroughly by different research groups *Ban & Dave* (*2004*), *Beler-Baykal et al.* (*2004*), *Ganrot et al.* (*2007a*), and *Lind et al.* (*2000*).
- Air stripping with N-recovery as ammonium sulfate was investigated by *Bischof & Meuler* (*2004*) and *Basakcilardan-kabakci et al.* (*2007b*).
- More advanced processes such as freez concentration was investigated by *Gulyas et al.* (*2004*) and *Ganrot et al.* (*2007a*). Formation of IBDU was investigated by *Behrendt et al.* (*2002*). Nanofiltration and electodialysis were investigated by *Pronk et al.* (*2006a*; *2006b*).

- Electrochemical treatment of human urine for its storage and reuse as flush water was investigated by *Ikematsu et al.* (*2007*).

Maurer et al. (*2006*) presented an overview of the different treatment options categorized according to seven objectives (volume reduction, P-recovery, N-recovery, stabilisation, hygienisation, removal of micropollutants, and biological nutrient removal) and found that no single process could fulfill all objectives at once.

So far, most processes were investigated at lab-scale only, however, experiences e.g. from struvite precipitation in other media and stripping processes in industry can be applied to some extent to source separated urine. Many of the so far investigated processes lean towards small scale implementation in decentralized systems e.g. at household level to spare the issue of long-distance transportation of urine. However, it can be assumed that process efficiency for volume reduction and nutrient recovery can be increased with increasing size of the treatment plant e.g. in industrial style.

For industrial style nutrient recovery and volume reduction, the processes of rectification, evaporation, and precipitation of MAP are the most promising ones, based on the large scale experiences from treatment of other substances and research by other institutions.

D.2. Focus of investigation

Consequently two main processes for volume reduction and nutrient recovery were investigated at pilot scale to obtain detailed information about the potential applicability as an industrial style processes. Additionally complementary processes were used for product finishing.

In detail, rectification was used for N-recovery and evaporation for volume reduction and production of a nutrient concentrate. Both were investigated at pilot scale. Additionally crystallization for obtaining highly concentrated nutrient crystals and MAP-precipitation were investigated, as well as the effects of a combination of these with rectification and evaporation and with AOP-processes.

The effect of the above-mentioned thermal treatment methods on micropollutants such as pharmaceutical residues was investigated, as well as the removal efficiency of ozonation and UVC-irradiation on these substances. Since the required energy input of the lab-scale UVC-irradiation was by a factor 100 higher than the required energy input for ozonation, results of the UVC-irradiation are displayed in the appendix in chapter I.3.3 only.

The following processes were investigated sorted according to their objective:

- Nutrient recovery / volume reduction
    - rectification/steam stripping (pilot plant)
    - concentration by evaporation (pilot plant)
    - crystallization
    - precipitation
- Removal of pharmaceutical residues

    - thermal processes for nutrient recovery and volume reduction (effects on pharmaceutical residues)
    - ozonation

These processes were evaluated regarding their effectiveness regarding nutrient recovery, removal of micropollutants, and volume reduction. All were related to the energy demand.

The individual questions to be answered were:

- Rectification / steam stripping
    - design of a rectification/steam stripping plant in pilot scale with operational and achievable parameters;
    - technical feasibility of rectification/steam stripping with hydrolyzed urine as feed substrate at pilot scale (study of specific problems during pilot plant operation due to the specific substrate such as foaming, crystallization within the stripping column or within the heat exchanging unit);
    - NH_4-N reduction rate;
    - NH_3 recovery rate; and
    - energy demand.
- Concentration by evaporation
    - technical feasibility of evaporation with hydrolyzed urine as feed substrate in pilot scale (study of specific problems during pilot plant operation due to the specific substrate such as foaming, splashing etc.);
    - maximum concentration factors (investigation of occurrence of crystallization during the process or during storage of concentrates);
    - nutrient concentration rates;
    - effect of pH reduction of feed substrate;
    - concentration of N-depleted substrate; and
    - energy demand.
- Removal of micropollutants
    - effect of thermal processes such as rectification (steam stripping) or concentration by evaporation on pharmaceutical residues;
    - additional and subsequent treatment steps for removal of pharmaceutical residues such as ozonation of hydrolyzed urine and of N-depleted urine; and
    - energy demand.
- Evaluation of potential synergetic positive treatment combinations according to defined boundary conditions.

D.3. Material and methods

The process descriptions here are built around those described in *Tettenborn et al.* (*2007*). The two main processes, the rectification / steam stripping and the evaporation with subsequent crystallization are presented in more detail. Especially for discussion of the results of the rectification (steam stripping) process and of the scaling up potential of this process, a more thorough description of the internal details is necessary. Precipitation and processes for removal of micropollutants were subsequent steps, complementing the results of the two main processes for nutrient recovery on an industrial scale.

D.3.1. Steam stripping / rectification

Introduction

Steam stripping is an efficient desorption process where temperatures of the feed substrate are high, in cases where steam is available, at high concentrations of the feed substrate, and in cases where the product, e.g. the produced ammonia-solution can be further used or marketed (*Santelli & Bomio 1990*). Different from air stripping in the case of ammonia stripping strong acid is not necessarily needed for collection of the product. The produced ammonia vapour can be collected by condensing.

In a stripping process a liquid loaded with one ore more components is carried through by a gas. The gas is loaded with the transient components and the liquid becomes depleted of these.

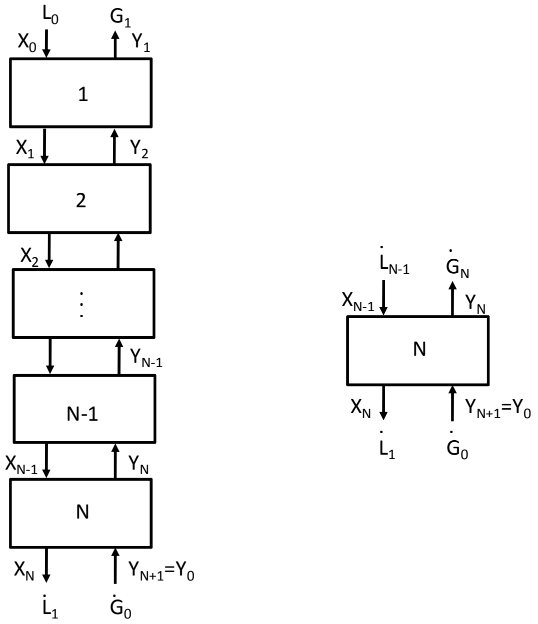

Figure 21: Scheme of stripping process

Eq 8: $$\dot{G}_0 Y_0 + \dot{L}_0 X_0 = \dot{G}_1 Y_1 + \dot{L}_1 X_N , \qquad \dot{G}_0 Y_{N+1} + \dot{L}_{N-1} X_{N-1} = \dot{G}_N Y_N + \dot{L}_1 X_N$$

$\dot{G}_0$:	molar input flux of a gas	$\dot{G}_1$:	molar output flux of a gas
Y_0:	initial loading of gas	Y_1:	final loading of gas
$\dot{L}_0$:	molar input flux of the liquid	$\dot{L}_1$:	molar output flux of the liquid
X_0:	initial loading of liquid	X_N:	loading of depleted liquid

For understanding the process and for designing a stripping plant the concept of theoretic separator stage can be used. The principle is shown in Figure 21. Each separator stage is defined as a stage where a thermodynamic equilibrium is reached between the streams leaving the stage.

The column is fed with a molar flux of the liquid ($\dot{L}_0$) with a loading (X_0) of a component and flown through counter-vise by the molar flux of a gas ($\dot{G}$) with the initial loading Y_0. After the process the depleted liquid has the loading X_N while the gas has a loading Y_1. By balancing the molar fluxes of in- and output streams the individual loadings can be derived (Eq 8).

Rectification versus steam stripping

The term *steam stripping* is often used when bubbling liquid substrate with steam. For processes yielding on nutrient recovery this is rather imprecise. In stripping processes a reduction of a volatile component of a substrate is objected. For nutrient recovery highly concentrated nutrient products are favored. Such can be achieved by rectification which is a combination of stripping and distillation in which the volatile component in the gaseous mixture is further separated by a fractionated condensation e.g. of the less volatile water.

Thermodynamics in rectification processes are similar to the ones in stripping processes. However, in rectification an additional concentration step of concentration in the off gas is included. The main difference between desorption (stripping) and rectification processes are conditions at the head and the bottom of the column. In a desorption column liquid input and gaseous output at the column head are not interacting while in a rectification column, part of the leaving gaseous flow is condensing and reentering (remaining in) the column similar to a distillation step. Likewise condensing steam at the bottom will dilute the liquid flow before leaving the reactor.

Both aspects (conditions at the head and bottom of the column) were not explicitly implemented in the design of the column. Focus was paid first to the technical realization of the desorption process using overheated steam.

Background

In stored urine nitrogen is present mainly as ammonium/ammonia. Ammonia (NH_3) is a readily soluble gas, not forming azeotropes with water, and can therefore be stripped well. In liquid solution it is in equilibrium with its iogenic form ammonium (NH_4^+). The equilibrium can be described by Eq 9.

Eq 9

$$NH_4^+ + OH^- \Leftrightarrow NH_3 + H_2O$$

The balance of molecular ammonia and dissociated ammonium is influenced by pH and temperature and can be described by Eq 10 (*Kollbach et al. 1996*). In Figure 22 the balance is plotted against pH for four different temperatures.

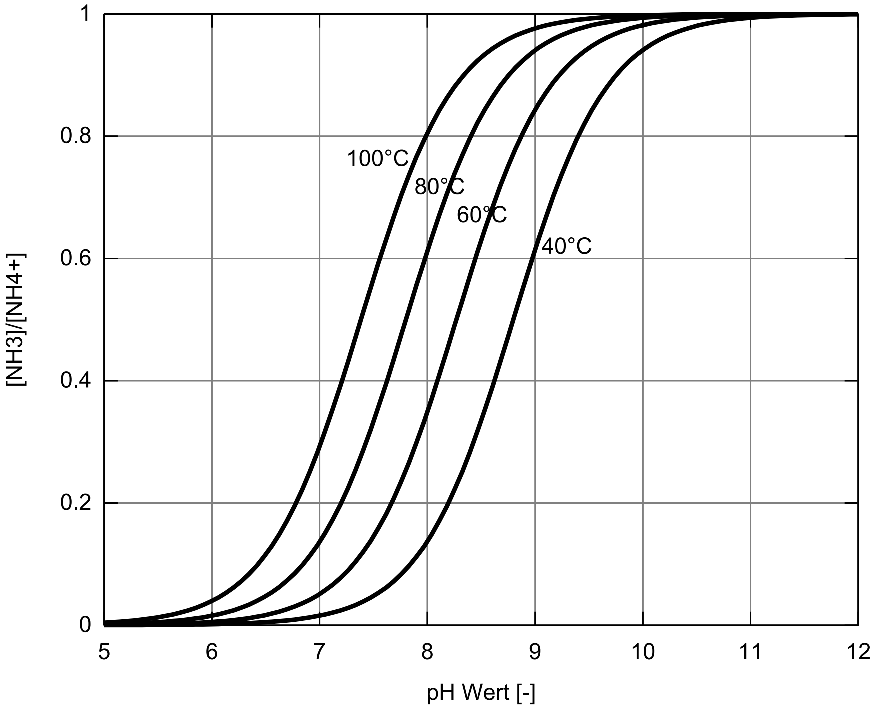

Figure 22: Dissociation equilibrium of ammonia/ammonium against pH for four different temperatures

Eq 10

$$\psi = \frac{[NH_3]}{[NH_4^+]} = \frac{10^{pH}}{\exp(6344/T[K]) + 10^{pH}}$$

ψ : ratio of dissolved ammonia and ammonium

T: temperatur

High temperatures and a high pH will shift the equilibrium towards molecular ammonia. E.g. at 100 °C and a pH of 9 a fraction of about 97.6 % is present as ammonia.

Additionally to the equilibrium between ammonium and ammonia in solution, there is equilibrium between ammonia in solution and ammonia in the surrounding gas phase:

Eq 11

$$NH_{3(aq)} \Leftrightarrow NH_{3(gas)}$$

The dynamic gas-liquid equilibrium is the equality of chemical potentials that determine the state of equilibrium for each set of conditions of temperature and pressure. For dilute concentrations of most gases, the equilibrium-distribution curves can be described by Henry’s law:

Eq 12

$$y = Hx \text{ or } \mathrm{Y_G} = H_C * X_L$$

x,y: mol fractions of compound A in liquid and gas phase respectively

H: Henry coefficient [gas mol fraction / liquid mol fraction]

X_L,Y_G: concentration of compound A in liquid and gas respectively [g/m^3]

H_c: Henry coefficient expressed in terms of volumetric concentrations [g/m^3 / g/m^3]

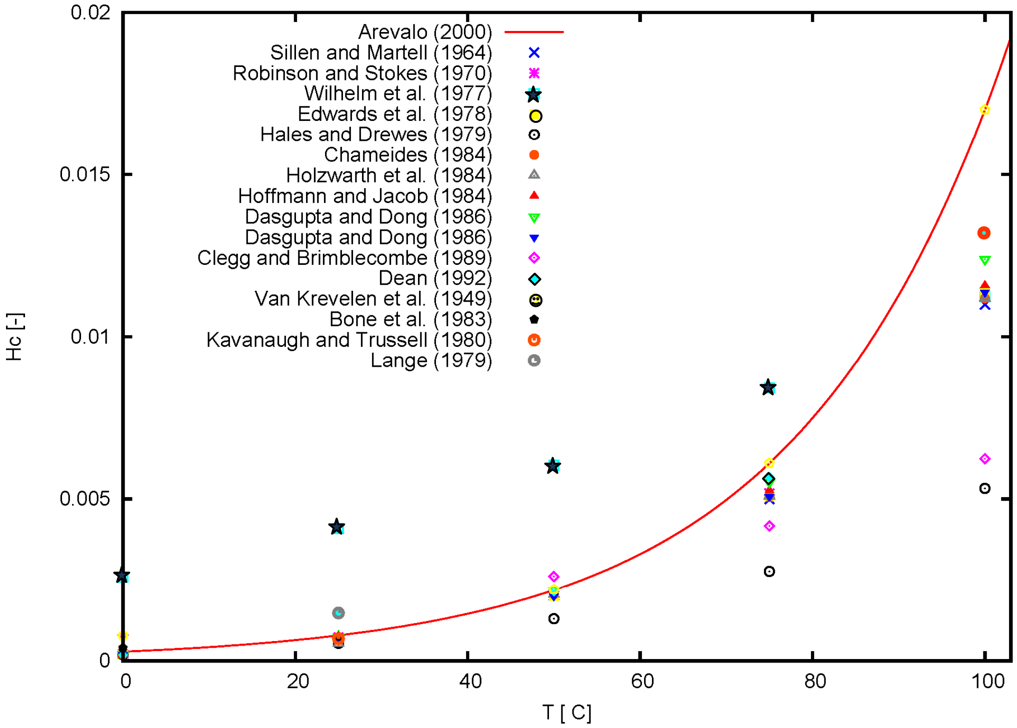

Figure 23: Henry coefficient for ammonia. Based on data from *Arevalo* (*2000*) and literature data listed in *Sander* (*1999*).

High values of H mean that at equilibrium a small concentration of A in the liquid phase will provide a larger concentration in the gas phase, meaning that compound A is volatile. Low values of H mean compound A is not very volatile but soluble in the liquid[8].

With lab experiments and model simulation the temperature dependency of the Henry coefficient for ammonia was derived by *Arevalo* (*2000*). Data are given in Appendix table 10 and Figure 23. For the temperature range between 10 and 100 the Henry coefficient for ammonia exhibited an exponential relationship.

The data of *Arevalo* (*2000*) were compared with literature data from *Sander* (*1999*) who lists Henry coefficients for ammonia with temperature dependency from 16 different literature sources. Henry coefficients (H_e [-]) range form $5.24*10^{-4}$ to $4.1*10^{-3}$ with a median of $7.05*10^{-4}$, all at atmospheric pressure and 25 °C. The data were combined with the individual temperature dependency listed to achieve data for 0 °C, 25 °C, 50 °C, 75 °C, and 100 °C all illustrated in Figure 23 (details see Appendix table 10). Up to temperatures of 50 °C the data of *Arevalo* (*2000*) match well with the collected literature data of *Sander* (*1999*). At higher

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[8] Attention has to be paid to whether the coefficient is representing solubility or volatility. Here H_c is used representing volatility (see appendix I.3.1)

temperatures the Henry coefficient derived by *Arevalo* (*2000*) is up to 40 % higher than the average of the collected literature values. However, the influence of temperature on the balance of dissolved ammonia and ammonium was not incorporated, which could explain the offset.

In the dissolved form, ammonia is forming hydrogen bonds leading to a relatively high boiling point and a relatively high evaporation enthalpy of about 23.4 kJ/mol. In comparison: water has an even higher evaporation enthalpy of about 41 kJ/mol.

Ammonia has a pK_b value of 4.76 and is therefore reacting with water as a strong base.

Design of a rectification plant in pilot scale

The rectification unit at pilot scale was designed to be able to treat the collected urine of all public waterless urinals in Hamburg, resulting in about 20 to 25 m³/month. Assuming 22 working days a month and 8 hours operation time per day, a volume of slightly more than 100 l/h should be treated. This capacity would also be suitable for a settlement of 500 to 1000 inhabitants, depending on operation time of the rectification unit.

The design parameters are presented in the following.

For the desorption process (the transfer from the dissolved into the gas phase) low pressure and high temperatures are favorable (*Mersmann et al. 2005*). Under optimum conditions this can be described by Eq 13, leading to the calculation of the minimum amount of required stripping gas, which is not depending on the concentration in the liquid phase but the flow. Thus, from equation Eq 13 the minimum gas flow can be derived, resulting in a minimum theoretical gas flow of 4.35 kg/h at 1 atm and a liquid feed of 100 l/h. The realistic demanded gas flow should be higher. For the design of the column a gas flow of 13 kg/h was assumed.

Eq 13:

$$\frac{\dot{G}}{\dot{L}} = H_c * p \rightarrow \dot{G}_{\min} = H_c * p * \dot{L}$$

$\dot{G}_{\min}$: minimum gas flux

H_c: Henry coefficient expressed in terms of volumetric concentrations

p: partial pressure

For further design the approach of HTU/NTU (*Kollbach & Grömping 1996*; *Mersmann et al. 2005*) based on the scheme in Figure 21 was used. In this approach the height of the column ‘h’ is derived from a specific number of transfer units ‘NTU’ (number of transfer units) which can be seen as the single separator stages with thermodynamic equilibrium between the streams leaving the stage in Figure 21. Since in each stage a specific amount of the volatile substance from the liquid phase is transferred into the gaseous phase, the number of transfer units is depending on initial and yield concentrations, the molar fluxes, the equilibrium curve, and the mass balance within the process.

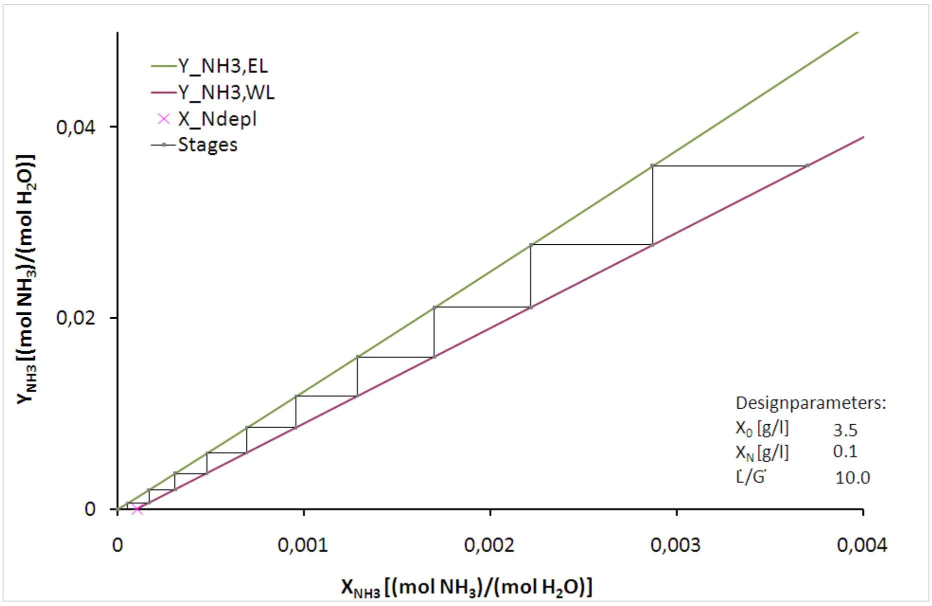

Figure 24: McCabe-Thiele diagram for high yielded output[9]

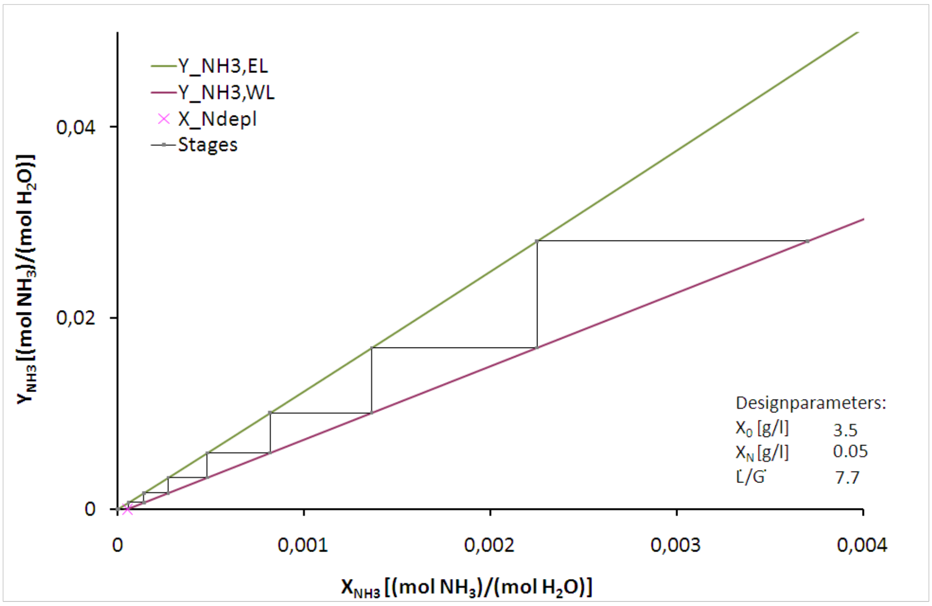

Figure 25: McCabe-Thiele diagram for regular operation[9]

For deriving the number of theoretical transfer units, an equilibrium line (EL in Figure 24) is plotted over a working line (WL in Figure 24), both over the concentration of the volatile substance in the liquid phase on the abscissa in a so called McCabe-Thiele-diagram. The working line describes the mass balance of the process and is here assumed to be linear. Beginning from the initial concentration (in Figure 24 in mol NH_3/mol H_2O) the construction of theoretic transfer units is started towards the equilibrium line. The point of intersection is

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[9] For plotting the McCabe-Thile diagram an excel-sheet was modified established as prerequisite of a simulation of the stripping process that was used by *Mojen (2009)* to evaluate the results gained from the pilot plant

describing the output concentrations of liquid and gaseous phase at the end of the first theoretic transfer unit which at the same time is the starting concentration (X_{NH3}) for the next transfer unit on the working line. This proceeds until the final concentration in the substrate is reached.

The initial concentration in the gas phase (Y_0) can be assumed to be zero. An initial substrate concentration X_0 of 3.5 g NH_3/l, a yielded end concentration X_N in the substrate of 0.1 g/l, a liquid flux of $\dot{L}$ = 100 kg/h and a gas flux of $\dot{G}$ = 10 kg/h would result in 10 stages (Figure 24). A higher $\dot{L}/\dot{G}$ would lead to even more stages and higher end concentrations Y_1 in the off gas could be reached, until the working line and equilibrium line become nearly parallel. A small change in yielded end concentration X_N in the substrate would not dramatically change the number of NTUs or the reachable concentration Y_1 in the off gas.

With the design parameters of $\dot{L}/\dot{G}$ ratio of 7.7, six theoretic transfer units would be needed (Figure 25).

The height of transfer units HTU can be derived from the planned fluxes, the specific inner surface area with a correction factor, the cross sectional area of the column, and the transfer coefficient:

Eq 14

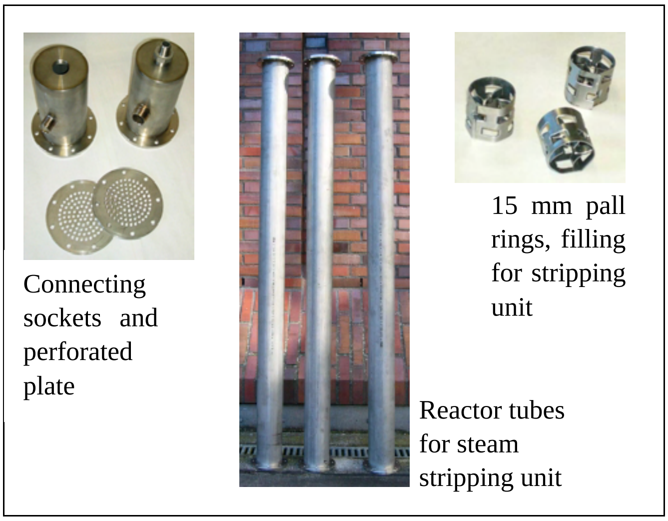
$$\mathbf{HTU} = \frac{\dot{G}}{k_g * \eta * a * A_Q}$$

HTU: height of transfer unit
$\dot{G}$: gas flux [mol/h]
k_g: transfer coefficient [m/h]
η: correction factor [-]
a: specific inner surface area [m^2/m^3]
A_Q: cross sectional area [m^2]

As filling material for reaction surface maximization, 15 mm metal pall rings with a surface of 360 m^2/m^3, and a free volume of 95 % were used. For the cross sectional area a diameter of 0.1 m was chosen. However, during the designing phase of the column sufficient and reliable data on feed concentration and possible fluxes were not available. Therefore assumptions were made based on estimated guesses for each unknown aspect. These combined with the known data and with the recommendation of *Kollbach & Grömping (1996),* who suggest 0.5 to 0.7 m as HTU in ammonia stripping processes, a 4.8 m high column was designed.

The stripping/rectification tower was manufactured from stainless steel and could be segmented into 5 parts: 1x connecting piece for urine input and steam exhaust, 1x socket for depleted urine outlet and steam intake, and 3 reactor tubes (Picture 1 to 3).

The column was insulated by mineral wool and protected from weather conditions by galvanized plates.



Connecting sockets and perforated plate

Reactor tubes for steam stripping unit

15 mm pall rings, filling for stripping unit

Picture 1, Picture 2, Picture 3: Parts of demo-size rectification reactor

Because of costs a commercially available plate heat exchanging unit with 37 panels and a surface area of 2.9 m^2 was used. As a condenser a single tube condenser was built, which was installed next to the rectification column. Hot steam from the head of the rectification column entered the condenser from top, while service water was used in a cross flow to cool down the steam. The condensate was captured at the bottom of the condenser. The heat exchanging surface was 0.47 m^2. Maximum flow of cooling service water was 300 l/h.

Electric energy, steam and process water was available at about 6 bar and a temperature of around 160 °C. Measuring of the steam quantity was conducted by volumetric measurement of the condensed steam, and fraction comparison of the in- and outlet streams.

Eq 15

$$\dot{G}_0 = \dot{L}_0 \frac{X_0 - X_N}{Y_1 - Y_0}$$

With a constant feed flow $\dot{L}_0$the demand of steam $\dot{G}_0$ can be expressed depending on the yielded loading X_N and Y_1 (Eq 15).

$$F = \omega_v \sqrt{\frac{\rho_v}{\rho_l - \rho_v}}$$

Eq 16

F: steam loading factor [$\sqrt{Pa}$]
ω_v: vapor velocity [$m/(m^2 s)$]
ρ_v: density vapor [kg/m^3]
ρ_l: density liquid [kg/m^3]

The maximum gas flow through the column is determined by the flooding factor of the column depending on free cross section available within the column for the gas stream. It can be determined by the steam loading factor F given in Eq 16. A steam column is normally operated at a flooding factor of about 60 %. Due to foaming substrates, the flooding factor can be even lower. For the pilot plant a maximum gas flux of 37 kg/h was calculated at a flooding factor of 60 %.

The theoretic output concentrations of the depleted liquid (X_N) and the enriched gas (Y_1) can now be calculated, depending mainly on input concentration (X_0), flux ratio of liquid ($\dot{L}$) and gas stream ($\dot{G}$) and also on the yielded output concentration X_N.

Picture 4: Demo scale rectification unit on the right side of the container. Feed substrate in 1 m^3 big-packs on the left side of container

Table 13: Maximum fluxes for the rectification column

Flooding factor	[%]	100	60
Liquid flux	[l/h]	100	100
Gas flux	[kg/h]	62.6	37.6

Theoretic yields

For comparison of results of ammonia concentration in the condensate achieved at different input parameters, equations Eq 8 and Eq 10 were combined achieving Eq 17, to achieve a theoretically achievable concentration in the condensate Y_t depending on input concentration of the feed substrate X_0, the yielded concentration in the depleted liquid (X_N), and on the $\dot{L}/\dot{G}$-ratio.

Eq 17

$$Y_t = Y_0 + \frac{\dot{L}}{\dot{G}}\left(\psi * X_0 - X_N\right)$$

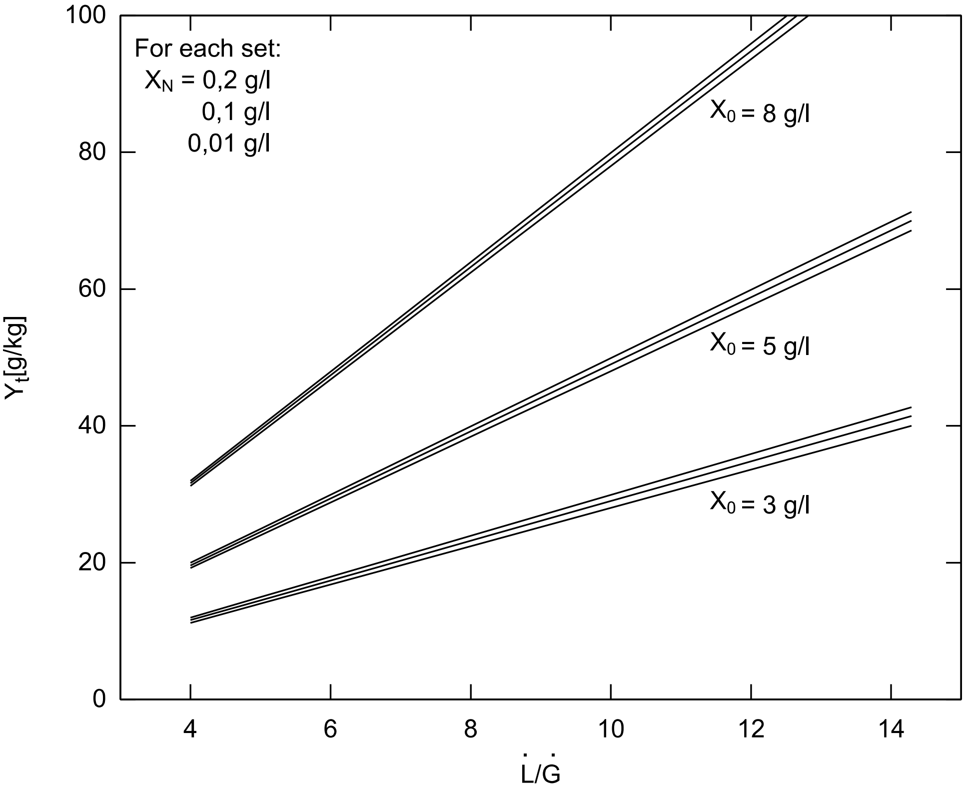


Figure 26: Maximum concentrations in off-gas (Y_t) depending on $\dot{L}/\dot{G}$-ratio (with an initial input concentration (X_0) of 3, 5, and 8 g/l), and yielded concentrations of the depleted liquid (X_N) of 0.01, 0.1, and 0.2 g/l

As can be seen in Figure 26, the largest influence of the theoretically reachable maximum concentrations in the off gas has the initial concentration of the liquid feed (X_0). The $\dot{L}/\dot{G}$-ratio is mainly depending on the steam flow. The smaller the steam flow, the higher concentrations can be reached in the off-gas. The theoretical minimum steam flow was calculated to be roughly 7 kg/h resulting at a liquid flow of 100 l/h in a $\dot{L}/\dot{G}$-ratio of 14 and a maximum concentration in the off-gas of about 50 g/kg steam at an initial feed concentration of 3.5 g/l. Realistic $\dot{L}/\dot{G}$-ratios would be lower. With a $\dot{L}/\dot{G}$-ratio of 12.5, and at a feed of 8 g/l about 100 g/kg steam could be reached theoretically in the off-gas.

A variation of the yielded concentrations X_N in the depleted liquid between 0.01 and 0.2 g/l is nearly not influencing the theoretically achievable concentration in the condensate Y_t.

Operation and varied parameters

Urine was pumped from the storage tank (feed container) through a flow meter and the heat exchanger into the column inlet at a height of 5.60 m. From the column sump the N-depleted urine flowed by gravity through the heat exchanging unit and a second flow meter into the collection tank. The filling height in the reactor was controlled by a visible tube outside the reactor connected to both the head and the sump of the column. The filling height could be adjusted by an overflow (variable in height) that was placed directly after the outlet flow meter.

Residence time within the rectification column was about 15 min.

Steam was introduced at the bottom of the column. The loaded steam was removed at the head of the column and send through a condenser. The condensate was captured at the bottom of the condenser in a 1 l measuring cylinder. Samples from the condensate were taken directly from the condenser. The energy from the condensing process was not integrated in the process. At the beginning of each set 0.5 l of a silicon based defoamer were added to the substrate. Temperature was measured on the outside of the steal cover of the rectification tower at the height of steam inlet, and at the height of the substrate inflow.

A total of more than 2 m^3 urine was processed in several sets in the rectification pilot plant while different parameters were varied. Volume flows, steam flow and feed pH were varied according to Table 14.

Table 14: Varied parameters during pilot plant rectification

	varied parameter
set 1	$\dot{L}/\dot{G}$
set 2	elevated pH of feed substrate, $\dot{L}/\dot{G}$
set 3	high $\dot{L}/\dot{G}$, analytics by external laboratory

During set 1, the ratio $\dot{L}/\dot{G}$ was varied between 2.4 and 3.6. In set 2, pH was elevated from pH 8.5 up to pH 11 by adding NaOH and KOH into the feed container. Because of heavy foaming problems, substrate flow had to be reduced drastically. In both cases ammonia analytics were performed using Dr. Lange test kits and stick tests. Within set 3 the ratio between $\dot{L}$ and $\dot{G}$ was varied between 3.5 and 4.1. The analysis of NH_3 was performed using the Büchi-distillation method. Additional process parameters can be found in Appendix table 13.

Samples were taken from the inflow in the feed container, from the N-depleted substrate directly from the effluent, and from the condensate directly from the condensate effluent. Volume and time were recorded at constant intervals, and the samples were analyzed for pH, TN, NH_4-N, and TP. Additionally spot samples were analyzed for SO_4, K and COD.

In total, a volume of 2.5 m^3 was processed in several sets. In each set the plant was operated after a heat-up phase of 30 to 45 min between 2 and 4 h. At times, more than one set was conducted in a row. Thus the maximum operation time of the plant was 8h/d. After reaching a stable flow of condensate, samples of condensate and N-depleted substrate were taken in intervals of 15 or 30 min. Values given below are generally average values.

The steam flux was derived from the volume of condensate collected per time and a flux balance involving in- and output fluxes (Eq 18).

Eq 18

$$\dot{L}_0 + \dot{G}_0 = \dot{G}_1 + \dot{L}_1$$

The amount of steam condensed within the reactor can be derived from Eq 19:

Eq 19

$$\dot{L}_1 - \dot{L}_0 = \dot{G}_0 - \dot{G}_1 = \dot{G} \text{ condensed within the reactor to } \dot{L}$$

leading to a dilution factor (df), expressed in Eq 20:

Eq 20

$$df = \frac{\dot{L}_0}{\dot{L}_1}$$

Prior to the operation at pilot scale, a lab scale plant was used to verify the calculated process parameters. Design and operation parameters as well as results are given in the appendix in chapter I.3.1.

D.3.2. Concentration by evaporation

Introduction

Evaporation can be used as a separation and concentration process. By evaporating an aqueous solution of inorganic salts with a low vapor pressure, the produced vapor will be nearly completely water vapor (however, small droplets containing small quantities of salts can be carried over by the vapor). For mixtures with components of similar vapor pressure, the produced vapor will contain all of these components (*Mersmann et al. 2005*). Figure 27 represents a simplified scheme of the evaporation process with subsequent condensation.

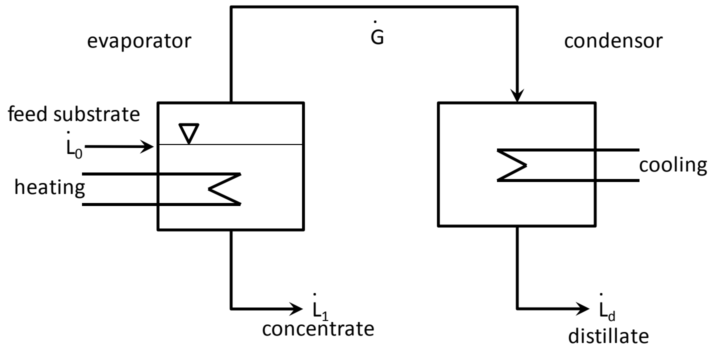


Figure 27: Scheme of evaporation process

Picture 5: Evaporation plant Provadest 'Mini', KMU

Setup

To demonstrate technical feasibility and to obtain larger quantities of concentrates for subsequent tests, an evaporation unit in demo-scale from the KMU Umweltschutz GmbH was operated in batch (Picture 5). With an evaporation rate of 4–10 l/h and temperatures between 70–80 °C at a pressure of -300 mbar, a substrate equivalent of 80 persons was processed. Distillate was constantly extracted from the system. The evaporation bulb was refilled automatically when the liquid level dropped below a defined level within the bulb.

Feed substrate was drawn into the evaporation bulb. The filling level of the evaporation bulb was sensor controlled, and the bulb itself was heated in an oil-bath. The saturated off gas was cooled and the produced distillate was collected. The volume that was extracted via distillate output was replaced automatically through the feed tank. Foaming and splashing within the evaporation bulb were controlled by automatic addition of a silicon based defoamer.

Eq 21

$$c_f = \frac{TS_1}{TS_0}$$

c_f: concentration factor
TS_1: TS in concentrate
TS_0: TS in feed substrate

The concentration factor is derived from the analysis of total solids (TS in Eq 21). In pre-tests the concentration factor derived from TS proved best to be equivalent to the concentration factor derived from volumetric balance. In the case of the demo-scale evaporation plant the large volumes did not allow a precise volumetric balance.

The theoretic and expected concentration of any substance (X_t) can be calculated by Eq 22.

Eq 22

$$X_t = c_f * X_0$$

X_t: expected concentration of substance X
X_0: initial concentration

Substrates, operation and varied parameters

During the evaporation two individual substrates were processed: stored urine and N-depleted urine. In several sets the pH of the stored urine was lowered using H_2SO_4 and H_3PO_4 to avoid excessive losses of nitrogen in the form of ammonia (Table 15) resulting in the input parameters given in Table 16.

Table 15: Parameters varied in the pilot scale evaporation plant

Substrate	urine N-depleted urine
pH	pH of feed urine was varied between pH 4.5 and pH 8.9 using H_2SO_4 and H_3PO_4

Table 16: Feed substrates for evaporation process (average values)

Feed substrate	pH	cond. [mS/cm]	TOC [mg/l]	TN [mg/l]	NH_4-N [mg/l]	PO_4-P [mg/l]	K [mg/l]	S [mg/l]	COD [mgO_2/l]	TS$^{(*}$ [g/l]
Stored urine	8.8	21		3 970		360	1198	354	4 120	7.8
Stored urine, pH7, H_3PO_4	7.3	23		4 180	3 640	1 140			4 300	8.0
Stored urine, pH4.7, H_2SO_4	4.7	24	2 190	3 150		408	1 360			30
N-depl. urine	6.7	11.5	75	210	120	299	930	298	1720	14.4

$^{*)}$ TS stands for total solids

D.3.3. Crystallization

Introduction

After observing crystallization processes within the concentrates produced at pilot scale evaporation, lab tests were preformed to investigate crystallization processes of concentrated urine. Concentration by atmospheric evaporation was conducted over a period of 20 days.

Background

Crystals are solid, homogeneous, anisotropic solids with a three-dimentional, periodic arrangement of their constituents. The constituents can be atoms, ions, or molecules (*Okrusch & Matthes 2005*). The process of crystallization is the transformation of one or more substances from an amorphous solid, liquid, or gas phase in a crystalline status (*Mersmann et al. 2005*). Crystallization from solutions is a thermal separation processes involving the formation of at least one solid crystalline phase and is mainly used for concentration or preparation in a pure state (*Sattler 2001*).

Process fundamentals such as the definition of a metastable state, required amount of nuclei, and the phenomenon of over saturation were first described by *Ostwald* (*1897*; *cited in Wilke & Bohm 1988*).

Solutions with molecular-disperse distribution of one or more molecules are often pre-concentrated by evaporation in several steps. The pre-concentrated solution is thereafter oversaturated by cooling or further evaporation (*Sattler 2001*). Other crystallization processes are based on vacuum evaporation or displacement/reaction. Crystal characteristics can be influenced by choosing the right combination of temperature, over-saturation, and additives during the crystallization process. The process of crystallization from an over-saturated solution involves the formation of nuclei and the growth of the crystal itself thereafter.

Setup

The substrate in the lab tests for crystallization was a 10 fold urine concentrate produced by thermal evaporation at 100 °C and atmospheric pressure. Nitrogen was lost during this step and pH decreased to pH 6. Initial concentrations of the solution are presented in Table 17.

To reach a stepwise over-saturation of the solution, the initial 10 fold concentrate was further concentrated in three sets under atmospheric conditions and room temperature by increase of surface area via a lab scale unit (Figure 28). Small amounts (0.12 mol) of calcium hydroxide and magnesium oxide were tested to initiate the crystallization process, in two of the three sets (Table 18).

Phosphate and ammonium in the concentrated solution were monitored during the stepwise concentration process.

Figure 28: Picture of lab scale unit for evaporation under atmospheric conditions (design and picture by Bursche 2008)

Table 17: Feed substrates for atmospheric evaporation process (average values)

	pH	cond. [mS/cm]	TN [g/l]	P [mg/l]	Ca [mg/l]	Mg [mg/l]	TS(* [g/l]
concentrated urine, 10.3 x	5.7	162	8.2	3.5	29	12	96

*) TS stands for total solids (not the German 'Trockensubstanz')

Table 18: Parameters varied in the pilot scale evaporation plant

Substrate	10.3 x concentrated urine
Initiation crystals	addition of initiation crystals in form of MgO and $Ca(OH)_2$

D.3.4. MAP-precipitation

Precipitation was also investigated at lab scale. Stored urine, nitrogen depleted urine, and concentrated urine were used as source. Different precipitants (MgO, $MgCl_2$, $CaCO_3$, $Ca(OH)_2$, $CaCl_2$) were used for precipitation. The precipitants and the precipitation processes in the different solutions were compared with regards to their phosphate removal efficiencies. Details are described in *Tettenborn et al.* (*2007*).

For crystallization and for precipitation an ernergy demand of 20 and 30 MJ/m³ is assumed for puming and dosage of precipitant.

D.3.5. Removal of pharmaceutical residues by thermal nutrient recovery processes

For seven from 21 analyzed pharmaceutical substances the behavior during urine treatment could be investigated. The seven substances for which concentrations could be measured were: ibuprofen, bezafibrate, ß-sitosterol, diclofenac, carbamazepine, phenacetin, pentoxifylline, and phenazone. Details on the initial concentrations in the source separated urine can be found in chapter B.4.2.

During the nutrient recovery processes rectification and evaporation, samples of the feed substrate and the end products were analyzed regarding the content of pharmaceutical residues to investigate the behavior of pharmaceutical residues in urine during processes involving heat over a defined time (Table 19).

Table 19: Process parameters

process	**time**	**temperature**
rectification	10–15 min	100 °C
evaporation	1–4 d	80 °C

D.3.6. Ozonation (removal of pharmaceutical residues)

Ozonation was tested as a second treatment option for the removal of pharmaceutical residues. Ozone is highly reactive. Materials/molecules that have chemical double bonds, or that contain oxidizable elements can be oxidized by ozone. The specific and direct reaction of ozone with organic substances is due to an electrophilic addition that leads to a splitting of the C=C double bonds (Crigée mechanism). Reaction rate constant is rather slow in the range of $k_D = 1.0–10^3\ M^{-1}s^{-1}$ (*Gottschalk et al. 2000*). During indirect reaction secondary oxidants, especially hydroxyl radicals (OH°), are formed by the decay of ozone. These react non-selectively and immediately ($k = 10^8–10^{10}\ M^{-1}s^{-1}$) with solutes.

Most pharmaceuticals have an aromatic molecular structure. These aromatic rings can be attacked by ozone according to the Crigee-mechanism (*Huber et al. 2003*).

During the experiment, substrate was percolated with ozone produced by an ozone-generator from Sander. The ozone-uptake was derived from the difference between O_3 in-put ($\dot{G}_0$) and O_3 out-put ($\dot{G}_1$), measured by ozone-analyzers.

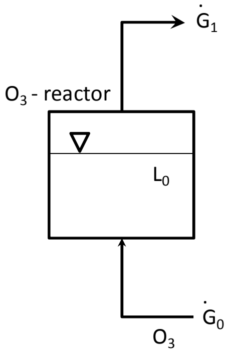


Figure 29: Scheme of ozonation

Table 20: Input parameter of ozonation

Urine

pH	dosage * [g O_3/L]
8.9	1.0
8.9	1.6
8.8	0.6
8.8	4.1
8.7	0.8
8.7	5.9

N-depleted urine

pH	dosage * [g O_3/L]
7.9	0.7
7.9	4.8

* dosage is representing the consumed amount of ozone

Stored urine and N-depleted urine at different pH values were ozonated. The gas flow was 10 l/h, and the initial volume of the substrate was 600 ml. Samples were taken after 1, 3, 6, and 24 hours. Therefore the consumed ozone dosages (ozone-uptake) varied depending on the ozonated substrate. Results are given in respect to consumed ozone dose (Table 20).

Again, the dosages used were selected according to prior experiments for removal of urochromes from urine (*Tettenborn et al. 2006*).

D.3.7. Substrate

During the investigation of the treatment processes, source separated urine was used as well as processed urine from the semi-technical plants. The source separated urine was collected within the SCST-project from two different locations:

- public toilets Hamburg (L3, see chapter B.3.1), and
- separation system at Stahnsdorf Berlin (for details see appendix I.1.1).

As discussed in chapter B.5, concentrations differed from literature values (Table 21).

The ammonia-depleted urine from the rectification which is also referred to as N-depleted urine was further processed by evaporation, crystallization, precipitation and ozonation.

The concentrates from the evaporation step were further treated by crystallization (see chapter D.3.3).

Table 21: Substrate parameters

	X_0 (1)(2)
TN [mg/l]	3 500–4 200
TP [mg/l]	210–410
COD [mg O_2/l]	2 600–5 350
TS [g/l]	20–30
pH	8.8–9.0

(1) Waterless public urinals, Hansaplatz, Hamburg, May 2005

(2) Separating System WWTP Stahnsdorf, Berlin, Nov 2005

D.3.8. Analytics

Main parameters

The samples were analyzed in respect to pH, conductivity, ammonium nitrogen and phosphate. A detailed characterization of the analytical procedure can be found in the appendix in chapter I.1.2.

Further analytics

Additional analyitics were conducted towards total carbon, potassium, calcium, sodium, sulfate-, and chloride ions. Details can be found in the appendix in chapter I.1.2.

Pharmaceutical residues

All analyses regarding pharmaceutical residues were conducted by IWW (Rheinischwestfälisches Institut für Wasser Beratungs- und Entwicklungsgesellschaft mbH) using HPLCMS (Butzen et al. 2005). Levels of detection in urine matrix and in aqueous solutions such as the condensates of the rectification process or the distillates from evaporation processes are given in Appendix table 2.

Statistics

Runaway values were detected according to *Grubbs (1969)* and (*NORM 1985*) and removed if runaway was proven.

D.4. Results and discussion

D.4.1. Rectification / ammonia stripping at pilot scale

Ammonia could be depleted from the original substrate very well. The ammonia concentration in the depleted substrate was in nearly all cases less than at least 10 % of the original value. As long as the initial pH was not adjusted, the pH of the depleted substrate was decreased by steam stripping from nearly pH 9 to below pH 6, which makes the process very expedient e.g. prior to the evaporation where low pH values reduce losses of the remaining ammonia. Ammonia concentrations in the condensate were between 15 and 34 times higher than in the initial substrate. Thus ammonia solutions of nearly 13 % (~120 g NH_3/l) could be reached (Table 22 and Appendix table 14).

Since different parameters influence the yielded ammonia concentration (see e.g. Figure 26), the yields from the pilot plant are plotted over the expected concentration in Figure 30. Since all relevant input parameters are integrated in the calculated theoretical yield (the expected concentration Y_t) different operational sets can be displayed in one plot. Due to a wide range of input parameters, data are displayed on a double logarithmic scale. The bisecting line $Y_1=Y_t$ indicates where measured data from operation would match the expected concentrations. Data above the bisecting line indicate higher concentrations from operation than expected, data below the bisecting line indicate lower concentrations from operation than expected. The trend of the measured concentrations is displayed as a well as the 80 % deviation of the trend.

Ammonia concentrations in the collected condensate Y_1 were on average 13 % higher than expected from theoretical calculations (see comparison between Y_1 and Y_t in Appendix table 14) with a standard deviation of 0.24 and a R^2 of 0.915. Variations in pH did not affect the results significantly since the influence was too small in respect to the variation of $\dot{L} / \dot{G}$ ratio.

The high concentrations are due to an occurrence of fractionated condensation in the head of the column which was not insulated. Since a large part of the sets were conducted in winter time, temperatures at the head of the column were significantly below 100 °C, probably in a range of the feed temperature which was normally around 75 °C or below. At this temperature, water in the gas phase is already condensating within the head of the column before entering the steam condenser while ammonia is still in its gaseous phase. In Figure 31 the potential ammonia concentration in the gas phase is plotted over the temperature depending on pressure; e.g. at atmospheric pressure and a temperature of about 70 °C, a 10 % ammonia solution can be reached, equivalent to 100 g NH_3/l. For a 20 % ammonia solution a temperature of 40 °C at atmospheric pressure would be needed at the head of the column. Another consequence of this fractionated condensation is the result of a smaller gas output than the minimum stripping gas requirement, changing the stripping process into a rectification process as described in chapter D.3.1.

The discrepancy between yielded concentration and expected concentration is probably due to fluctuation during operation. Although operation was stable, condensate was not produced constantly, leading to further concentration processes of the condensate in the head of the column.

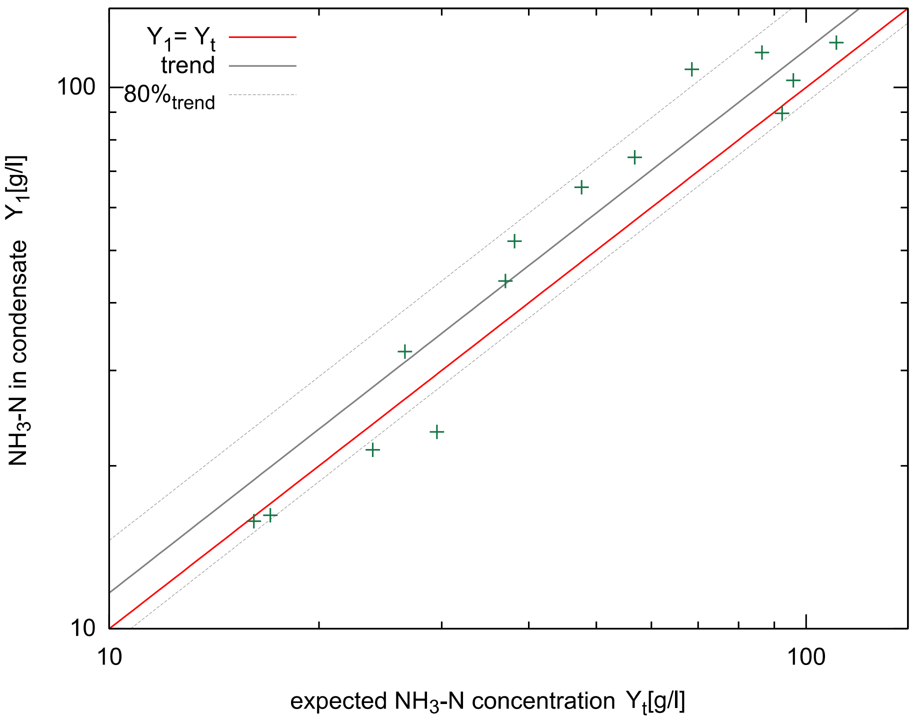
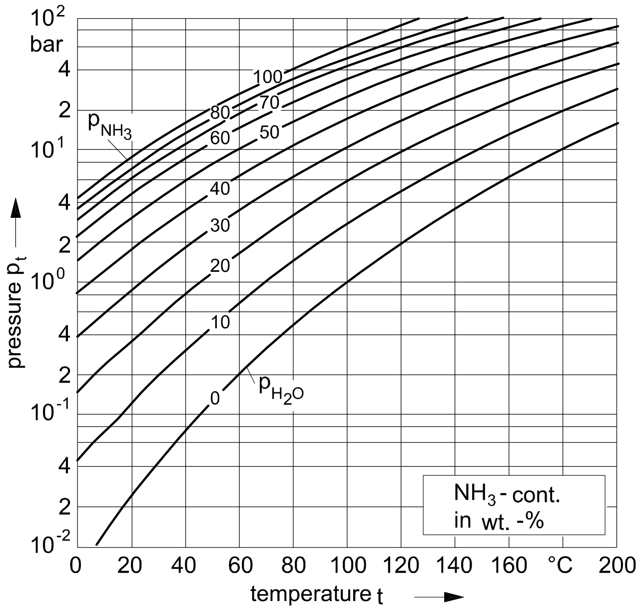


Figure 30: Yielded ammonia concentrations in condensate (Y_1) over expected concentration (Y_t) in pilot scale plant



Source: *Mersmann et al.* (*2005*)

Figure 31: Boiling point of ammonia/water solution.

The water condensing within the column leads to a dilution of the liquid flux and thus to lower concentrations X_N. The dilution factor is calculated according Eq 20 and is based on the in- and output fluxes presented in Appendix table 14. This has to be added to concentration values in the depleted substrate (X_N) which was between 0.1 and 0.25 g/l. This is considered in the calculation for the theoretically attainable NH_3 concentrations in Appendix table 14 and in Figure 30.

Table 22: Results of rectification yielding high concentrated ammonia solution

	X_0	$Y_{0\text{ (steam)}}$	$X_{1\text{ (N-depl)}}{}^{(*}$	$Y_{1\text{ (cond)}}$
Q [l/h]	81–98	23–25	102–120	2.2–3.5
N-NH_3 [g/l]	2.1–3.6	0	0.08–0.33	108–121
TP [%]	100 %	0 %	100 %	0 %
pH	8.8–9	-	5.4–5.5	9.5–9.6
mico-pollutants*	100 %	0 %	~ 70 %	0 %

*) dilution factor included in concentrations

In the condensate no phosphorous could be detected (see Table 22 and Appendix table 15). The reduction of the concentration in the N-depleted substrate was nearly completely compensated by integrating the dilution factor. Therefore, it can be concluded that no precipitation occurred within the stripping/rectification column. This was proved by visual observation of the filling material as well as of substrate pipes.

Also potassium and sulfur were detected nearly completely in the depleted substrate. Reduction was more or less equivalent to the dilution factor (see Appendix table 16).

Energy

During the lasts sets of the demo-scale rectification unit about 25 kg steam at 160 °C were used per 100 l substrate. This would be equivalent to an energy demand of 680 MJ/m^3 (188 kWh / m^3 processed substrate). From this energy the main part was used for heating up the substrate in the column. Only about 1/6 of that energy was used for the stripping of ammonia itself. An energy potential of 65 MJ/m^3 resulting from the condensing and cooling down of the condensate was not recovered. The rectification plant in this project was semi-technical size and was not designed towards energy efficiency.

Thus, major energy savings can be obtained by improvements in heat insulation of the column, heat exchanger and pipes and by energy recovery. With proper insulation, energy recovery, and slight changes in process control such as a fractionated condensation, it should be possible to reach literature values of 150 -200 MJ/m^3 (40–55 kWh/m^3).

D.4.2. Concentration by evaporation

For an overview of the concentration results in the concentrates of the three different sets, the measured concentrations of phosphorous, nitrogen, potassium and sulfur are plotted over the expected concentrations according to Eq 22 in Figure 32 and Figure 33.

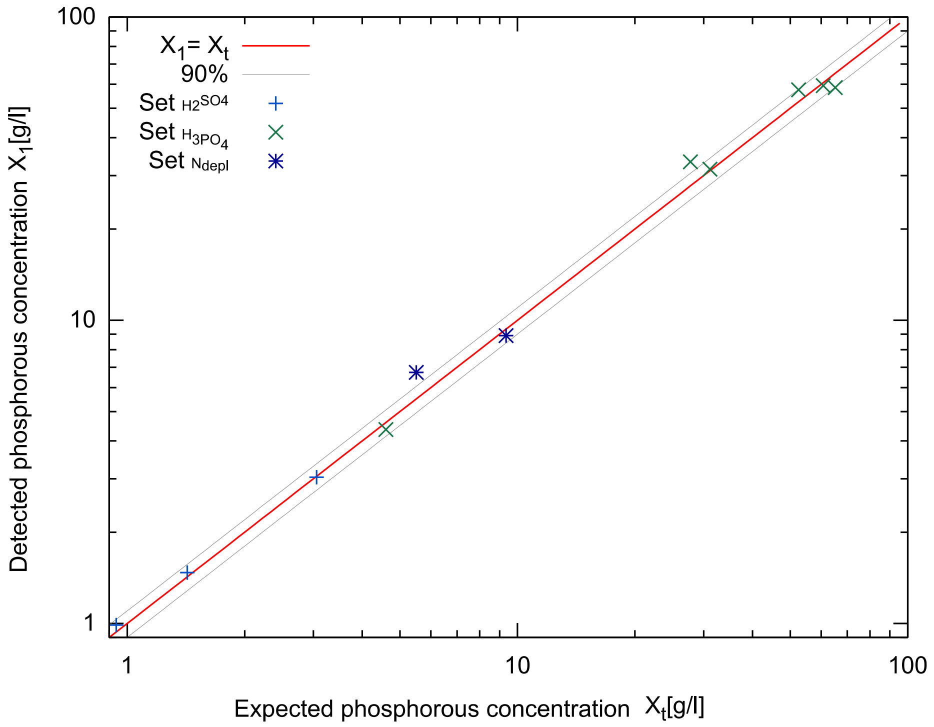


Figure 32: Detected phosphorous concentration in concentrate solution (X_1) over expected phosphorous concentration (X_t) during evaporation process in pilot scale during three different sets.

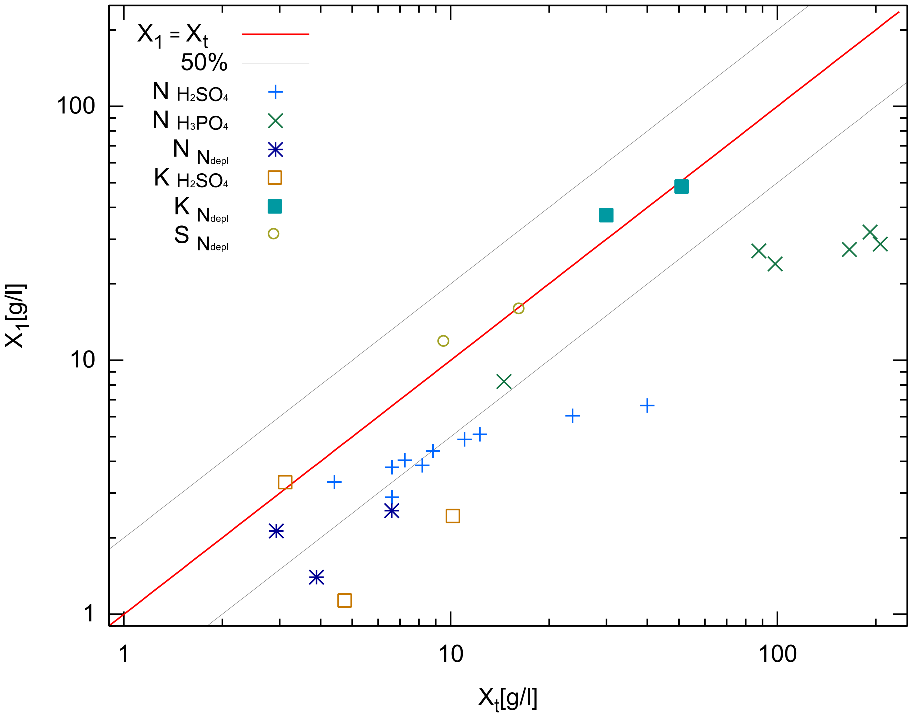


Figure 33: Detected concentrations (X_1) of ammonia (N), potassium (K), and sulfur (S) in concentrate solution over expected concentration (X_t) during evaporation process in pilot scale

Again, due to the wide range of concentrations a double logarithmic scale is used, the bisecting line $X_1=X_t$ indicating where measured data from operation would match the expected concentrations. Data above the bisecting line indicate higher concentrations from evaporation than expected, whereas data below the bisecting line indicate lower concentrations from operation than expected.

As can be seen in Figure 32 andAppendix table 21 the phosphorous concentrations in all three sets were in the range of the expected concentrations. No losses and precipitation did occur up to concentrations of 60 g P/l.

Parameters displayed in Figure 33 are discussed in the chapters regarding the individual sets.

Evaporation of stored urine acidified with H_2SO_4

About 300 liters of urine were acidified by adding 4 ml of 96 % H_2SO_4/l urine. The theoretical concentration factor c_f was derived by measuring total solids (Appendix table 17). Phosphorous and sulfur were captured in the concentrate in the expected concentrations, while losses of nitrogen and potassium did occur (Appendix table 18, Figure 32, and Figure 33). Losses of nitrogen can be explained by ammonia evaporation while lower potassium values than expected could be due to the addition of sulfuric acid and the concequent formation of potassium sulfate. However, formation of crystals was not visually observed in this step. Metals relevant for crystal-formation were detected in the expected concentrations in the concentrates (see Appendix table 19).

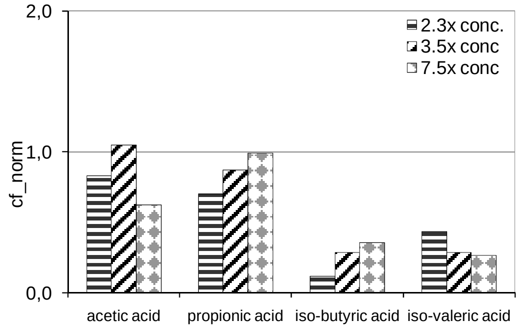


Figure 34: Organic acids after three different concentration steps by evaporation

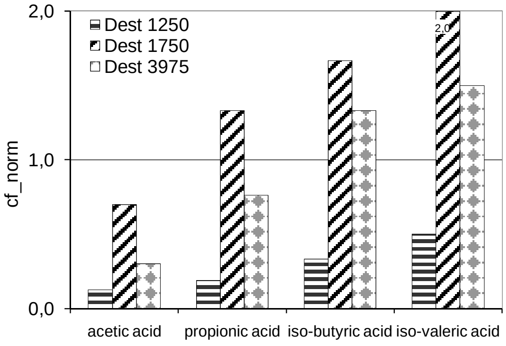


Figure 35: Organic acids in distillates after three different concentration steps by evaporation

The samples of feed substrate, three concentrates, and three distillates were also analyzed regarding carbon acids (Appendix table 20). Hexanoic acid and n-valeric acid were not detected in any sample. While the boiling point of the organic acids is increasing with their complexity (from acetic acid: 118 °C to valeric acid: 187 °C), the concentration factor of the latter ones is lower in the concentrates and higher in the distillates (Figure 34 Figure 35) leading to significant concentrations of TOC in condensates of stripping and rectification (Appendix table 21) and in distillates of the evaporation process.

Evaporation of stored urine acidified with H_3PO_4

Since sulfuric acid causes surface corrosion on non high-grade steel elements within the evaporation plant, 3 ml of 85 % H_3PO_4/l urine were added to yield the same pH-lowering potential as 4 ml of 96 % sulfuric acid.

Nearly 1 m^3 was concentrated by evaporation down to a concentrate of 17 l which is equivalent to a concentration of 57 times. Because of the large volumes and sequential feeding, the average pH of feed substrate varied around pH 7.3 causing losses of ammonia into the distillate.

By the addition of H_3PO_4 into the urine, the phosphorous content of the feed substrate more than tripled from initially 320 mg P/l to 1150 mg P/l.

From Appendix table 21 and Figure 32 it can be seen that all the expected phosphorous was found in the concentrates, while the distillate was nearly free of phosphorous. Again losses of nitrogen can be explained by ammonia evaporation (Figure 33).

Evaporation of N-depleted urine after rectification

More than 900 l N-depleted urine from the rectification process were concentrated by evaporation yielding in 17 l of 55 fold concentrate.

The results of phosphorous, potassium and sulfur presented in Appendix table 22, Figure 32, and Figure 33 lead to the conclusion that the measurement of dried solid content was not precise, since the normalized concentrations of P, K, and S have all the same small off-set. It can be assumed, that P, K, and S are completely captured in the concentrates.

Again, in this evaporation step, extraction of ammonia into the distillate occurred.

Boiling point

Behavior of foaming and splashing was investigated. In the small lab-scale RotaVap evaporator the phenomenon could be observed visually. Foaming occurs normally only with fresh substrate, especially with stored urine. N-depleted urine from the rectification process showed drastically less foaming. While foaming will still occur, even after a while at high temperatures, the phenomenon of splashing is even more severe. Because of the very diverse matrix of urine, splashing happens even at high agitation as occured in the RotaVap. Foaming could be controlled in most cases by addition of small fractions of oil or even better by addition of a silicone based defoamer.

The change of boiling point was also observed under atmospheric pressure. The higher concentrated a concentrate is, the higher the boiling point. At the same time the temperature within the concentrate is still increasing leading to constant boiling retardation (delay of boiling point). This leads to heavy splashing which can cause system failures. In the case of the demonstration plant, splashing of the concentrate led to several shut downs of the plant, since level sensors were affected. In Figure 36 it can be seen that the N-depleted substrate and the distillate of the evaporation process show a very similar boiling behavior to water.

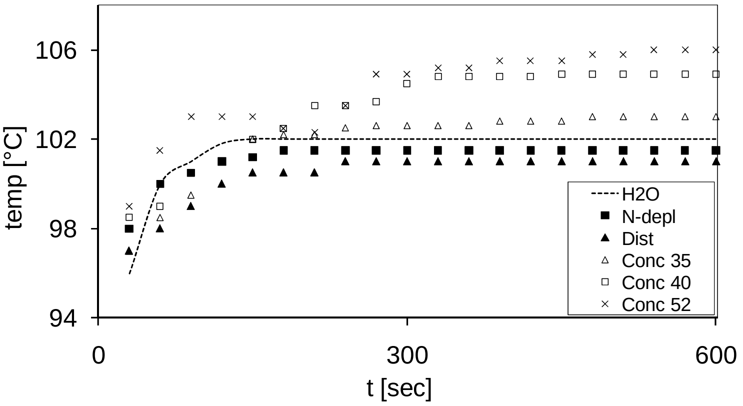


Figure 36: Determination of boiling point of concentrates, distillates, N-depleted substrate compared to H_2O

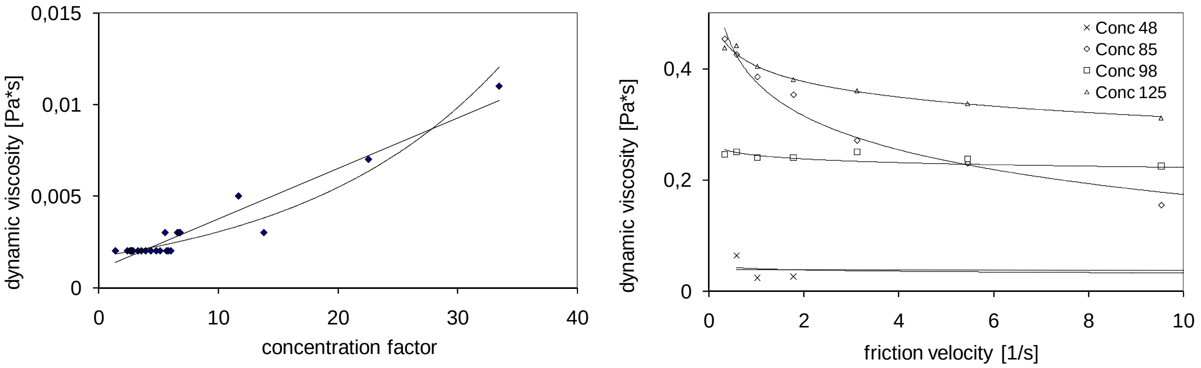


Figure 37: Dynamic viscosity (η) of different concentrates behaving as Newton liquids with exponential and linear trend

Figure 38: Dynamic viscosity (η) of high concentrated concentrates over friction velocity with logarithmic trends showing thixotropic behavior

Viscosity

The concentrates obtained in the above-described sets all behaved like Newton liquids and were not depending on friction velocity. However, there was an increase in viscosity with increasing concentration, as can be seen in Figure 37. Even at concentrations of about 50 fold the viscosity was less than e.g. olive oil ($\eta_{\text{olive oil}}$ = 0.084 Pa*s).

For more detailed information a part of the concentrate obtained from the demo-size evaporation plant was further concentrated in a laboratory evaporator (RotaVap). Thus concentrations of up to 125 fold were obtained which resembled a thick sludge.

With increasing friction velocity G, the dynamic viscosity decreased (see Figure 38) which is described as thixotropic behavior, allowing processing by pumping and mixing without excessive energy input.

Energy

The evaporation process is energy consumptive. The small evaporation unit for testing purposes was operated to process urine and N-depleted urine at demo-scale. Since it was electrically heated and not energetically optimized, it had an energy demand of about

2 200 MJ/m^3 (611 kWh/m^3). Efficient evaporation processes are available, such as those used in large-scale desalination plants with vapor compression distillation (VCD) and specific energy requirements of as low as 150–180 MJ/m^3 (*Maurer et al. 2006*). Industrial evaporation plants e.g. in the food industry with evaporation rates of 14–250 t/h have energy requirements ranging from 10 to 36 kWh/t (35–130 MJ/m^3)(*GIG 2011*; *Hansen 2011*).

However, because of the high salt content and the extremely diverse matrix of urine, processes such as thin film evaporation might be prone to failure caused by a break in the film leading to precipitation and thus to incrustation. In the evaporation unit Prowadest Mini, precipitation or incrustation was observed neither within the bulb, nor in pipes. This is explained by the high temperatures within the bulb and of the concentrate, preventing the formation of stable crystals.

D.4.3. Crystallization

In all three sets a reduction (difference from the expected concentrations) of phosphate and ammonia was observed starting at a concentration between 7.2 to 10 g PO_4-P / l and between 19.7 to 22.2 g TK-N/l. Figure 39 shows the deviation of measured PO_4-P concentrations (X_1) from the expected PO_4-P concentrations (X_t). The starting point of the deviation was equivalent to a concentration factor of 30 times (see Figure 40). An over-saturation can be assumed at this concentration. However, nitrogen and phosphorous concentrations did not remain constant during the ongoing concentration process, and a decline in dissolved concentrations was observed. In each set crystal formation was observed after exceeding the over-saturation.

In the two sets spiked with 0.12 mol of MgO respectively $Ca(OH)_2$ measured PO_4-P and NH_4-N values were slightly lower than the expected ones right from the beginning.

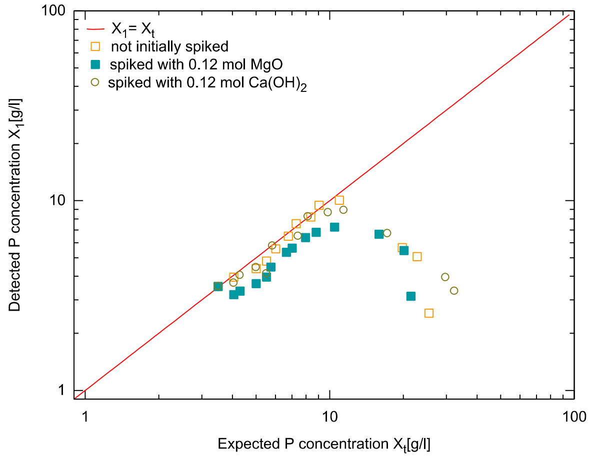


Figure 39: Detected phosphorous concentration in concentrate over expected phosphorous concentration during atmospheric evaporation process.

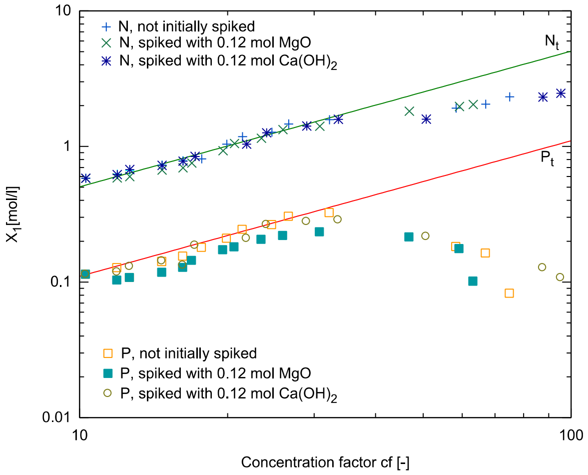


Figure 40: Detected nitrogen and phosphorous concentrations over concentration factor during atmospheric evaporation process.

Magnesium concentrations showed a slight increase from 12 to up to 59 mg Mg/l over the concentration process. Calcium concentrations varied between 20 and 41 mg Ca/l without showing any trend. In the set spiked with 0.12 mol of MgO, the magnesium concentration remained stable between 100 and 150 mg/l. The deviation of measured PO_4-P and expected PO_4-P correlates with the MgO-dosage at low concentration factors. In the set spiked with 0.12 mol of $Ca(OH)_2$, the calcium concentration increased from 70 mg/l (concentration factor 10) to 370 mg/l with a concentration factor of 95.

pH showed in all three sets only a slight increase from pH 6.0 to 6.3 over the whole concentration process.

The decline of the detected phosphorous concentration in the concentrate solution is due to crystal formation that was already observed during storage of the concentrates of the evaporation process at pilot scale.

Eq 23 $$\Delta_P\left(c_f\right) = X_{tP}(c_f) - X_{1P}(c_f)$$

Eq 24 $$\Delta_N\left(c_f\right) = X_{tN}(c_f) - X_{1N}(c_f)$$

Eq 25 $$c_f\Big|_{10}^{30}: \Delta_N\left(c_f\right) \approx \Delta_P\left(c_f\right) \approx 0$$

Eq 26 $$c_f\Big|_{35}^{99}: \Delta_N\left(c_f\right) \approx 2 * \Delta_P\left(c_f\right)$$

Δ_P, Δ_N:	difference of detected concentration to expected concentration of P and N
X_t:	expected concentration
X_1:	detected concentration
c_f:	concentration factor

Starting at a concentration factor of 30, the detected phosphorous concentrations and detected nitrogen concentrations were significantly lower than expected. The ratio of the molar delta at nitrogen was two times higher than for phosphorous (see Figure 41), meaning that for each mole of phosphorous that could not be detected in the concentrate solution, the concentrate solution lost two moles of nitrogen. This leads to the assumption that ammonium-phosphate was formed.

Ammonium phosphate can be formed in several ways resulting in e.g. sodium ammonium phosphate, diammoniumhydrogenphosphate, and/or triammonium orthophosphate trihydrate.

After storage of the concentrates of the pilot scale evaporation, several ammonium phosphate species were detected by X-ray-diffractometry, including sodium ammonium hydrogen phosphate hydrate $Na(NH_4)HPO_4 \cdot 4H_2O$ (stercorit), potassium ammonium phosphate ($0.73\ NH_4H_2PO_4 \cdot 0.27\ KH_2PO_4$), and sodium ammonium sulfate NH_4NaSO_4.

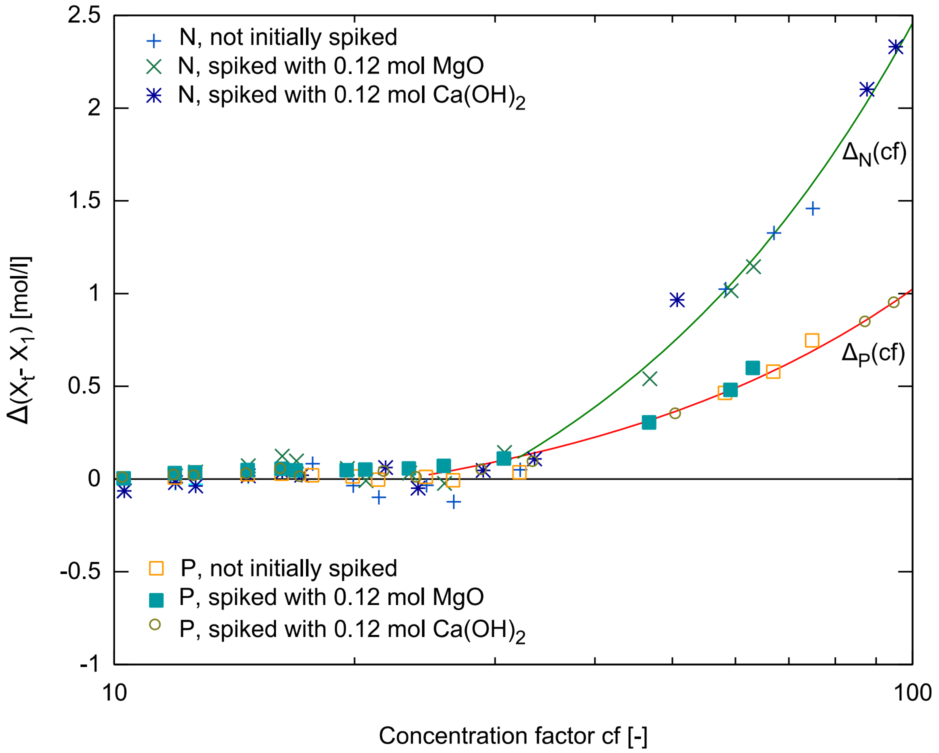
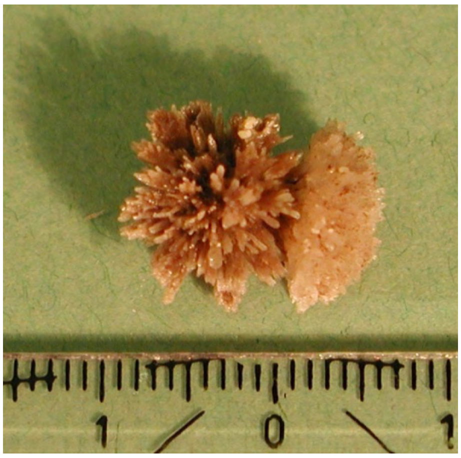


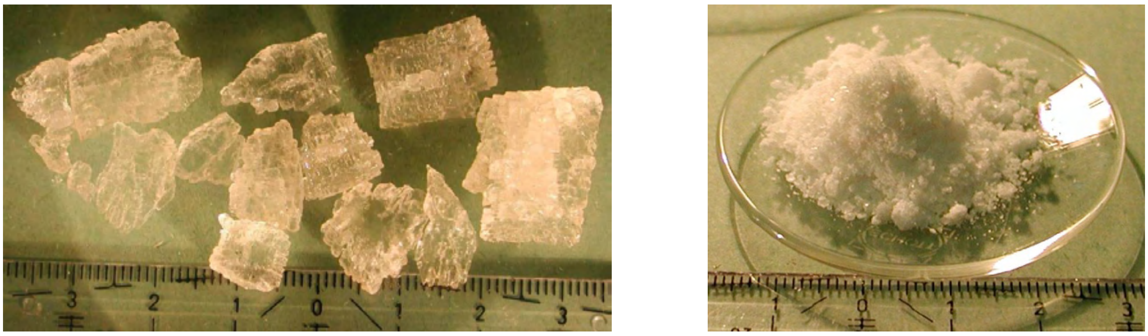
Figure 41: Delta of measured mol-concentration (X_1) minus expected concentration (X_t) for phosphorous and nitrogen. Starting from a concentration factor of 30, the slope of the delta curve for N is twice as high as the one for P.

Bursche (2008)

Picture 6: Picture of crystals from lab tests



Picture 7: Potassium Ammonium Phosphate ($0.73\ NH_4H_2PO_4 \cdot 0.27\ KH_2PO_4$)



Picture 8: Stercorit ($H(NH_4)Na(PO_4) \cdot 4H_2O$) washed and grinded crystals from concentrated urine

From a total of 105 l of urine with a dry mass of 990 g TS (1 %) a total of 670 g could be extracted in the form of crystals. About 90 % of the phosphorous was recovered by crystallization during the tests. Since the set up was a rather simple lab set up, optimization of the crystallization procedure should allow even higher recovery rates.

The different obtained crystals are displayed in Picture 6 to 8.

D.4.4. Effect of thermal processes on removal of pharmaceuticals

Lab scale steam stripping

In a lab scale stripping unit the behavior of pharmaceutical residues regarding operation temperature was investigated. Design and operation parameters are given in the appendix in chapter I.3.1.

Table 23: Average initial concentration (X_0) of pharmaceutical residues and concentrations in N-depleted substrate (X_1) and condensate (Y_1) at lab scale

values in µg/l (resp. % of X_0)	X_0	X_1		Y_1	
Ibuprofen	411	296	*(72 %)*	3.70	*(0.9 %)*
Bezafibrate	202	166	*(82 %)*	1.01	*(0.5 %)*
β-Sitosterol	31	25	*(82 %)*	-	*(nc)*
Diclofenac	26	17	*(67 %)*	0.70	*(2.7 %)*
Phenacetin	23	8.1	*(35 %)*	0.25	*(1.1 %)*
Carbamazepine	23	11	*(48 %)*	0.28	*(1.2 %)*
Pentoxifylline	8	-	*(nc)*	-	*(nc)*

In two different sets one sample of the feed substrate and each two samples of the N-depleted substrate after steam stripping and each two samples of the condensate were analyzed. Results are presented in Table 23.

The degree of dilution by steam condensed within the stripping column was on average 81 %. When excluding the dilution effect of the steam, it can be calculated that there was obviously no reduction in bezafibrate and in β-sitosterol in the N-depleted substrate. A retention time of 3–5 min at 100 °C could not affect theses substances. Diclofenac and ibuprofen could be reduced by 18 and 12 % respectively. Phenacetin and carbamazepine were reduced by 57 and 41 % respectively. Pentoxifylline was not detected; neither in the N-depleted substrate nor in the condensate. The individual reductions during lab scale stripping are displayed in Figure 42 where a stronger offset of the initial concentration can be observed for the lower concentrated pharmaceutical residues.

About 1 % of the amount of the initial substrate concentration of ibuprofen, phenacetin, and carbamazepine could be found in all samples of the condensed off-steam. In three of four samples of the condensed off-steam, traces (0.1–2.5 µg/l) of bezafibrate could be found. β-sitosterol could not be found in any sample of the condensate. The amount of diclofenac in the condensed off-steam was between 1.5 and 4.2 % (0.4–1.1 µg/l) of the initial concentration in the feed substrate. All concentrations were significantly below the level of detection in the urine matrix and could only be measured due to the lower level of detection in aqueous solution (Figure 43).

Demo scale rectification

During the rectification process in demo-scale with overheated steam, one sample of the feed substrate, one of N-depleted substrate and one condensate sample of two different sets were analyzed regarding pharmaceutical residues. The detected concentrations are given in Table 24.

The residence time inside the rectification column was about 15 min. The temperature inside of the column was around 100 °C.

In this case ß-Sitosterol and pentoxifylline were eliminated completely. Carbamazepine was apparently not affected. Reductions of ibuprofen, bezafibrate and diclofenac were more or less in the range of the dilution of the N-depleted substrate by steam condensed within the rectification column. The individual concentrations during pilot scale rectification are displayed in Figure 42 showing nearly no offset in respect to the initial concentration.

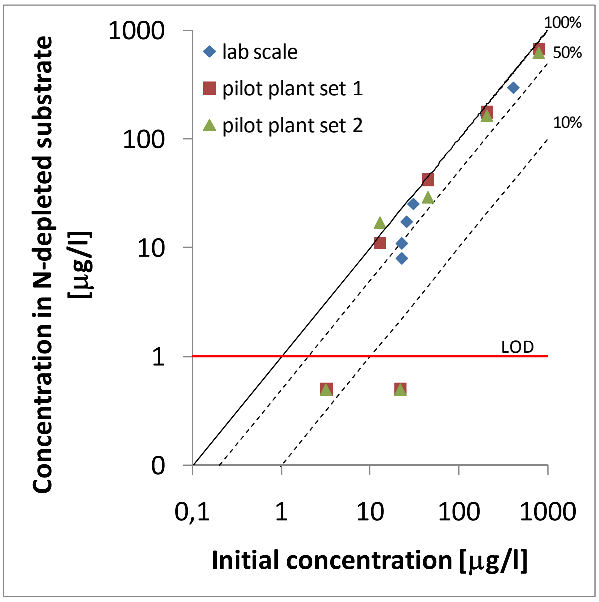


Figure 42: Concentrations of pharmaceutical residues in N-depleted substrate after rectification / steam stripping.

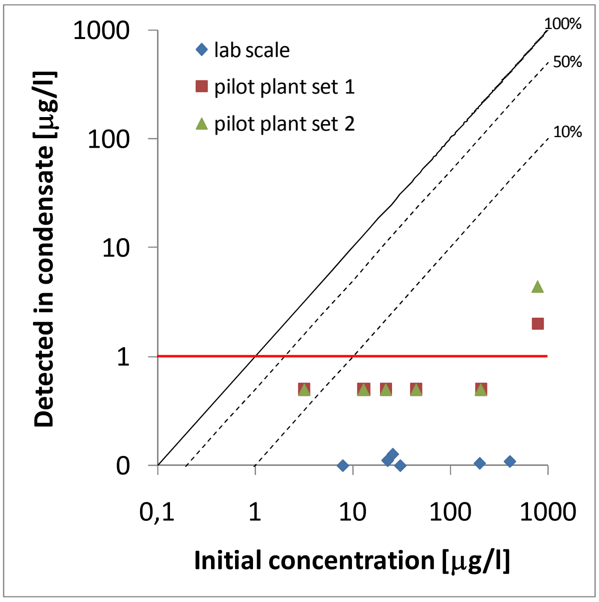


Figure 43: Concentrations of pharmaceutical residues in condensate after rectification / steam stripping of urine.

The horizontal line indicates LOD in urine matrix.

The condensate was nearly free of pharmaceutical residues, only traces of ibuprofen were found (0.25–0.55 % of the initial concentration). Since a phosphorous-balance did not show substrate overflow into the condensate, and also no other pharmaceutical residues could be detected in the condensate, traces of ibuprofen were apparently stripped from the substrate.

Since concentrations of pharmaceutical residues in the condensate were below the level of detection of 1 µg/l, data are displayed as 0.5*LOD in Figure 43.

Table 24: Average initial concentration of pharmaceutical residues in feed substrate (X_0) and concentrations in N-depleted substrate ($X_{1\ (Ndep)}$) and condensate ($Y_{1\ (Cond)}$) of two sets of demo scale rectification

values in µg/l (resp. % of X_0)	X_0	Set 1		Set 2	
		$X_{1\ (Ndepl)}$	$Y_{1\ (Cond)}$	$X_{1\ (Ndepl)}$	$Y_{1\ (Cond)}$
Ibuprofen	794	675 *(85 %)*	2.0 *(0.3 %)*	611 *(77 %)*	4.4 *0.6 %*
Bezafibrate	207	176 *(85 %)*	<1 *(nc)*	162 *(78 %)*	<1 *nc*
Diclofenac	45	42 *(93 %)*	<1 *(nc)*	29 *(64 %)*	<1 *nc*
ß-Sitosterol	22	<1 *(nc)*	<1 *(nc)*	<1 *(nc)*	<1 *nc*
Carbamazepine	13	11 *(85 %)*	<1 *(nc)*	17 *(131 %)*	<1 *nc*
Pentoxifylline	3.2	<1 *(nc)*	<1 *(nc)*	<1 *(nc)*	<1 *nc*

Evaporation

During the evaporation process samples of two concentrates and two distillates were analyzed regarding their content of pharmaceutical residues (Table 25). For the 3.5 fold concentrate an evaporation time of 30 h was needed, and for the 12 fold concentrate the evaporation time was 4 d 6 h at approximately 80 °C and 0.7 bar.

In the 3.5 fold concentrate more or less the expected amount (3.5 x of initial concentration) of carbamazepine, phenacetin, phenazone and pentoxifylline could be detected. 60 % of the expected amount of bezafibrate and less than 30 % of the expected concentrated amount of diclofenac and ibuprofen were detected. In both concentrates ß-sitosterol was not detected.

In the distillate belonging to the 3.5 x concentrate, 40 % of the initial concentration of ibuprofen and 12 % of the initial concentration of carbamazepine was detected.

Table 25: Initial concentrations of pharmaceutical residues (X_0) and concentrations in concentrate ($X_{1\ (Conc)}$) and in distillate ($Y_{1\ (Dist)}$) during two concentration sets.

all values in µg/l (resp. (% of X_0)/cf)	X_0	3.5 fold concentration		12 fold concentration	
		$X_{1\ (Conc)}$	$Y_{1\ (Dist)}$	$X_{1\ (Conc)}$	$Y_{1\ (Dist)}$
Ibuprofen	417	274 *(19 %)*	166 *(40 %)*	37 *(4 %)*	231 *(55 %)*
Bezafibrate	230	494 *(61 %)*	<1 *(nc)*	86 *(5 %)*	<1 *(nc)*
ß-Sitosterol	18	<1 *(nc)*	<1 *(nc)*	<1 *(nc)*	<1 *(nc)*
Diclofenac	17	17 *(29 %)*	<1 *(nc)*	6.3 *(11 %)*	<1 *(nc)*
Phenacetin (*	2.4	8.9 *(106 %)*	<1 *(nc)*	<1 *(nc)*	<1 *(nc)*
Carbamazepine (*	20	85 *(121 %)*	2.5 *(12 %)*	141 *(48 %)*	<1 *(nc)*
Pentoxifylline (*	6.6	25 *(108 %)*	<1 *(nc)*	50 *(58 %)*	<1 *(nc)*
Phenazone (*	4.4	17 *(110 %)*	<1 *(nc)*	79 *(136 %)*	<1 *(nc)*

**) Uncertainties due to single sample of X_0, low concentrations in initial substrate, and matrix influences in concentrate*

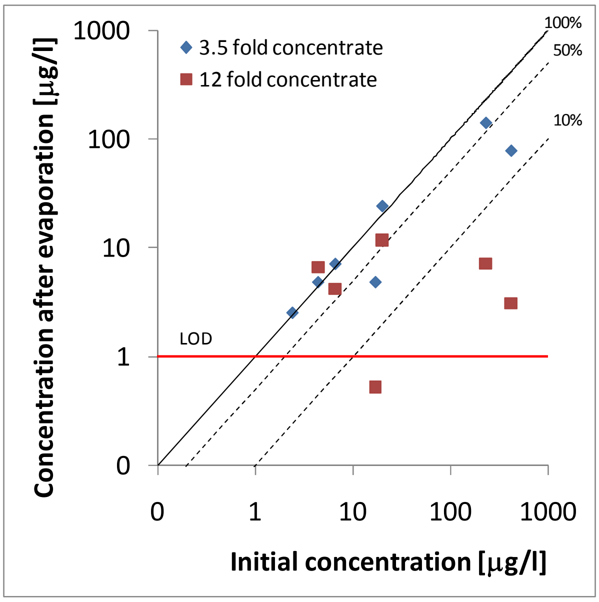


Figure 44: Concentrations of pharmaceutical residues in concentrates after evaporation of urine.

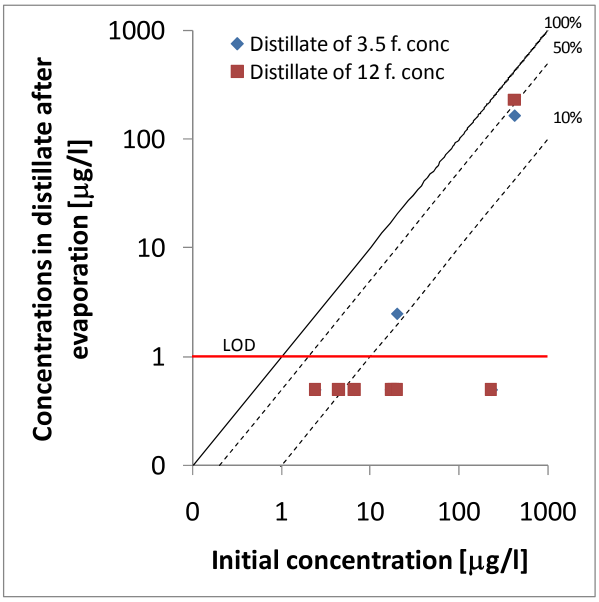


Figure 45: Concentrations in distillate after evaporation of urine.

The horizontal line indicates LOD of urine matrix.

In the 12 x concentrate the amount of phenazone exceeded the expected amount (12 x of initial concentration). About 50 to 60 % of the expected amount of carbamazepine and pentoxifylline was detected in the concentrate. Traces of diclofenac, ibuprofen and bezafibrate could also be detected, but no phenacetin at all.

In the distillate belonging to the 12 x concentrate, 55 % of the initial concentration of ibuprofen was detected, but none of the other parameters.

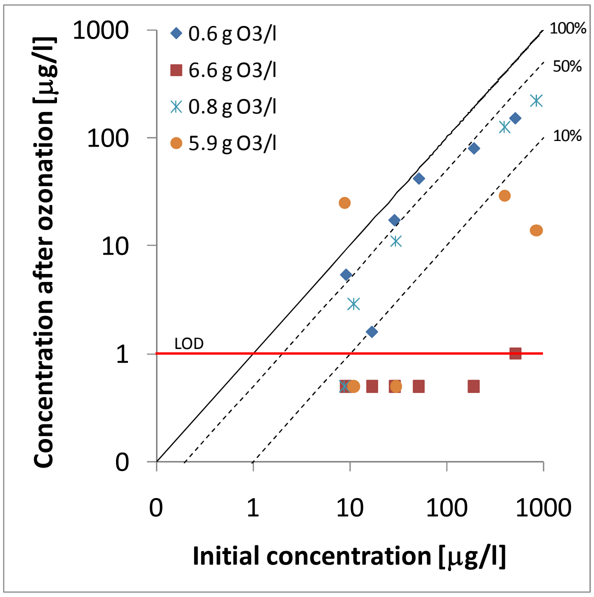
For a quick visualization of the removal efficiency of evaporation on pharmaceutical residues, the results are also displayed in Figure 44 and Figure 45. Since concentrations of pharmaceutical residues in the distillate were below the level of detection of 1 µg/l data are displayed as 0.5*LOD.

It can be noted, that with an evaporation time of 4.25 d at temperatures of 80 °C, five out of the eight pharmaceutical residues could be reduced by more than 97 %. Phenazone was not affected. Also pentoxifylline and carbamazepine proved to be persistent to thermal treatment. Ibuprofen and to a slight extent carbamazepine proved to be volatile compounds.

D.4.5. Ozonation for removal of pharmaceutical residues

Two sets of untreated stored urine from different locations and one set of N-depleted urine after rectification / steam stripping were treated with ozone at different dosages. An overview of the results is given in Figure 46 and Figure 48 respectively Figure 47 and Figure 49.

In Figure 46 and Figure 47 the concentration after ozonation is plotted over the initial concentration. The horizontal line is indicating the LOD of most pharmaceutical residues in the urine matrix. Values below LOD are displayed as 0.5*LOD. The 100 % line indicates substances that are not affected by ozonation. The dotted lines represent 50 % and 10 % of the initial concentration. It can be well observed that concentrations in nitrogen depleted substrate after rectification and after ozonation (Figure 47) are significantly lower compared to concentrations in urine after ozonation (Figure 46).



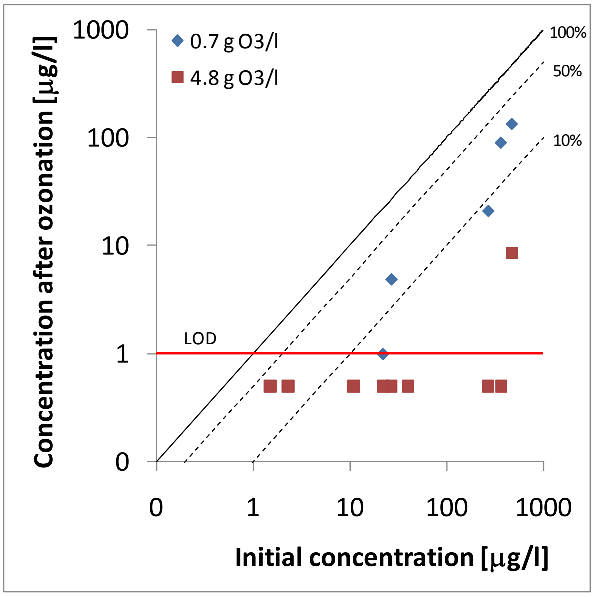
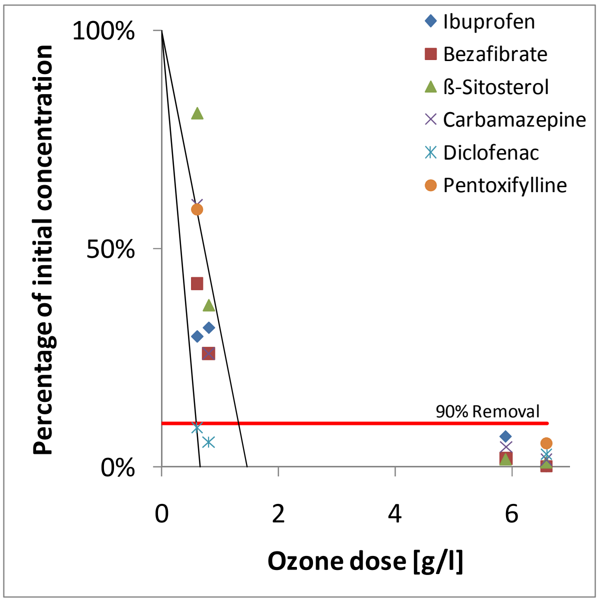


Figure 46: Concentrations of pharmaceutical residues in urine after ozonation.

Figure 47: Concentrations of pharmaceutical residues in N-depleted urine after ozonation.



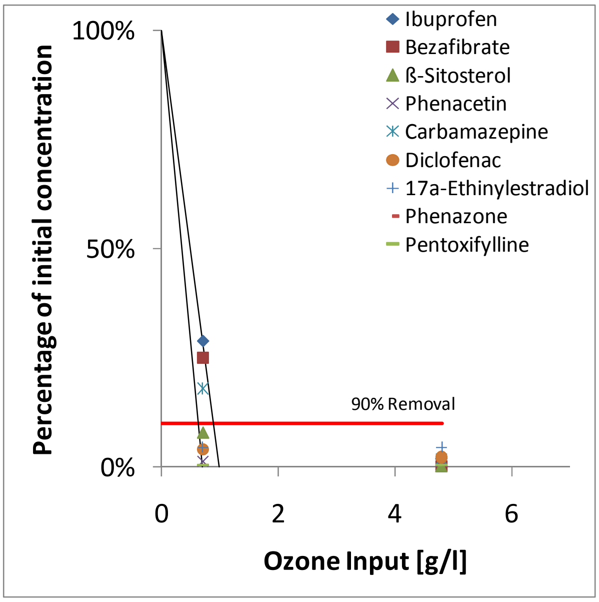


Figure 48: Concentrations in urine after ozonation.

Figure 49: Concentrations in N-depleted urine after ozonation.

At dosages less than 1 g O_3/l all pharmaceutical residues were drastically reduced but still detectable. Only diclofenac was removed completely at an ozone dose of less than 1 g O_3/l. ß-sitosterol in urine was the most persistent compound. At an ozone dosage of 5.9 g O_3/l, 2 % of the initial concentration of bezafibrate could be detected and 7 % of the initial concentration of ibuprofen was detected. The value for diclofenac exceeded the initial concentration by nearly 3 times after ozone dosages of 5.9 g O_3/l. Since in all other cases of all investigated pharmaceutical residues diclofenac was the one that was reduced the most by ozone, this fact is disregarded.

The substance 17α-ethinylestradiol was detected in the initial N-depleted substrate but could not be found after dosages of 0.7 or 4.8 g O_3/l. The initial concentrations of phenazone and pentoxifylline were just above the LOQ. So the complete removal at ozone dosage of 0.7 g O_3/l was expected. Phenacetin was completely eliminated at the lowest dose. ß-sitosterol, which seemed to be the most persistent pharmaceutical during the other ozonation experiments, was removed more than ibuprofen, bezafibrate and carbamazepine.

In Figure 48 and Figure 49 the percentage of the initial concentration of the individual pharmaceutical residues are plotted over the ozone dose. In this case the horizontal line is indicating a 90 % removal. Assuming a linear reduction of the pharmaceutical residues by ozonation within the first 90 % of removal, the ozone demand can be estimated for an average removal rate. To cope with values above the initial concentrations or far away from the average concentrations such as ß-Sitosterol in Figure 48, these values can be excluded in the estimation of an average removal rate. Based on this agreement, the removal rate would be valid for about 90 % of the detected pharmaceutical residues.

In Table 26 the ozone demand for a 90 % removal efficiency is presented. For a 90 % removal rate of 90 % of the detected pharmaceutical residues in urine, an ozone dosage of 1.14 g O_3/l urine was required which is equivalent to 16 kWh/m^3 urine. For N-depleted urine after rectification / steam stripping, the ozone demand for a 90 % removal of 90 % of the detected pharmaceutical residues was 0.84 g O_3/l urine (11.8 kWh/m^3 urine). The amount of energy required for ozone input into the substrate was neglected.

Table 26: Ozone demand for 90 % removal

all values in g O_3/l	**stored urine**	**N-depleted urine**
Ibuprofen	0.92	(0.89)
Bezafibrate	0.95	0.84
ß-Sitosterol	1.14	0.68
Phenacetin	- (*	0.64
Carbamazepine	(1.16)	0.77
Diclofenac	0.68	0.66
17a-Ethinylestradiol	- (*	0.66
Phenazone	- (*	0.63
Pentoxifylline	1.02	0.63

*) Phenacetin, 17a-ethilylestradiol, and phenazone were not detected in the initial substrate of stored urine$_{HH}$ and urine$_B$, additionally pentoxifylline was also not detected in the initial substrate of stored urine$_B$

Ozone dosages were about a factor of 10^3 higher than the ones used for ozonation of river water or wastewater treatment plant effluent (*Huber et al. 2003*; *Ternes et al. 2003*; *von Gunten et al. 2005*). The reason for this is the high COD of about 10 g O_2 /l in urine and its complex matrix. At the same time concentrations of pharmaceutical residues are in the range of a few micrograms up to several hundred micrograms also a factor of 10^3 higher than in surface waters or effluents of wastewater treatment plants.

Effect of pH

Carbon double-bounds can be attacked via a direct selective reaction of ozone according to the Crigée-mechanism. While at high pH values a large quantity of ozone is consumed for formation of radicals that attack components rapidly but non-selectively, in an acidic environment the direct selective reaction is supported.

For testing the relationship of ozonation efficiency and pH, stored urine and N-depleted urine were acidified and subsequently ozonated. Pharmaceutical residues in urine were reduced by 15 % on average by acidification to pH 4. Pharmaceutical residues in N-depleted urine were reduced by 20 % on average by acidification to pH 4.

Thereafter stored urine and an acidified batch of the same stored urine were treated. Samples were taken at intervals of 80 and 230 minutes. The O_3-uptake within the same time intervals was reduced by roughly 50 % by acidification (Table 27) since a chain-reaction of OH° radicals and O_3-molecules was not supported in the acidic environment.

Within the same timeframe only half of the amount of O_3 was consumed by the acidified sample. At similar ozone consumption for most cases, except for ß-sitosterol, reduction rates were in the same range. Detailed results are presented in the appendix in chapter I.3.3.

In the case of N-depleted urine, reduction rates at similar ozone consumption were significantly higher in the acidified substrate. When excluding the competitor ammonium, the effect of enhancing the direct reaction according to the Crigée-mechanism was supported.

Nearly all pharmaceutical residues seem to have a more intensive reaction with ozone itself than with the produced radicals. However, it seems that most pharmaceutical residues can also be reduced by radicals to some extent, except ß-sitosterol, which seems to be persistent against UVC-irradiation and reaction with radicals.

Table 27: Ozone uptake at different pH

	t = 80 min	t = 230min
stored urine pH 8.9	0.97 g O_3/l	1.59 g O_3/l
stored urine at pH 4	0.50 g O_3/l	0.86 g O_3/l
N-depl urine pH 7	1.37 g O_3/l	1.94 g O_3/l
N-depl urine pH 4	0.63 g O_3/l	1.26 g O_3/l

Due to the fact that most pharmaceutical residues have aromatic structures, the average removal rate for pharmaceutical residues during ozonation was slightly affected by pH adjustment. An acidification to pH 4 decreased the 50 % removal rate from an ozone demand of 0.53 g O_3/l urine for stored urine with a pH of about 9 to an ozone demand of 0.37 g O_3/l urine which is equivalent to an energy demand of 5.18 kWh/m^3 urine.

Side products of ozonation

Due to the high TOC content, the selective reaction of ozone might also lead to fragmentary mineralization products from organic components (*Gulyas 2003*). Because of the high chloride concentration of up to more than 4 g/l, the formation of AOX could be expected by the ozonation of urine.

Analytics regarding AOX formation were conducted. However, because of its high ammonium and chloride content, the LOD for AOX was in the range of 1 mg/l. This value was not exceeded during the ozonation of urine for removal of pharmaceutical residues.

D.4.6. Indicators for removal of pharmaceutical residues

Since the analytics regarding pharmaceutical residues in urine are costly, other indicators for the removal of pharmaceutical residues might be beneficial. In the case of UVC-irradiation and ozonation, the change of color throughout ozonation was measured. Chroma and turbidity were derived as described in (*Tettenborn et al. 2006*). Both chroma and turbidity decreased as expected during ozonation but not during low UVC-irradiation (see *Tettenborn et al.* (*2007*)).

However, color reduction in all experiments was little after ozone-input sufficient for removal of pharmaceutical residues. During UVC-irradiation a color reduction could not be obtained by the applied dosages. From previous experiments (*Tettenborn et al. 2006*) it is known that color can be removed by ozone and UVC-irradiation dosages about two to three times higher than for the removal of pharmaceutical residues needed. Thus it can be stated that decolorized urine will not contain any pharmaceutical residues, but color as an indicator for the removal of pharmaceutical residues seems not to be sensitive enough.

D.4.7. Energy

According to (*Bünning 1996*) energy requirements per kg produced O_3 are between 6.5 and 10 kWh when using oxygen as feed gas. For preparation of oxygen another kWh is needed for 1 N m^3 of oxygen. When producing ozone from air 14 kWh are needed per kg ozone. The amount of energy required for ozone input into the substrate is neglected.

With an energy input of 16 kWh/m^3 substrate (1.14 g O_3/l) 90 % of all pharmaceutical residues in urine could be reduced by 90 %. For acidified N-depleted urine the amount would be lower. While the energy input for ozonation of urine was about 500 times higher than values reported by *Ternes et al. (2003)* for the treatment of wastewater treatment plants effluent, the volume per person and day of urine is about 300 times lower than the average wastewater load processed at German wastewater treatment plants per person and day (estimation see Appendix 3.3, page A-18) and concentrations of pharmaceutical residues in urine are about 10^3 times higher than in the effluent of wastewater treatment plants. Therefore the energy demand per concentration unit of pharmaceutical residues in urine and in wastewater treatment plant effluent is in a similar range.

D.5. Intermediate discussion

D.5.1. Rectification / steam stripping

Before testing nitrogen recovery on an industrial scale, a pilot plant was first designed and set up. The plant was designed to process substrate from all public waterless urinals in Hamburg, resulting in a capacity of about 100 l/h. For every cubic meter of urine, 25–35 l of an ammonia solution containing up to 120 g NH_3 /l could be produced from a rather low concentrated feed. Thus up to 50 times of the initial NH_3-content could be condensed in 1/40th of the volume. In the remaining N-depleted substrate, a reduction of 90 to 98 % could be reached. Thus the resulting NH_3 concentration of 6–10 mmol/l in the depleted substrate would be somewhat equivalent to the phosphorous concentration of 5 mmol/l and could be completely removed by subsequent MAP-precipitation.

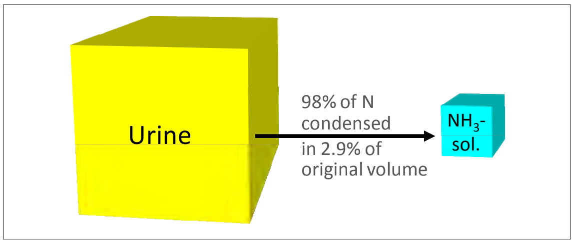
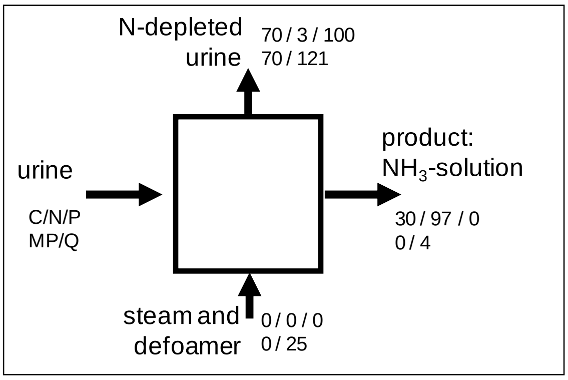


Figure 50: Volume reduction urine feed versus NH_3 solution as product



MP: Micropollutants; Q: quantity

Figure 51: Scheme of rectification process with quantitative in- and output according *Maurer et al. (2006)*.

Pre-treatment of the substrate was not necessary, and the pH of hydrolyzed urine was in a range suitable for the rectification and stripping process. Precipitation within the system was not observed.

A fine steam adjustment as well as slight process modifications such as improved energy recovery and a fractionated condensation would lead to even higher ammonia concentrations in the yielded product and a lower energy consumption. By cooling the head of the stripping column the process could be modified to a rectification process allowing yield concentrations of up to nearly 20 % ammonia by weight. Lower temperatures than 45 °C are not recommended due to economical reasons (*Mersmann et al. 2005*), instead a second rectification stage at high pressure (e.g. 20 bar) could be used to produce pure liquid ammonia

(*Mersmann et al. 2005*). The big advantage towards air stripping is that no additional chemicals are needed for production purposes.

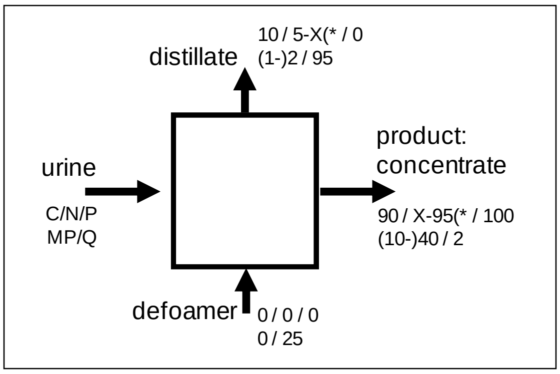
D.5.2. Evaporation

Highly concentrated solutions could be obtained from stored urine and from N-depleted urine after steam striping. Concentration factors of 55 fold and more could be reached which is equivalent to a volume reduction from 1 m^3 down to 18 l.

Concentrates up to 60 fold had a low viscosity and phosphorous, potassium, and sulfur were captured in the concentrates. The distillate contained organic acids, leading to COD values in the range of the initial COD.

As *Maurer et al.* (*2006*) stated, the two main obstacles for evaporation of urine are loss of ammonia and the high energy demand. Acidification of stored urine requires large quantities of acid. Since sulfuric acid can lead to corrosion in most regular evaporation plants, phosphoric acid could be used within conventional evaporation units. While prices for phosphoric acid are about three times higher than for sulfuric acid, the yielded concentrate will also be more valuable because of its high P-content. During the conducted evaporation experiments approximately 1.5 g P/l was added to the source separated urine. It has to be noted that for producing P-fertilizer, phosphoric acid is one of the main source materials. However, since phosphorus is a limited resource, the practice of using phosphoric acid in the treatment of urine to enhance the process and for improving product value is questionable and can be further discussed.

Evaporation of N-depleted urine is an option. pH and buffer capacity are both reduced by the stripping of ammonia. In this project the pH of the large volume of N-depleted substrate was not as low as it possibly can be, since the N-depleted substrate was produced in several sets, where focus was not always on maximum N-depletion.



*) strongly depending on pH of initial substrate. Values in brackets based on assumptions.

Figure 52: Process scheme for evaporation according to *Maurer et al.* (*2006*)

In laboratory tests it could be proved that foaming of yellow water can be controlled easily by the addition of anti-foaming agents. Because of the very heterogeneous nature of urine, a constantly occurring boiling retardation (delay of boiling point) leads to splashing if no action is taken. The most effective counter-measure seemed to be agitation of the substrate. However, in the used pilot plant this was not possible.

Within the evaporation bulb no crystal precipitation was observed. Some flock forming and after a while crystal growth occurred in the stored concentrates.

D.5.3. Crystallization, precipitation

Self crystallization occurred in highly concentrated urine solutions, starting at a concentration factor of 30. About 90 % of the phosphorous could be recovered by crystallization. Thus per cubic meter of low concentrated source separated urine, about 250 g of phosphorous could be recovered. The simple lab procedure left space for optimization.

According to the results of *Tettenborn et al.* (*2007*) the removal efficiency for phosphate in all investigated substrates was found to be depending on the dosage of precipitants. MgO and $MgCl_2$ addition had resulted in over 95 % phosphate removal by lower dosages. According to literature, recovery rates of 98 % should be possible. For nitrogen depleted urine except MgO, a pH adjustment was needed and high phosphate removals could be achieved for MgO, $Ca(OH)_2$ and $CaCl_2$.

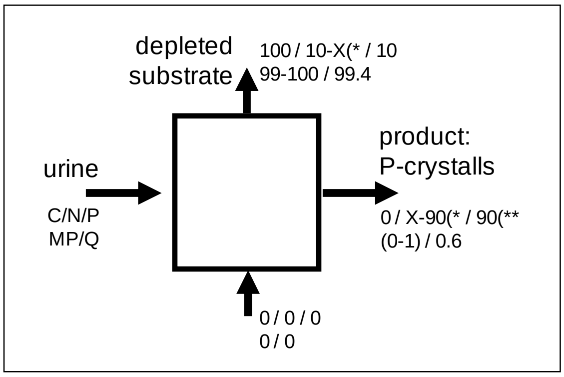


Figure 53: Process scheme for crystallization according to Maurer et al. (2006).

*) strongly depending on pH of initial substrate. Values in brackets based on assumptions.

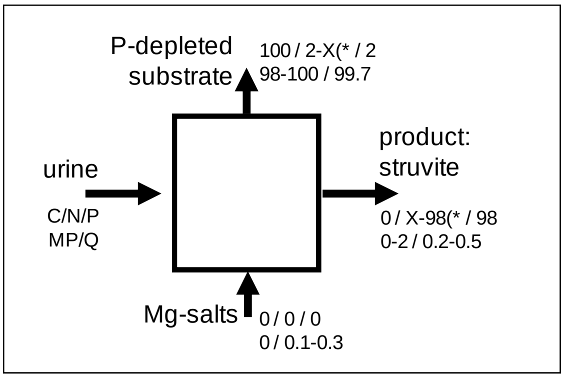


Figure 54: Process scheme for precipitation according to (Maurer et al. 2006).

*) strongly depending on pH of initial substrate.

D.5.4. Removal of pharmaceutical residues

During nutrient recovery processes

All thermal processes for nutrient recovery did affect pharmaceutical residues concentrations in urine.

During steam stripping at lab scale slight reductions of carbamazepine (41 %) and phenacetin (57 %) were observed. However, steam stripping with overheated steam at demo scale could not verify this result. Here only ß-sitosterol and pentoxifylline were eliminated completely. The condensate from the rectification / steam-stripping unit was mainly free of pharmaceutical residues. Residence times within the column were between 5 and 15 minutes, and temperatures around 100 °C.

After 4d 6h in the evaporation process at a temperature of around 80 °C, all pharmaceutical residues except carbamazepine, phenazone, and pentoxifylline were reduced by more than 95 %. Significant traces of carbamazepine could be found in the distillate after 30 h. Also, more than 50 % of the initial concentration of ibuprofen was found in the distillate after 4d 6h. It can be assumed that at higher concentration steps with longer process times, the remaining amounts of pharmaceutical residues in the concentrate will be smaller.

During ozonation

With ozonation a dose of 2.5 g O_3/l was sufficient to eliminate all pharmaceutical residues. Ozonation was more effective when ammonia was stripped out beforehand.

Ozonation of N-depleted urine showed a higher reduction of most pharmaceutical residues than from the matrix of urine. While ß-sitosterol in untreated stored urine was reduced by 32 % and 79 % per 1 g O_3/l, a complete removal in the N-depleted substrate should be achieved at a dosage of about 1 g O_3/l. Bezafibrate and carbamazepine showed similar tendencies. Only diclofenac and ibuprofen did not show such a clear effect. The high initial concentration of diclofenac could be one reason why it was not affected by pretreatment of the urine. For the other substances, the reduction of ammonium in the urine might have increased the efficiency, since one competitor (in this case the ammonium) for the ozone molecules was removed by stripping.

An ozonation dosage of 2.5 g O_3/l (35 kWh/m^3) was found to be sufficient to eliminate all pharmaceutical residues. For a 90 % removal of 90 % of the pharmaceutical residues an ozone dosage of 1.14 g O_3/l urine was required, equivalent to 16 kWh/m^3 urine.

Ozonation was more effective when ammonia was stripped out before, since competition of substances such as ammonium was reduced. The reduction of pH seemed to influence ß-sitosterol the most.

Ozone consumption over time was less in the acidified urine, but reduction of pharmaceutical residues was depending more on ozone consumption than on time. However, in both acidified substrates, and even more in the N-depleted acidified substrate, the reduction efficiency of pharmaceutical residues was slightly increased by combination of acidification and ozonation.

Due to the insufficient barrier of today’s wastewater treatment plants pharmaceuticals are still entering the environment (*Castiglioni et al. 2006*; *Paxéus 2004*; *Strenn et al. 2004*). Separation of urine could be one approach to collect a large quantity of these micropollutants at source and reduce the load entering the environment.

E. Implementation potential

The two aspects *quality* and *quantity* of substrate in an urban urine separation system with central or semi-central treatment influence the choice of elements and the efficiency of this system. The two aspects depend on:

- location of source collection (public, work, private);
- type of collection device,
    - waterless and/or low flush urinals (dilution effects by flushing water),
    - separation toilets with flush and without flush of urine section (dilution effects by flushing water or during cleaning, and rate of separation),
    - nutrient losses within a collection system.

To evaluate the potential of a system with separate urine collection in the context of a European city, different scenarios where set up and investigated according to the data derived.

E.1. Focus of investigation

The focus is to find boundary conditions for separate collection and treatment of yellow water and/or urine, and to find an optimum treatment chain with its efficiency parameters (here explicitly: energy demand and yield of recovered nutrients).

E.2. Scenario building

Based on the previous results, different scenarios were derived corresponding to the context of a western European city (Hamburg). The scenarios were varied in

- type of collection
    - waterless urinals (wlu)
    - urine diverting toilets (udt)
- location of collection
    - public area
    - office buildings
    - private households
- coverage
    - the coverage described the number of people involved, and was varied from a current state of about 0.04 % of the total population of Hamburg, up to a coverage of 80 % of the total population
- state of technical development of separation toilets
    - current state
    - optimized separation and reduced dilution

Consequentially amount (quantity) and concentrations (quality) of the substrate were varied according to the scenario variations.

Scenario 1

Scenario 1 was developed based on the current situation in Hamburg with public waterless urinals equipped with collection tanks with the following framing parameters:

- time frame: current state;
- amount of collected urine: 0.04 % of total collectable volume of Hamburg; equivalent to 730 capita based on volume;
- concentrations based on the measuring campaign outlined in chapter B.

Scenario 2

Scenario 2 was a possible extension of the current situation based on public waterless urinals in Hamburg. It was based on the idea of including additional sites and developing a collection system for large public events such as city fairs, with mobile public toilets. The framing parameters were:

- possible implementation would be immediate or within the next three years;
- amount of collected urine: 0.13 % of total collectable volume of Hamburg; equivalent to 2 200 capita based on volume;
- concentrations based on the measuring campaign outlined in chapter B.

Scenario 3

Scenario 3 was also quite a realistic scenario. In addition to scenario 2, the collection of urine from waterless urinals from office buildings, schools, and universities was assumed. Many office buildings and also buildings e.g. on the Hamburg University campus are equipped with waterless urinals already for water saving purposes. Collection tanks and a collection and transport system (including additional urine pipes in houses) would be needed to be implemented. The framing parameters were:

- implementation time frame would be presumably three to five years;
- amount of collected urine: 0.6 % of total collectable volume of Hamburg; equivalent to 10 000 capita based on volume;
- concentrations assumed according to chapter E.2.1.

Scenario 4

In scenario 4 the implementation of urine diverting toilets was additionally assumed for selected office buildings, schools, etc. The framing parameters were:

- implementation frame could be presumably within five to seven years;
- amount of collected urine: 1 % of total collectable volume of Hamburg ;
- separation ratio of urine diverting toilets in office buildings: 40 %;
- dilution grade of urine diverting in office buildings toilets: 20 %;
- concentrations assumed according to chapter E.2.1.

Scenario 5a

In scenarios 5a to 5d the implementation of waterless urinals and urine separation toilets was additionally assumed for selected private households. The framing parameters were:

- implementation frame could be presumably within seven to ten years;
- amount of collected urine: 20 % of total collectable volume of Hamburg;
- concentrations according assumptions in chapter E.2.1.

Scenario 5 b – 5 e

Similar to scenario 5a with change in scale and separation efficiency of the urine diverting toilets. The framing parameters were:

- Scenarios 5a and 5b:
  - separation ratio of urine diverting toilets in office buildings: 45 %,
  - dilution grade of urine diverting in office buildings toilets: 20 %,
  - separation ratio of urine diverting toilets in private buildings: 55 %,
  - dilution grade of urine diverting in office private buildings: 10 %.
- Scenarios 5c and 5d
  - separation ratio of urine diverting toilets in office buildings: 70 %,
  - dilution grade of urine diverting in office buildings toilets: 10 %,
  - separation ratio of urine diverting toilets in private buildings: 90 %,
  - dilution grade of urine diverting in office private buildings: 5 %.
- Amount of collected urine (substrate volume in Table 28)
  - in scenario 5b: 60 % of total collectable volume;
  - in scenario 5c: 25 % of total collectable volume (because of improved separation technique by optimized urine diverting toilet);
  - in scenario 5d: 77 % of total collectable volume (because of improved separation technique by optimized urine diverting toilet).
- concentrations according assumptions in chapter E.2.1.

A summary of the scenario boundary conditions is given in Table 28.

Table 28: Parameter variations of the different scenarios

	Collection location and type of collection device			**Substrate volume [m^3/d]**	**Implementation time scale**
	public	**work, etc.**	**private**		
Scenario 1	wlu	-	-	1	immediat
Scenario 2	wlu	-	-	3	very soon
Scenario 3	wlu	wlu	-	14	quite soon
Scenario 4	wlu	wlu + udt	-	22	soon
Scenario 5 a b	wlu	wlu + udt	wlu + udt	455 1450	later
Scenario 5 c d	wlu	wlu + o-udt	wlu + o-udt	560 1790	later

wlu: wateless urinal; udt: urine diverting toilet; o-udt: optimized urine diverting toilet

E.2.1. Assumptions

General assumtions

The total amount of collectable urine is dependent upon:

- rate of coverage (numbers of users);
- separation ratio of urine diverting toilets (parts of urine are lost via brownwater);
- dilution grade (from flushing water entering collection systems).

Since reliable data for only some aspects are available, the following assumptions are made:

- urine from pubic toilets is only collected via waterless urinals, not via urine diverting toilets;
- where relevant, the ratio of toilet usages between private places and office, school, etc. is 50/50;
- at all locations a gender ratio of 50/50 is assumed;
- usage of waterless urinal in private places and office, school, etc.: 75 % of the male population would use a waterless urinal on average;
- dilution rates of urine diverting toilets depend on location and grade of technical optimization, influencing the concentration characteristics of the substrate.

Substrate parameters

Based on these assumptions the consequential base parameters for the scenarios were derived. The derived average concentrations for the different collection systems at different locations are given in Table 29.

Table 29: Assumed concentrations for different collection systems at different locations

	waterless urinals (wlu)			**separating systems (udt)**		**optimized separating systems (o-udt)**	
	public	**work**	**private**	**work**	**private**	**work**	**private**
Separation ratio [%] (collectable fraction)	100	100	100	45	55	70	90
Dilution [%] **				20	10	10	5
Resulting concentrations							
C [g/l]	2.25	5.00	5.00	4.00	4.50	4.50	4.75
N [g/l]	3.74	8.30	8.30	6.64	7.47	7.47	7.89
P [g/l]	0.28	0.80	0.80	0.64	0.72	0.72	0.76
K [g/l]	0.87	1.90	1.90	1.52	1.71	1.71	1.81
S [g/l]	0.45	1.00	1.00	0.80	0.90	0.90	0.95
MP [%]	45	100	100	80	90	90	95
base on	*	***	***	**	**	**	**

work includes schools, universities, office buildings, etc. Separation systems are based on the following assumption: 50 % male, partially covered by urinals; 50 % females. * based on previous results; ** derived from previous results and Meinzinger & Oldenburg (2009); *** based on Meinzinger & Oldenburg (2009)

A detailed overview of the base parameters (quantities and concentrations) of the scenarios differentiated by type and location of collection are given in Appendix table 29, Appendix table 30, and Appendix table 31.

Table 30: Process parameters

Process		**Input**	**Additive**	**Product**	**Secondary product**	**energy [MJ/m^3]**
Rectification	type		Steam, defoamer	NH_3-solution	N-depleted urine	150
	Q [%]	100	25	4	121	
	C [%]	100	0	30	70	
	N [%]	100	0	97	3	
	P [%]	100	0	0	100	
	MP [%]	100	0	0	70	
Evaporation	type		Defoamer	Concentrate	Distillate	200
	Q [%]	100	1	2–5	95–98	
	C [%]	100	0	90	10	
	N [%]	100	0	X–95	5–X	
	P [%]	100	0	100	0	
	MP [%]	100	0	10-40	1-2	
MAP-precipitation	type		Magnesium salts	MAP	P-depleted urine	~ 30
	Q [%]	100	0.1-0.3	0.2-0.5	99.8	
	C [%]	100	0	0	100	
	N [%]	100	0	XX-98	2-XX	
	P [%]	100	0	98	2	
	MP [%]	100	0	0–2	98–100	
Crystallization	type			P-crystals	P-depleted urine	~ 20
	Q [%]		-	0.64	99.36	
	C [%]	100	-	0	100	
	N [%]	100	-	XX-90	10-XX	
	P [%]	100	-	90	10	
	MP [%]	100	-	0-1	99-100	

X: depending on N-retention after rectification; XX: depending on N:P ratio after rectification

Process parameters

In the scenario calculation for the treatment aspects, the rectification efficiency was optimized depending on the concentrations and the subsequent processes. Defoamer volume during evaporation was not included in products volumes. Process parameters are displayed in Table 30. The assumed energy demand for each process was not altered in the scenario building.

Process chains

The two investigated process chains were:

- rectification with subsequent MAP precipitation (process chain 1); and
- rectification with subsequent evaporation and crystallization (process chain 2).

E.3. Results

Results were derived for the system elements of collection, transport and treatment.

E.3.1. Collection

Based on the scenario set ups, concentrations are varying. In scenarios 1 and 2 the integration of source separated urine from public toilets only was assumed. Thus concentrations are low due to the contribution of low concentrated urine, reflected also in the percentage of contained micropollutants, all equivalent to the previous results from public toilets. In scenario 3 concentrations are significantly higher due to the fact of integrating source separated urine from collection systems where higher concentrations can be assumed. The lower concentrations in scenario 4, 5a, and 5b are due to the integration of urine diverting toilets and the assumption of dilution effects from flushing water as experienced in previous pilot projects and stated in (*Maurer et al. 2006*).

The final quantities and concentrations of the substrates of the different scenarios are given in Table 31.

Table 31: Input parameters of scenarios based on previous assumptions

Scenario	1	2	3	4	5a	5b	5c	5d
Q [m^3/d]	1.00	3.01	14.0	22.2	455	1450	560	1790
C [g/l]	2.25	2.25	4.41	4.26	4.64	4.65	4.78	4.79
N [g/l]	3.74	3.74	7.32	7.07	7.70	7.72	7.94	7.95
P [g/l]	0.28	0.28	0.69	0.67	0.74	0.74	0.76	0.77
MP [%]	45	45	88	85	93	93	96	96

E.3.2. Transport

From the results in chapter C.5 the energy demand for each scenario was derived resulting in the values given in Table 32.

Table 32: Energy demand for transport within the different scenarios

Scenario	1	2	3	4	5 a	5 b	5 c	5 d
kWh/m³	15.2	15.2	15.2	15.2	7.5	5.1	7.5	3.8
kWh/d	15.2	45.8	212	337	3 414	7 395	4 202	6 788

E.3.3. Treatment

Process chain 1

In process chain 1 rectification was supposed to be used for nitrogen recovery in the form of highly concentrated ammonia solution, optimized for a subsequent MAP precipitation. The subsequent MAP precipitation was designed for recovery of phosphorous and the remaining nitrogen in form of struvite crystals.

Table 33: Results from rectification process optimized for subsequent MAP-precipitation

Scenario	1	2	3	4	5 a	5 b	5 c	5 d
Rectification in: see Substrate (Table 31)								
Rectification addition = Steam								
Q [m^3/d]	0.25	0.75	3.49	5.55	115	368	141	448
Recticfication product = NH_3 solution								
Q [m^3/d]	0.04	0.12	0.56	0.89	18.5	58.8	22.5	71.7
C [g/l]	16.9	16.9	33.1	31.9	34.8	34.9	35.9	35.9
N [g/l]	90.1	90.1	175	169	184	185	190	190
P [g/l]	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
MP [%]	0	0	0	0	0	0	0	0
Rectification secondary product for further processing in MAP precipitation or in evaporation plus crystallization								
Q [m^3/d]	1.21	3.65	16.9	26.9	558	1779	680	2170
C [g/l]	1.30	1.30	2.55	2.46	2.68	2.69	2.77	2.77
N [g/l]	0.11	0.11	0.27	0.26	0.28	0.28	0.29	0.29
P [g/l]	0.23	0.23	0.57	0.55	0.61	0.61	0.63	0.63
MP [%]	32	32	62	60	65	65	67	67

Depending on the feed nitrogen concentrations, 40 to 120 l/d of a 9.4 % ammonia solution (90 g N/l) would be gained within scenarios 1 and 2, and 600 to 900 l/d of a 19 % and 18 % ammonia solution would be gained within scenarios 3 and 4 (175 and 169 g N/l). In scenarios 5a and 5c roughly 20 m^3/d of 20 % and 21 % ammonia solution would be gained (185 and 190 g N/l), and about 60 to 70 m^3/d in scenarios 5b and 5d (Table 33).

In this scenario-building the different output concentrations are due to different input concentrations of the scenarios and due to assumed optimized process operation but not directly due to scale.

To allow a subsequent MAP precipitation, nitrogen concentrations in the secondary product of the rectification process are in the range of 100 to 300 mg/l.

The phosphorous and the remaining nitrogen would be recovered by MAP precipitation, yielding between 2.2 kg MAP/d in scenario 1 up to 11 t MAP/d in scenario 5d (Table 35).

Table 34: Nutrient recovery and micropollutant removal rates of process chain 1

	Min	Max
N-recovery	99.5%	99.8%
P-recovery	97.3%	98.4%
MP-removal	30.0%	30.0%

The range of nutrient recovery and micropollutant removal rates of process chain 1 are displayed in Table 34. The total N-recovery, including rectification and MAP-precipitation raged in all scenarios between 99.5 and 99.8 %, P-recovery by MAP precipitation ranged from 97.3 to 98.4 %, while micropollutants would be removed by the process combination of rectification and MAP-precipitation by 30 %.

The remaining secondary product after rectification and MAP precipitation would be almost free of nitrogen and phosphorous. Concentrations would be around 10 mg/l and lower (Table 34).

Concentrations of organics would be in a range of 1.3 to nearly 3 g/l requiring a follow up treatment. It can be assumed that most organics should be readily biodegradable. Due to the low nutrient concentrations a treatment together with other source separated streams with low nutrient concentrations such as greywater would be sufficient.

Remaining pharmaceutical residues in the secondary product would be in the range of 32 to 67 % of initial concentrations in undiluted urine. These substances could be removed by e.g. ozonation, requiring an additional energy demand of 12 to 16 kWh/l.

The energy demand for process chain 1 without ozonation is given in Table 36.

Table 35: Results from MAP precipitation process after rectification (process chain 1)

Scenario	1	2	3	4	5 a	5 b	5 c	5 d
MAP in: see Rectification secondary product (optimized for subsequent MAP precipitation process)								
MAP addition (calculated with $MgCl_2$)								
Q [kg/d]	1.29	3.89	44.3	68.6	1 578	5 039	1 983	6 334
MAP product								
Q [kg/d]	2.22	6.68	76.1	118	2 708	8 648	3 402	10 869
N [kg/d]	0.12	0.37	4.25	6.58	151	484	190	608
P [kg/d]	0.27	0.83	9.42	14.6	335	1071	421	1 346
MP [%]	0-0.06	0-0.06	0-0.124	0-0.124	0-0.125	0-0.125	0-0.125	0-0.125
MAP secondary product = final secondary product								
Q [m^3/d]	1.21	3.64	16.8	26.7	556	1770	677	2159
C [g/l]	1.30	1.30	2.55	2.46	2.68	2.69	2.77	2.77
N [g/l]	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
P [g/l]	0.00	0.00	0.01	0.01	0.01	0.01	0.01	0.01
MP [%]	31.9	31.9	61.9	59.9	64.9	64.9	66.9	66.9

Table 36: Energy demand in for process chain 1

Scenario	1	2	3	4	5 a	5 b	5 c	5 d
Rectification [MJ/kg N]	42	42	21	22	20	20	20	20
Precipitation [MJ/kg P]	133	133	53	55	49	49	48	48

Process chain 2

Concentrations in the yielded ammonia solution in process chain 2 are slightly lower than in process chain 1 to leave enough remaining nitrogen for the subsequent evaporation and crystallization.

Table 37: Results from rectification process optimized for subsequent evaporation and crystallization

Scenario	1	2	3	4	5 a	5 b	5 c	5 d
Rectificaiton in: see Substrate								
Rectification addition = Steam								
Q [m^3/d]	0.25	0.75	3.49	5.55	115	368	141	448
Rectification product = NH_3 solution								
Q [m^3/d]	0.04	0.12	0.56	0.89	18.5	58.8	22.5	71.7
C [g/l]	16.9	16.9	33.1	31.9	34.8	34.9	35.9	35.9
N [g/l]	87	87	167	162	176	176	181	182
P [g/l]	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
MP [%]	0	0	0	0	0	0	0	0
Rectification secondary product for further processing in MAP precipitation or in evaporation plus crystallization								
Q [m^3/d]	1.21	3.65	16.9	26.9	558	1779	680	2170
C [g/l]	1.30	1.30	2.55	2.46	2.68	2.69	2.77	2.77
N [g/l]	0.21	0.21	0.51	0.50	0.55	0.55	0.57	0.57
P [g/l]	0.23	0.23	0.57	0.55	0.61	0.61	0.63	0.63
MP [%]	32	32	62	60	65	65	67	67

Depending on the feed nitrogen concentrations, 40 to 120 l/d of a 9.1 % ammonia solution (87 g N/l) would be gained within scenario 1 and 2, and 600 to 900 l/d of a 18 % and 17.4 % ammonia solution would be gained within scenario 3 and 4 (167 and 162 g N/l).

In scenario 5a and 5c roughly 20 m^3/d of a 19 % and 19.6 % ammonia solution would be gained (176 and 181 g N/l), and about 60 to 70 m^3/d in scenarios 5b and 5d (Table 37). The different output concentrations are due to different input concentrations of the scenarios, due to assumed optimized process operation but not directly due to scale.

To allow a subsequent crystallization after the evaporation process, parameters for the rectification process were designed to obtain nitrogen concentrations in the secondary product of 200 to 600 mg/l (Table 37).

The phosphorous and the remaining nitrogen would be recovered by evaporation and crystallization, yielding between 30 l concentrate per day in scenario 1 up to 54 m^3 concentrate per day in scenario 5d (Table 38).

Of this concentrate between 2 kg of crystals per day in scenario 1 up to 10 t of crystals per day in scenario 5d could be produced (Table 39).

Table 38: Results from evaporation process after rectification (process chain 2)

Scenario	**1**	**2**	**3**	**4**	**5 a**	**5 b**	**5 c**	**5 d**
Evap in = Rectification secondary product (optimized for subsequent evaporation and crystallization process)								
Evap product = concentrate								
Q [m^3/d]	0.03	0.09	0.42	0.67	13.8	44.1	16.9	53.8
C [g/l]	47	47	93	89	97	98	100	101
N [g/l]	4.21	4.21	10.35	10.08	11.16	11.19	11.51	11.53
P [g/l]	9.32	9.32	22.91	22.32	24.69	24.76	25.46	25.52
MP [%]	8	8	15	15	16	16	17	17
Evap secondary product								
Q [m^3/d]	1.19	3.57	16.6	26.3	547	1743	667	2126
C [g/l]	0.13	0.13	0.26	0.25	0.27	0.27	0.28	0.28
N [g/l]	0.11	0.11	0.26	0.26	0.28	0.28	0.29	0.29
P [g/l]	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
MP [%]	0.64	0.64	1.26	1.22	1.32	1.32	1.36	1.37

Table 39: Results from subsequent crystallization after rectification (process chain 2)

Scenario	**1**	**2**	**3**	**4**	**5 a**	**5 b**	**5 c**	**5 d**
Cryst in = Evap product								
Cryst product								
Q [kg/d]	1.89	5.68	64.7	100	2 305	7 364	2 896	9 256
N [kg/d]	0.11	0.34	3.90	6.04	136	435	175	555
P [kg/d]	0.25	0.76	8.64	13.4	302	965	385	1 230
MP [%]	0	0	0	0	0	0	0	0
Cryst secondary product								
Q [m^3/d]	0.03	0.08	0.35	0.57	11.5	36.7	14.0	44.5
C [g/l]	47	47	93	89	97	98	100	101
N [g/l]	0.45	0.45	1.23	1.19	1.35	1.34	1.40	1.40
P [g/l]	0.99	0.99	2.71	2.63	2.95	2.96	3.07	3.08
MP [%]	8	8	15	15	16	16	17	17

The final secondary product of process chain 2 would still contain substantial amounts of organics and nutrients and could therefore be treated with similar source separated streams with similar concentrations such as brown water (Table 41).

Remaining pharmaceutical residues in the secondary product would be in the range of less than 2 % of the initial concentrations in undiluted urine. An energy consumptive additional treatment step for removal or pharmaceutical residues would not be necessary.

The range of nutrient recovery and micropollutant removal rates of process chain 2 are displayed in Table 40. The total N-recovery, including rectification, evaporation and crystallization raged in all scenarios from 94.8 to 96.1 %, P-recovery by evaporation and crystallization ranged from 89.3 to 90.3 %, while micropollutants would be removed by the process combination of rectification, evaporation and crystallization by 98 %. In this scenario building, N-recovery by rectification was optimized for the subsequent evaporation and crystallization. Higher N-recovery rates could be yielded resulting in a disbalance of N-P ratio in the subsequent processes and consequentially in lower P-recovery by crystallization.

Table 40: Nutrient recovery and micropollutant removal rates of process chain 2

	Min	Max
N-recovery	94.8%	96.1%
P-recovery	89.3%	90.3%
MP-removal	98.2%	98.2%

The total energy demand for process chain 2 is given in Table 42.

Table 41: Secondary product of process chain 2 after rectification, evaporation, and crystallization

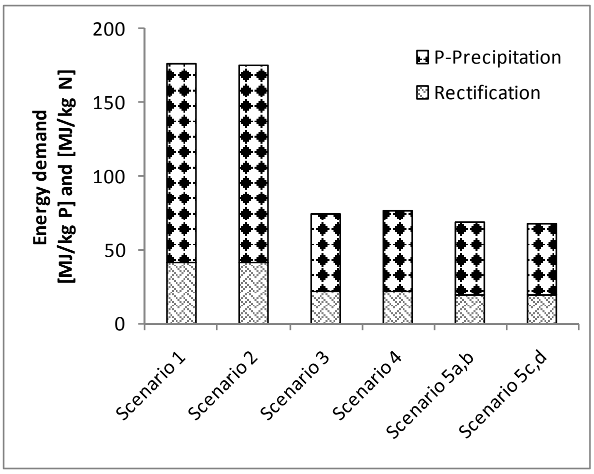
Scenario	1	2	3	4	5 a	5 b	5 c	5 d
Final secondary product = secondary product Evap + cryst								
Q [m^3/d]	1.21	3.66	16.9	26.9	551	1 756	678	2 163
C [g/l]	1.22	1.22	2.19	2.13	2.27	2.28	2.33	2.34
N [g/l]	0.11	0.11	0.28	0.27	0.30	0.30	0.31	0.31
P [g/l]	0.02	0.02	0.06	0.06	0.06	0.06	0.06	0.06
MP [%]	0.81	0.81	1.55	1.50	1.62	1.63	1.68	1.68

Table 42: Energy demand for process chain 2

Scenario	1	2	3	4	5 a	5 b	5 c	5 d
Rectification [MJ/kg N]	43	43	22	23	21	21	21	21
Evaporation, Crystallization [MJ/kg P]	960	960	390	400	370	360	350	350

E.3.4. Discussion of the specific energy demand

For the presented nutrient recovery processes a general energy demand per treated volume was assumed (Table 30). As can be seen in Table 36, Table 42, Figure 55, and Figure 56, higher concentrated substrates, depending on the scenario, lead to lower specific energy demand per nutrient load.



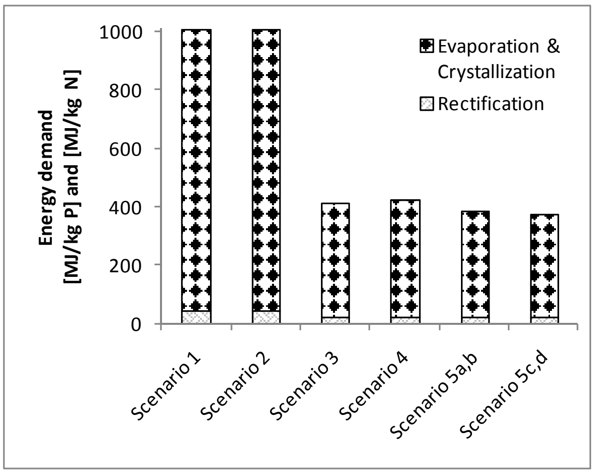


Figure 55: Energy demand for process chain 1

Figure 56: Energy demand for process chain 2

Compairison of product values and specific energy demand for production

The efficiency of the two different treatment chains can also be estimated by comparing product values and monetarized energy demand for production.

Based on commercially available fertilizer products and prices for 2005 *Dockhorn* (*2007*) evaluated specific prices for the individual contained elements to obtain values of new products gained from e.g. new sanitation concepts. This estimation was revised by *Esemen & Dockhorn* (*2009*) based on data from 2008, a year with very high prices for fertilizers. Although fertilizer prices are expected to rise further within the near future, only 50 % of the calculative product values from *Esemen & Dockhorn* (*2009*) were used for the present compairison of product values and energy demand for production and are given in Table 43, due to strong price fluctuations on the fertilizer market.

Table 43: Calculative product values

NH_3-solution	0.51	€/kg N	(*
MAP	382	€/t MAP	(*
Crystals	250	€/t	(**
Mg^{2+}	1.55	€/kg	(*
Primary energy	0.015 (0.01)	€/MJ	(***

*) based on 50 % of *Esemen & Dockhorn (2009)*
**) assumption based on 50 % of *Esemen & Dockhorn (2009);*
***) derived from *BMWi* (*2010*) and *Destatis* (*2010*);
0.01 €/MJ assumed for scenario 5a–d

According to additional literature sources market prices for MAP range from 100 to 250 €/(t MAP) (*Herbst 2008*; *Montag 2008*; *Wilsenach 2006*). Based on these data and on a comparison of the data 2005 and 2008 of *Dockhorn* (*2007*) and *Esemen & Dockhorn* (*2009*), the values for the P-products MAP and crystals might be overestimated, values for a NH_3-

solution on the other side are underestimated. The evaluated values can therefore only be used for rough orientation.

Energy special tariffs were assumed for the scenarios with high energy demand due to high coverage. For converting energy consumption an efficiency factor of 0.9 was integrated.

Efforts for transport were not included in the compairison of product values and energy demand for production of the two process chains. The energy demand for transport would be in the range of 10–15 % of the energy demand for the treatment processes.

With the assumed estimated product values rectification and MAP-precipitation could be an economical solution for nitrogen recovery in scenario 3 and higher (Figure 57). While reductions of energy prices were assumed for scenarios 5a to 5b, the positive total monetary benefit per cubic meter in scenarios 3 and 4 is mainly due to higher concentrations in the collected substrate leading to higher product outputs. At higher product values (70 % instead of 50 % of *Esemen & Dockhorn* (*2009*)) process chain 1 could be economical in all scenarios.

With the assumed estimated product values, rectification and MAP-precipitation could only be an economical solution for nitrogen recovery in scenario 5 (Figure 58). At higher product values (72 % instead of 50 % of *Esemen & Dockhorn* (*2009*)) process chain 2 could be economical already in scenario 3 and higher.

Due to the high energy demand of evaporation and the small output of crystals from a subsequent crystallization, the combination evaporation – crystallization can be recommended only in situations where excess energy or low cost energy is available. The costs for magnesium salts could be spared while obtaining a similar product.

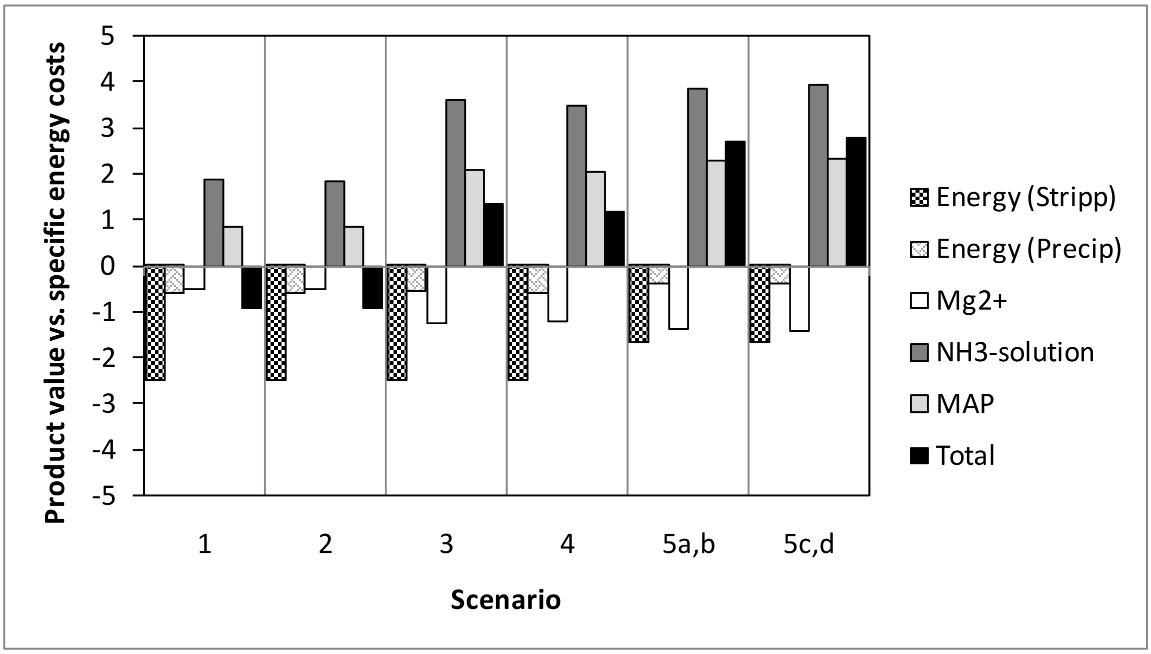


Figure 57: Product value versus energy costs for production chain 1 [€/m³]

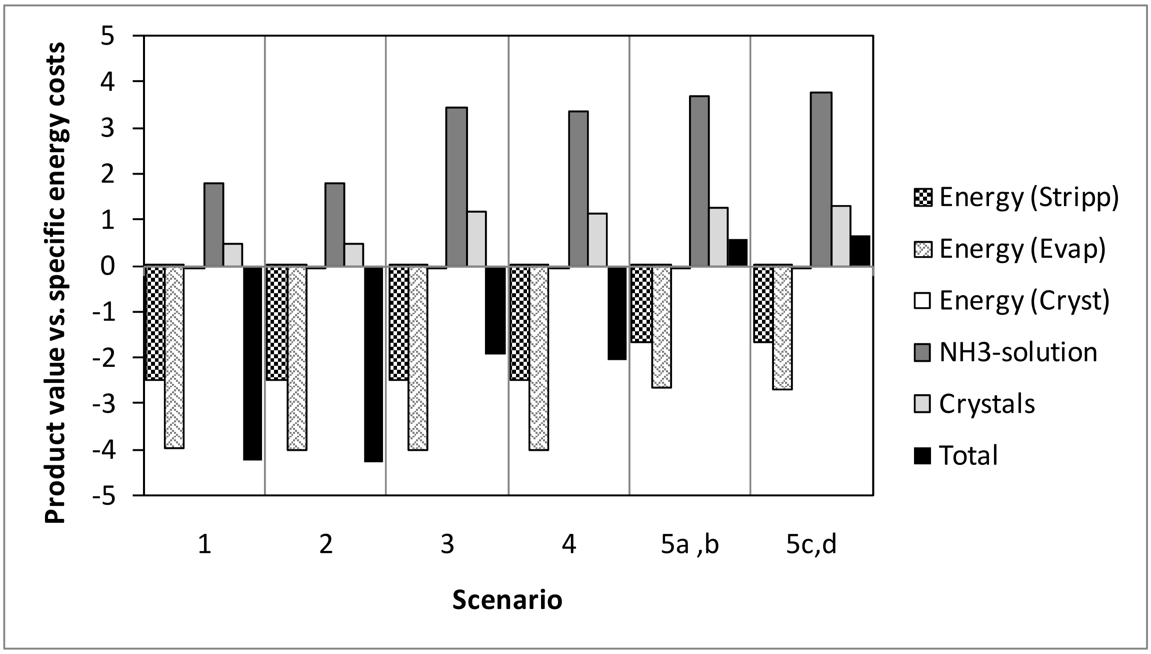


Figure 58: Product value versus energy costs for production chain 2 [€/m³]

E.4. Intermediate discussion

Based on the current situation where different elements of new sanitation systems such as waterless urinals are implemented already, five different scenarios were set up to investigate the systematic chain of collection, transport and treatment. The scenarios varied by location of collection (stepwise additional locations were included), type of collection device and by coverage. Scenario 1 could be implemented right away, scenario 2 and 3 could be implemented within a short time frame and at little extra effort e.g. for collection tanks. For scenario 4 the implementation of urine diverting toilets would be needed in office buildings, etc., and for the four sub-scenarios under scenario 5, a large number of private households would needed to be equipped with waterless urinals, urine diverting toilets, and collection tanks. The sub-scenarios under scenario 5 were varied both in scale and in separating efficiency of the urine diverting toilets.

With the different scenarios a stepwise integration of a system for source separated urine is described, based on the motto “change without catastrophe” (*Londong 2008*). The system is not seen as a direct competitor to the conventional system, but more as an additional tool for a stepwise and continuous improvement of the existing system. In areas without sufficient sanitation infrastructure, systems including source separated urine could be thought of as immediate solutions.

The different collection locations and the different collection devices had a significant influence on the quality of the collectable substrate. From public places, concentrations can be expected to be as low as 40 to 45 % of concentrations of average urine referred to in literature e.g. (*Meinzinger & Oldenburg 2009*), since the substrate itself is low concentrated. Waterless urinals are supposed to collect pure urine. However, dilution effects from cleaning water could occur. The implementation of urine diverting flushing toilets will currently lead to dilution effects of the collected substrate, since separation efficiency is often not functioning satisfyingly, allowing flushing water to enter the collection tanks for urine.

According to the results of chapter C.5, transport efficiency in this scenario framework was mainly influenced by coverage. For smaller systems with up to 10 000 people (scenarios 1 to 3), the involved energy demand would be around 54 MJ/m^3 (15 kWh/m^3). By tour optimization and increase of the number of main collection and treatment points, the energy demand for collection and transportation on a vehicle base could be reduced to 14.4 MJ/m^3 (4 kWh/m^3) transported substrate.

Because of the rather small amounts to be treated in scenarios 1 and 2, a pilot scale plant similar to the one used in the present study would be sufficient. This pilot plant should be connected to a steam generating process, since small but efficient steam generators are currently not commercially available. For scenarios 3 and 4, commercially available steam generators (starting at 160kg steam/h) could be used for the rectification process. However, synergetic effects from other industries such as industries involving combustion processes could be used, especially since e.g. waste combustions plants use ammonia solutions for flue gas scrubbing. The demand of two plants in Hamburg is between 400 t/a (MVA Stellinger Moor) (*Franck 2009*) and about 1500 t/a (Borsigstraße) (*Seger 2009*).

Kollbach & Grömping (*1996*) are recommending flow rates of more than 200 l/h for economical operation of equivalent steam stripping plants. This could be reached in scenarios 2 and higher.

Energy costs for the treatment by process chain 1 could be covered by the assumed product values in scenarios 3 and higher. For process chain 2 costs could be covered in scenario 5. In both cases a positive outcome would mainly be due to collection of high concentrated substrate.

The advantage of process chain 1 lies in the cost-benefit under the current assumptions. However, additional chemicals (magnesium salts) are needed. In cases where alternative energy sources (solar etc.) are available but no funds for additional chemicals, process chain 2 could be the favorable option for large densely populated areas.

Implementation potential is seen especially in areas with very high population density such as districts with many extreme high-rise buildings. As an extreme example, the never realized vision “The Mile-High Illinois” of Frank Lloyd Wright from 1956 is named (*Raschke 1996*). The building was supposed to have about 1.71 million m^2 usable floor area which could host probably far more than 10 000 people. The order of magnitude would therefore be in between scenarios 3 and 4. Although “The Mile” was only a vision, many projects since then have led to the assumption that the era of extreme high rise building has not ended while the question of a sustainable water infrastructure of these buildings can probably be questioned in many cases. It is believed that urine separation could be one part improving the ecological sustainability of such building projects.

F. Summary and final discussion

Objectives and motivation

Different objectives can be fulfilled by urine separation systems. They can contribute to prevent the release of nutrients into the environment in areas with insufficient wastewater management systems, they can lead to nutrient reductions at wastewater treatment plants with the consequence of reduced energy demand and reduced required area, and they can be beneficially combined with anaerobic treatment steps in new sanitation systems, since nutrients are not reduced by this technique. At the same time nutrients in urine can be recovered for usage in agriculture and/or industry.

With this motivation, aspects and potential of a source separated system for urine were investigated targeting industrial scale treatment in an urban context, complementing existing studies on urine separation and decentralized treatment.

The starting point of this study was the current situation in the city of Hamburg, Germany, where a larger collection system for urine has already been established by the implementation of waterless urinals equipped with collection tanks.

Collection and substrate quality

Within this study a broad measuring campaign was realized giving reliable information on concentrations of nutrients and other parameters such as pathogens and micropollutants in terms of pharmaceutical residues. Concentrations of nutrients and similar substances were in the range of 45 % of literature values. From concentration ratios it could be concluded that ammonia losses due to out-gassing most likely did not occur, while about 10% of the phosphorous was lost probably by uncontrolled precipitation processes in the collection system. Concentrations from other collection systems will differ from the values of public urinals based on collection device (separation ratio, dilution by flush water) and user habits (e.g. diluted urine due to drinking large quantities).

Transport

With the current collection system based on 11 public urinals with collection tanks, about 1 m^3/d of urine is collected but currently not used. Based on this current system which is relying on a wheel based transport if urine is supposed to be used, a possible scenario was developed on how the current system could be improved and expanded. It was assumed that within the next steps of development more locations could be integrated, not necessarily close to each other. To gain information on the possibility and limitation of a wheel based pick up and transportation system within a city like Hamburg, a transport scenario was developed similar to a waste disposal and pick up system. Since the amount of urine per person is comparable to the amount of waste per person, equivalent logistics can be used. The required energy for transport would be in the range of 10 to 15 % of the energy required for treatment. However, the higher the number of connected contributors to one single collection point, the more efficiently transport could be managed.

Treatment

For the treatment options, techniques were investigated allowing a semi-central treatment on an industrial scale versus the often discussed decentralized onsite systems.

The main goals for the treatment of separately collected urine were:

- nutrient recovery;
- to improve the handling of the substrate; and
- to remove contained pharmaceutical residues, synthetic hormones andmore.

All of this is possible. However, no process suits every purpose. In Table 44 on page 108 aims and beneficial side effects of each process are listed as well as beneficial pre-processes and necessary follow-up steps.

Rectification proved to be technically feasible. While the process is well known from other industrial applications, the behavior of the urine substrate and the potential of treatment efficiency were unknown, so far. The main advantage of steam stripping (rectification) in comparison to air stripping is the high temperature, where a high ammonia mass transfer is guaranteed. During the tests at pilot scale, not only did the rectification process work satisfyingly, it also could be shown that a highly concentrated product of 12 % ammonia solution could be produced (20 to 30 liters of ammonia solution could be obtained from every cubic meter of treated substrate) while the initial substrate could be depleted of nitrogen by more than 97 %. The high quality of the primary output (ammonia solution) was due to fractionated condensation step combined with the stripping process. 20 % ammonia solutions could probably be achieved when treating substrate from other locations with higher nitrogen content by an optimized rectification process.

Both objectives to reduce the volume and yield a highly concentrated nutrient solution could be reached through the use of evaporation, whereby more than 50 fold concentrates could be obtained (20 l concentrate from 1 m^3 substrate). While phosphorous was completely recovered in the concentrate, nitrogen losses due to ammonia volatilization were high. Acidification of stored urine cannot be an economical solution because of the high buffer capacity of stored urine. Steam stripping, rectification, biological pretreatment, or acidification of fresh urine are some options to avoid excessive losses into the distillate during evaporation. The concentrate could be used as a highly enriched nutrient solution or further processed by crystallization to gain an easy to handle solid product for agriculture. The distillate would need further treatment to remove COD and depending on the circumstances, to either remove or recover nitrogen.

Crystal formation due to concentrations above saturation level was observed at storage of the concentrates and during a lab scale crystallization test. This process could be enhanced by technical means, yielding pure and dry products. Self crystallization occurred in highly concentrated urine solutions at concentration factors of 30 and above. About 90 % of the phosphorous could be recovered by crystallization. Thus per cubic meter of low concentrated source separated urine, about 250 g of phosphorous could be recovered. The simple lab procedure should have left space for further optimization.

Adding one more aspect to the many studies on the very efficient way for phosphorous recovery, the MAP-precipitation: Pretreatment by steam stripping did not enhance the process as expected. However, low pH values obtained by steam stripping could be regulated by the precipitant since the buffer capacity was also lowered by the stripping process due to removal of ammonia and carbon dioxide.

The two thermal treatment processes also had an impact on pharmaceutical residues. Concentrations of ß-sitosterol and pentoxifylline were affected the most by the stripping/rectification process itself. More important might be the generally higher removal rates in the subsequent ozonation after rectification/steam stripping.

During evaporation only carbamazepine, phenazone and pentoxifylline were found in a 12 fold concentrate. Especially phenazone proved to be very stable under evaporating conditions. Even in higher enriched concentrates phenazone is likely to be detected in large quantities. Ibuprofen and to a small extent carbamazepine could be detected in the distillate, thus they can be considered volatile.

With the ozonation of stored urine, all pharmaceutical residues could be removed. Again, concentrations of phenacetin and diclofenac were affected the most. Acidification of stored urine did not lead to significant improved removal of pharmaceutical residues, probably because of ammonium present as competitor for the direct ozone reaction. However, acidification of N-depleted substrate did show a significant increased removal for bezafibrate, ß-sitosterol, and pentoxifylline. The formation and fate of metabolites was not investigated and is still unclear.

The energy demands of the investigated nutrient recovery processes were in comparable ranges to nutrient removal at conventional wastewater treatment plants and fertilizer production. Also, the energy demand of the pharmaceutical residue removal was in a comparable range as in conventional wastewater treatment concepts. At the same time, space requirements for the ozonation of urine would be drastically lower than for the ozonation of wastewater treatment plant effluent.

Implementation potential

To describe a possible stepwise integration of a system for source separated urine and to investigate the systematic chain of collection, transport and treatment, five different scenarios were set up based on the current situation. The scenarios varied by scale and substrate quality.

Because of the rather small amounts to be treated in scenarios 1 and 2, a pilot scale plant similar to the one used in the present trials would be sufficient. Scenarios 3 and 4 could be designed as small stand alone rectification plants with integrated steam generators.

Costs for the treatment by process chain 1 could be covered by the assumed product values in scenarios 3 and higher. For process chain 2, costs would be covered in scenario 5, both mainly due to coverage of high concentrated substrate. Higher product values and lower energy costs e.g. due to usage of excess energy could make process chain 2 also feasible at lower coverage with collection of a lower concentrated substrate.

With this case study it could be shown that efficiency improves with increasing coverage, mainly due to concentrations and therefore a more efficient process operation. Consequentially collection of a high concentrated substrate is one of the key points of an economic urine separation system.

Costs of urine treatment in general

For a detailed statement regarding costs and value of the obtained products several economical tools are needed such as a market evaluation, analysis of competitive products, cost-benefit analysis including sustainability indicators and factors respecting environmental protection. Assessments of the economic sustainability of urine separation systems are hard to find in scientific literature. Reliable cost data of large-scale separation systems are not available. However, cost estimations and data from pilot projects can be used as a basis for cost projections.

Oldenburg et al. (2007) compared different separation systems with a conventional system by cost calculation based on *LAWA* (*1998*), stating that operational costs can be lower with separation systems, whereas total project costs are presumably comparable or higher. Previous calculations resulted in economic benefits for separation systems. However, data for costs were based on first estimations *(Peter-Froehlich et al. 2004)*. According to both publications local boundary conditions (e.g. population density, energy prices, water prices etc.) can influence the results of the cost comparison significantly.

Dockhorn (2007) made a detailed cost analysis of a large urban sanitation system (350 000 inhabitants) and identified economic benefits for separation systems, mainly due to reduced costs for treatment of the remaining wastewater and saved costs for mineral fertilizer. These economic incentives should also be visible during a gradual implementation of separation systems into the existing systems.

In general, projects established in recent years have shown that separation systems have the potential to yield economic benefits by reducing costs for the wastewater treatment plant and by substituting mineral fertilizer. Rising prices for energy and fertilizer will certainly increase the economic benefits of resource recovery from wastewater in the future. At the moment, separation systems seem to be at least comparable to the conventional system regarding the total costs.

Also, the economic sustainability of a urine separation system has still to be proven in a full-scale case study. In this study, focus was directed towards technical aspects of realization of urine treatment on an industrial scale. Energy requirements and quantities of additional chemicals or other required input material were measured, and discussed for each process. These data can be used for an estimation of individual process costs. Since urine has an energy potential of at about 900 MJ/(m^3 urine) (*Dockhorn & Dichtl 2004*; *Maurer et al. 2003*; *Niederste-Hollenberg 2003*; *Patyk & Reinhardt 1997*) the energy demand of the investigated processes were in comparable ranges with nutrient removal at conventional wastewater treatment plants and fertilizer production.

As presented, the costs of separate collection and treatment of urine can be expected to be in a comparable range with conventional methods since the processes investigated in this study are similar to the ones used in conventional wastewater treatment and fertilizer production using fossil resources. While the scale of the fertilizer industry using fossil resources currently cannot be reached, the benefits regarding sustainability and environmental protection of separate collection and treatment of urine have to be included in a cost analysis; e.g. reduced accumulation of nutrients in surface waters and costal ocean regions especially in areas with inadequate wastewater treatment, and reduced requirements for nutrient removal in conventional wastewater treatment plants. Additionally the benefit of spared fossil resources and of a more sustainable system with closed loops has to be noted.

Alternatives to urine separation

While urine separation and treatment of separated urine is seen as one part of sustainable water and resource infrastructure, different alternatives are available to fulfill the goals stated in chapter A.4.1:

For reduction of the nutrient load generated from wastewater, well established techniques are developed and described e.g. in standard literature by *Metcalf & Eddy* (*2003*) or in detail e.g. regarding reduction of phosphorous from wastewater treatment plants by *Barjenbruch & Exner* (*2009*). These are targeting mainly an implementation in existing systems or the design according to traditional wastewater management.

Additionally, different approaches targeting more resource recovery in the field of sanitation exist. However, regarding the “recovery of nitrogen via atmosphere” the “biological nutrient removal processes combined with the modern industrial ammonia synthesis process (Haber-Bosch) is seen in many cases just as (or even more) energy efficient as direct ammonia recovery” (*Wilsenach 2006*).

There are currently many approaches for large-scale recovery of phosphorous from wastewater, sludge and ashes. Amongst others, v*on Horn* (*2007*) investigated the phosphate precipitation from sludge liquor from return sludge while *Montag* (*2008*) described the precipitation from liquor of excess sludge. In an international survey of experts, the topic of P-recovery from wastewater and some of the main advantages and disadvantages of the different approaches were evaluated by *Sartorius et al.* (*2011*). It is believed by the expert community “that P-recovery will become an established process over the next 20 years in industrialized countries for economic reasons” (*Sartorius et al. 2011*). Separation of material flows (e.g. urine separation) was seen by nearly half of the experts as an option.

For the removal of micropollutants from wastewater, advanced wastewater treatment techniques are available such as membrane filtration and oxidative techniques (*Abegglen et al. 2009*). Efficiency and energy demand are often depending on the large volumes to be treated.

A completely different sanitation approach is the concept of blackwater loop or also called LooLoop (*Braun et al. 2008*). Here the toilet wastewater is treated by MBR and subsequent hygienization steps allowing a reuse as flushing water. Nutrients brought into the system accumulate and can be recovered in a small volume. The process was investigated by *Lindner* (*2007*) and is further optimized by *Antholz et al.* (*2010*). Due to its water saving characteristics, it is well suited as a resource efficient alternative especially as a decentralized high tech solution in water scares regions.

The terra preta concept which is currently investigated more in depth is integrating sanitation, bio-waste, and agriculture on a settlement level (*Factura et al. 2010*). It is involving a lacto-fermentation step of concentrated wastewater mixed with biochar made from bio- and garden-waste. The obtained product shall be usable as a nutrient rich organic soil conditioner with high water retention capacities. According to *Krüger & Scheinemann* (*2011*) pathogens can be reduced by the lacto-fermentation process. The high content of organic matter in the obtained product becomes important, since many types of highly intensive agriculture are not only demanding sufficient nutrient supply but are also requiring organic rich soils. Therfore integration of urine (within the blackwater without separation) into composting processes balanced e.g. by woody materials such as in the terra preta concept needs now to be further investigated.

Summary

For each situation there should be an evaluation of adequate solutions.

Urine separation is already realized in many cases by the implementation of waterless urinals in pubic and semi-public toilets. In some cases even the collection of large quantities has been performed. In new buildings or within rehabilitation measures of existing buildings more collection could be implemented.

Concentrations of the collected substrate are often low. To counter this phenomenon dilution effects and nutrient losses within the systems should be minimized where possible.

Treatment for nutrient recovery is technically feasible on a larger scale and becomes economically more efficient when treating higher concentrated substrates due to higher valued product outputs, or once product values rise due to changing market situation and/or energy prices.

For implementation of larger urine separation and treatment systems, areas with a high population density such as high rise building districts seem to be promising to reduce transportation efforts.

Overall, the raw material 'urine' proved to be a very good resource for products usable in agriculture and industry. Combined with further demonstration, an up-scaling of the pilot plants seems promising.

Table 44: Main factors of investigated processes

Process	**yield; significant side effects**	**requirements; beneficial conditions**	**possible synergetic effects**	**beneficial pre-process**	**necessary follow up regarding**	**energy requirements [MJ/m^3]**	**obtained product**
Rectification / Steam stripping	N-recovery; pH reduction; reduction of buffer capacity	hydrolyzed substrate needed	energy for heating process	storage. MAP-precipitation	P-recovery . PhaR thereafter with grey water	150	NH_3-solution
Evaporation	volume reduction; reduction of most PhaR	low pH for reduction of loss of ammonia	energy for heating process	steam stripping; other pH reducing processes	post processing of concentrate and distillate; PhaR	200	Concentrated nutrient solution
MAP-precipitation	P-recovery	low buffer capacity		steam stripping and evaporation possible. but not necessary	N-recovery. PhaR thereafter with grey water	~ 30	Struvite
Crystallization	P-recovery	high N and P concentrations	cooling after concentration processes at high temperatures	evaporation	post processing of depleted substrate (N. COD. PhaR)	~ 20	P-crystals
Ozonation	PhaR-removal	reduction of NH_3. COD. and pH	radical reaction increased at high temperatures (**	steam stripping acidification	nutrient recovery and COD removal	115	Substrate free of PhaR
Storage	hygienization/ stabilization				N. P. COD. PhaR treatment / usage	-	Substrate free of pathogens

*) high amounts of required adsorbent in lab-scale

**) was not investigated

***) very high energy requirements in lab set-up

G. Conclusion and outlook

In the world of sanitation systems new approaches of solutions for tomorrow are becoming more and more relevant. A trend towards decentralizing structures, scaling down large processes and technical improvements of on-site systems can be well observed within the scientific community and in the world of responsible stakeholders such as decision makers and operators. However, process efficiency and operation management is still often linked to a specific minimum scale, giving industrial scale processes a certain advantage.

While on-site systems are a solution especially for areas with low to medium population density, a future option for those with medium to high population density could be new systems with a combination of decentralized and centralized (onsite and offsite respectively) elements tailored to the particular circumstances. The aim hereby is to combine the advantages of both sides by implementing new and well-known elements to obtain economically efficient, reliable, and safe systems, remaining flexible enough to be able to react to changes of the boundary conditions, and integrating resource recovery for lasting sustainability. Source separation has become more and more accepted in the world of wastewater management as a good option to fulfill these requirements.

A system for source separated urine with industrial style treatment in an urban context is therefore one important part of the innovative approach of new sanitation concepts.

Waterless urinals are widely used especially in public and semi public areas, while urine diverting toilets are only used to a larger extend within different research projects. The collection of urine from waterless urinals for example is already realized in larger scale by the city of Hamburg, driven by economical and environmental reasons. The same concept could be applied in other cities as well. In form of transportable urinals (partially already applied, mostly without further use of the collected material), the concept of waterless urinals with collection tanks could also easily be applied in semi-public areas at large events with lots of people getting together. From such installations enough material could be collected for future treatment at pilot scale or even small industrial size.

Since the current collection points are widespread, and since during the coming years a satellite-like expansion of collecting systems can be expected, transport from these collection points to treatment facilities can be realized by truck based pick-up. Long piping systems would lead again to more inflexible systems with – in case of urine – probably higher demand for maintenance. Truck based pick up systems and their logistics are well investigated. Since the amount of urine per person and time is similar to the amount of solid waste per person and time, parameters can be applied from the area of waste collection and maybe even combined. Future transportation systems could later be implemented in infrastructure channels which might become the state of the art in large densely populated areas some day.

Marketable products can be extracted from separately collected urine on an industrial scale e.g. by rectification and evaporation with subsequent crystallisation. Pharmaceutical residues are reduced by thermal treatment and can be completely removed by oxidative processes such as ozonation.

A market analysis was not conducted, although energy requirements and quantities of required input material were discussed and counterbalanced with obtainable product values. In most cases the overall costs for production of pure substances or fertilizer products from urine is expected to be in a similar range compared to production from fossil materials without the risk of rising prices due to scarcity.

The combination of rectification and MAP-precipitation would be the most economic one in the current context. High valued products could be achieved in the form of highly concentrated ammonia solution and struvite. However, additional chemicals in the form of magnesium salts are needed but also further used in case of agricultural utilization. In cases where alternative energy sources (solar etc.) would be available but no monetary sources for additional chemicals, rectification combined with evaporation and subsequent crystallization could be the favorable option for large densely populated areas. In case micropollutants should be removed, an additional treatment step such as efficient ozonation should be added to the process of chain rectification and MAP-precipitation since reduction rates were lower compared to ozonation of untreated urine. This removal of micropollutants could be conducted together with other treated wastewater streams.

Following the idea of ecological economy, an integration to other industrial thermal processes such as e.g. thermal waste treatment should be considered for synergetic effects in combination with rectification and evaporation.

The results have a major influence for reaching the aim to allow more effective wastewater treatment systems with obtaining valuable products e.g. fertilizers.

While no process allows nutrient recovery and removal of micropollutants in the form of pharmaceutical residues at drastically lower economical conditions than the conventional treatment system, the investigated processes can be seen as one piece of a puzzle for new sanitation systems and allow their implementation where applicable.

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Appendix

List of appendix

List of appendix tables

List of appendix figures

Appendix 1. ad Collection

Appendix 1.1: Location

Locations for urine collection in Hamburg in 2009

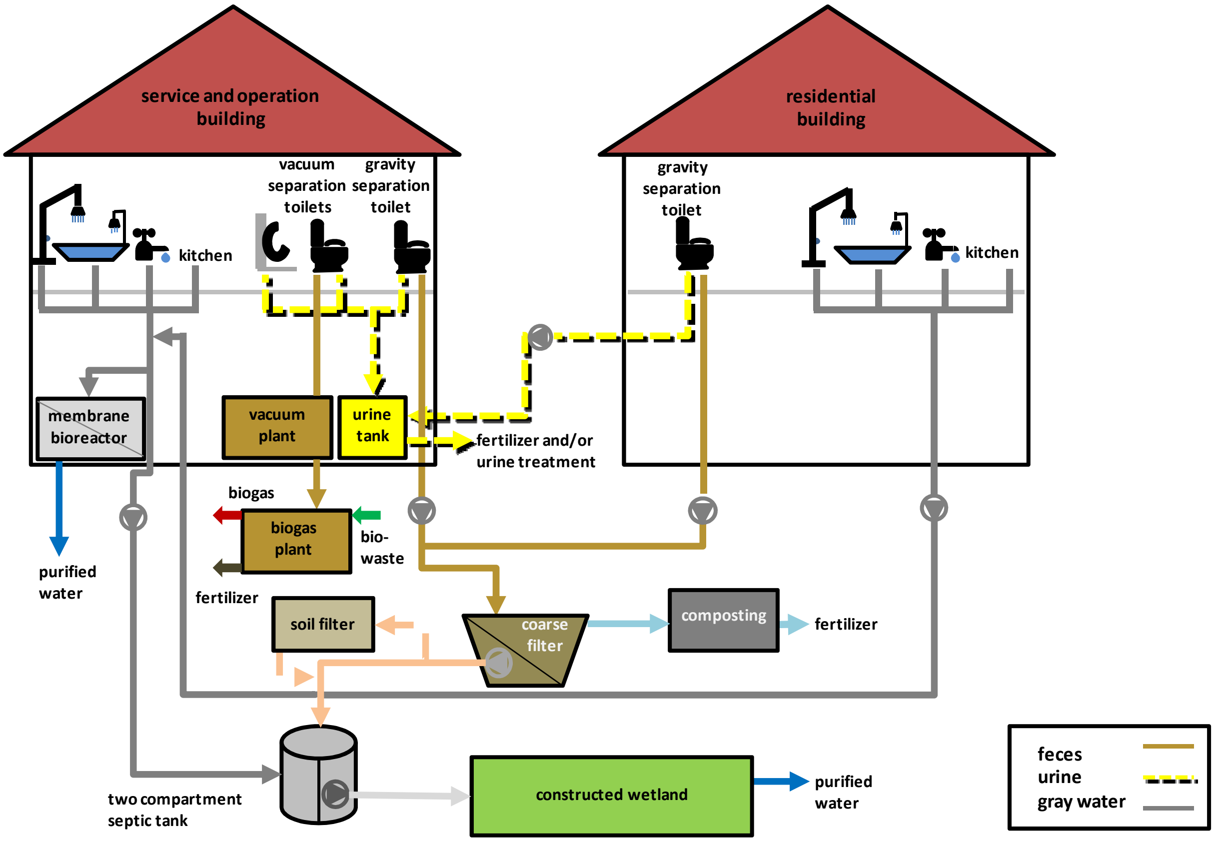
Appendix table 1: Details to public water urinals in Hamburg

	Location	Address	Initial volume [$l\ d^{-1}$]	Maximum volume [$l\ mo^{-1}$]	Full after [d]
L1	**Beim Trichter, St. Pauli**	Corner Beim Trichter Reeperbahn 20359 Hamburg	600	2700	31
L2	**Hamburger Berg, St. Pauli**	Corner Hamburger Berg Reeperbahn 20359 Hamburg	200	2700	38
L3	**Hansaplatz, St. Georg**	South-West of Hansaplace 20099 Hamburg	44	2000	17
L4	**Reeperbahn No.7, St. Pauli**	In front of Reeperbahn No. 7 20359 Hamburg	200	2700	56
L5	**S-Bahn Othmarschen, Othmarschen**	Auf dem Statthalterplatz 22605 Hamburg	200	2700	56
L6	**Rottunde, Central station, Altstadt**	Corner Steintorwall, Mönckebergstr. 20099 Hamburg	138	640	17
L7	**S-Bahn station Allermöhe, Allermöhe**	Auf dem Fleetplatz 21035 Hamburg	600	2700	-*
L8	**S-Bahn station Nettelburg, Nettelburg**	Edith- Stein- Platz 21035 Hamburg	900	2700	-*
L9	**S-Bahn station Stellingen, Stellingen**	End of Lederstraße 22525 Hamburg	200	2700	70
**	**S-Bahn station Reeperbahn, St. Pauli**	Corner Pepermölenbek Reeperbahn. 22767 Hamburg			
**	**Alma- Wartenberg- Platz, Ottensen**	Corner Friedensallee Bergiusstraße. 22765 Hamburg			
**	**S-Bahn Elbgaustraße, Eidelstedt**	Corner Weidplan Elbgaustraße. 22523 Hamburg			

* not filled during sampling period; ** not included in sampling

Location for urine collection at the SCST demonstration site. Berlin-Stahnsdorf. Germany

Urine as substrate for the treatment processes was also collected from the SCST demonstration site in Berlin-Stahnsdorf, a separating system of the Berliner Wasserbetriebe (BWB) (Appendix figure 1). For description of the system see *Peter-Fröhlich et al.* (*2007b*). At the time of collection the office building was fitted with waterless urinals of different systems and separating vacuum toilets, a modified Roediger separating toilet. At the building about 20 people were constantly working. Also meetings and seminars took place that increased the number of contributors.



according to *Peter-Fröhlich et al. (2004)*

Appendix figure 1: Scheme of SCST demonstration system

Appendix 1.2: Analytics

Since urine has a very complex matrix and a certain composition change depending on the source. the analytical methods have to be selected carefully. Some test kits. which are suitable for household wastewater. fail on the measurement of urine because of its matrix.

Ammonia

Nitrogen was analyzed according to (*NORM 1983*).

The analysis of ammonia was performed by using distillation and titration (Büchi 321; Distillation Unit. Dargatz). The method has three main steps: distillation. absorption in acidic solution (H_2SO_4) and titration with a base (NaOH). The advantage of this method is that since ammonia is distilled and caught in another solution. the complex salt matrix of urine cannot interfere with the measurement. Urea is also caught with this method as ammonia. Especially for fresh urine this has to be noted. Since in this work stored urine was used. it can be assumed that all the urea is already converted to ammonia.

Phosphate

For Phosphorous a modified stannous chloride method (*Greenberg 1992*) was used in a standard addition process according to (*NORM 1998*). Additionally Dr. Lange Cuvette-tests (LCK 350) and PE-Optima 2000 DV OES with ICP were used.

Other Parameters

- TOC. TC: Autoanalyzer Analytic Jena
- TS (total solids): according to (*APHA et al. 2005*)
- AOX: according to DIN EN ISO 9562. AOX-System M 2000 C
- BTEX: Headspace-gaschromatography PE Turbomatrix 40 Trap
- quantification of Na. K. Ca. Mg. P. Cu. Zn. Fe. N_{tot}. and trace elements
    - PE Elan DRC II ICP-MS
    - PE-Optima 2000 DV OES with ICP
    - PE SIMAA AAS
- carboxylic acid and anions:
    - Dionex-ionchromatography

Laboratory Equipment

- pH: pH-electrodes for wastewater. WTW Microprocessor pH Meter pH 196
- Conductivity: WTW Conductometer LF 191
- Ammonia: Büchi 321; Distillation Unit. Dargatz
- Jar Test: with mixing speed regulation (100-1600 rpm)

Pharmaceutical Residues

All analyses regarding pharmaceutical residues were conducted by IWW Rheinisch-westfälisches Institut für Wasser Beratungs- und Entwicklungsgesellschaft mbH using HPLC-MS (*Butzen et al. 2005*).

Appendix table 2: Levels of detection of pharmaceutical residues in urine matrix

	LOD in urine matrix [μg/l]	LOD in aqueous solutions (* [μg/l]	LOD_urine/ LOD_aq	DDD [mg]	Additional information
16α-Hydroxyestron	10	0.02	500		Metabolite of Estrone
17α-Ethinylestradiol	50	0.02	2500	0.025–0.05 (**	Estrogen (main Estrogencomponent in hormonal contraceptive)
Acetylsalicylic acid	0.5	0.02	25	3000	Analgesic
Bezafibrate	0.5	0.02	25	600	Lipid-lowering agent
β-Sitosterol	2	0.01	200		Phytosterol with lipid-lowering effect
Carbamazepine	0.5	0.02	25	1000	Antiepileptikum. Depressionsprophylaxe
Clofibric acid	0.5	0.02	25		Lipid-lowering agent
Diclofenac	0.5	0.02	25	100	Antiphlogistikum
Estradiol	10	0.01	1000	0.025–0.1 (**	Natural Estrogen
Estriol	100	0.02	5000	1-4 (**	Estrogen (metabolite of Estradiols
Estrone	10	0.01	1000		
Fenofibrat	0.5	0.02	25	200–300 (**	Lipid-lowering agent
Fenoprofen	0.5	0.02	25		
Gemfibrozil	0.5	0.02	25	1200	Lipid-lowering agent
Ibuprofen	0.5	0.02	25	800	Antiphlogistic
Indometacin	0.5	0.02	25	100	Antiphlogistic
Ketoprofen	0.5	0.02	25	150	Antiphlogistic
Mestranol	5	0.01	500		Prodrug of 17α-Ethinylestradiol
Pentoxifylline	0.5	0.02	25	1200	Vasodilatatoriat
Phenacetin	0.5	0.02	25		No concession any more
Phenazone	3	0.02	150	1000–4000 (**	Analgesic

LOD: level of detection; DDD: defined daily dose ; *) e.g. condensates. distillates; **) No information on DDD available. Value derived from commonly described dosages.

Appendix table 3: Concentrations in fresh human urine (literature values)

		[1]	[2]	[3]	[4]
TN	[g/d]	9.19–11.5	7.0–25.0	5.2–9.6	
P	[g/d]	0.8–2.0	0.5–1.5	0.5–1.1	1.00
S	[g/d]	1.24–1.49			
K	[g/d]	1.6–3.9	1.3–4.0	2.5–3.6	2.50
Ca	[g/d]	0.182–0.238	0.25–0.3		
Mg	[g/d]	0.107–0.131	0.1–0.2		

[1] (*Ciba Geigy AG 1977*); [2] (*Hofstetter & Eisenberger 1996*); [3] (*Jocham & Miller 1994*); [4] (*Alken & Walz 1998*)

Appendix 2. ad Transport

Appendix table 4: Population, area, and density of districts in Hamburg (Statistische Daten 2008)*

District	**Inhabitants [pe]**	**Area [km^2] in 2002**	**Population density [pe/km^2]**
Altona	242.560	80	3.130
Bergedorf	118.280	150	760
Eimsbuettel	244.350	50	4.880
HH-Mitte	226.750	110	2.120
HH-Nord	277.150	60	4.790
Harburg	200.320	160	1.250
Wandsbek	402.960	150	2.730
total	**1.712.364**	**755**	

Appendix table 5: House distribution in Hamburg (Statistische Daten 2008)*

District	**Buildings**	**Street [km]**	**[pe/ km]**	**Buildings/ km Street**
Altona	34.450	640	380	54
Bergedorf	19.640	480	247	41
Eimsbuettel	29.420	480	509	61
HH-Mitte	19.150	640	354	30
HH-Nord	29.610	540	512	55
Harburg	29.800	790	254	38
Wandsbek	69.580	1.010	400	69
total	**231.650**	**4.580**		

* Data collected by and cited of *Grünauer* (*2007*)

Appendix table 6: Assumption of number of main collection and treatment points depending on coverage

Coverage	Number of main collection and treatment points (n_{ctp})	Reduction factor of average tour distance ($\sqrt{n_{ctp}}$)
0.5–25 %	1	1
26–50 %	2	1.41
51–80 %	3	1.73
81–100 %	4	2

*Detour factor for S1: 1.4; detour factor for S2: 1.1

Appendix table 7: Assumption of time constraints during pickup

Average speed	$v_{ave.}$	30 km/h	
Routing time per tour	t_r	0.5 h	
Time per connection depending on vessel size	$t_c(v)$	0.05 m³	5 sec
		0.2 m³	10 sec
		0.5 m³	10 sec
		2.5 m³	20 sec
		10 m³	30 sec
Time per suction depending on collection vessel size[10]	$t_s(v)$	0.05 m³	1.0 sec
		0.2 m³	2.0 sec
		0.5 m³	6.0 sec
		2.5 m³	30 sec
		10 m³	60 sec

---

[10] derived from suction rates between 1600 and 2200 cbm/h of nowadays suction vehicles (source: http://www.mosbauer-gmbh.de/mosbauer-maschinenpark.php)

Appendix table 8: Number of persons involved and to be collected volume per day per assumed coverage

coverage [%]	No Persons [-]	Volume/day [m³/d]
0.1	*1 781*	2.45
1	*17 807*	24
10	*178 075*	245
50	*890 374*	1 224
100	*1 780 748*	2 449

Appendix table 9: Energy parameters

average fuel consumption	0.32	l/km
specific heating value diesel	42.5 11.8	MJ/l kWh/l
primary energy factor for fuel	1.1	-
CO_2-production from fuel combustion	0.296	kg CO_2/ kWh fuel

Data based on *IFEU & SGKV (2002), Geitmann* (*2010*), and *Krespach* (*2011*)

The number of collection vessels and the number of required collection vehicles resulted in a sum of investment costs per scenario and coverage. Labor costs of the staff and fuel costs resulted in a sum of operation costs per year.

From the consumed amount of fuel the required energy for transport was derived per scenario and coverage. Energy parameters are displayed in Appendix table 9.

Appendix 3. ad Treatment

Appendix 3.1: ad Rectification / stripping

Appendix table 10: Henry coefficient for ammonia, dimensionless, derived from *Sander* (*1999*) and *Arevalo* (*2000*)

	Sillen and Martell (1964)	Robinson and Stokes (1970)	Wilhelm et al. (1977)	Edwards et al. (1978)	Hales and Drewes (1979)	Chameides (1984)	Holzwarth et al. (1984)	Hoffmann and Jacob (1984	Dasgupta and Dong (1986)	Dasgupta and Dong (1986)	Clegg and Brimblecombe (1989)	Dean (1992)	Van Krevelen et al. (1949)	Bone et al. (1983)	Kavanaugh and Trussell (1980)	Lange (1979)	Arevalo (2000)
H_e [-] at 0 °C	1.97E-04	2.04E-04	2.58E-03	1.85E-04	1.89E-04	2.00E-04		2.00E-04	2.07E-04	2.01E-04	1.85E-04	7.95E-04			1.77E-04	7.76E-04	4.10E-04
H_e [-] at 25 °C	6.93E-04	7.17E-04	4.09E-03	6.70E-04	5.38E-04	7.05E-04	5.2E-04	7.05E-04	7.30E-04	7.30E-04	6.70E-04	1.51E-03	6.6E-04	7.6E-04	6.81E-04	1.48E-03	7.60E-04
H_e [-] at 50 °C	2.01E-03	2.08E-03	6.03E-03	1.99E-03	1.30E-03	2.04E-03		2.04E-03	2.12E-03	2.17E-03	1.99E-03	2.61E-03			2.13E-03		2.20E-03
H_e [-] at 75 °C	4.99E-03	5.17E-03	8.42E-03	5.07E-03	2.77E-03	5.08E-03		5.08E-03	5.26E-03	5.52E-03	5.07E-03	4.16E-03			5.67E-03		6.10E-03
H_e [-] at 100 °C	1.10E-02	1.14E-02	1.12E-02	1.14E-02	5.32E-03	1.12E-02		1.12E-02	1.16E-02	1.24E-02	1.14E-02	6.24E-03			1.32E-02		1.70E-02

For calculating the dimensionless Henry coefficient of ammonia at 25 °C Eq 27 was used with conversion factors according to *Sander* (*1999*).
For calculating the dimensionless Henry coefficient of ammonia at different temperatures Eq 28 was used combined with compiled data from *Sander* (*1999*).

Eq 27 $H_e^{25°C}$ [-] = 1/(k_H [M/atm]*24.46) at 298.15 K

Arevalo (2000) derived for the system water/ammonium in dependency of the temperature (T in °C) Eq 29.

Eq 29 f = 3709.1 * exp (-0.0416 * T)

Eq 28

$$k_H^T = k_H^{25°C} * \exp\left(\frac{-\Delta_{so\ln}H}{R}\right)\left(\frac{1}{273.15+T} - \frac{1}{273.15+25°C}\right)$$

Attention has to be paid to the units used and to the direction which is to be expressed whether the coefficient is representing solubility or volatility. While often k_H is used as Henry coefficient representing solubility, here H_c is used representing volatility:

Eq 30 $H_c = k_H = 1/k_H$

Appendix table 11: Input parameters for steam stripping lab scales tests

$\dot{L}_0$ [l/h]	$\dot{G}_0$ [l/h]	pH_0 [-]	X_0 [g/l]	T °C
0.51	1.41	8.9	4.7	20
0.48	0.85	8.9	4.7	20
1.08	1.13	11	4.7	20
0.34	0.99	11.2	4.7	20
0.37	1.29	11.4	4.7	20
1.61	0.74	11.6	4.7	20
0.51	1.01	8.2	4.7	93
0.61	0.97	10.8	4.7	93
0.67	1.09	11.4	4.7	93

Appendix table 12: Results of steam stripping lab scales tests

$\dot{G}_1$ [l/h]	$\dot{L}_N$ [l/h]	X_N [g/l]	Y_1 [g/l]	Y_t [g/l]	Y_1/Y_t [-]
0.67	1.25	0.13	2.93	0.82	3.57
0.50	0.83	0.18	2.96	0.99	3.00
0.70	1.51	0.74	5.90	6.26	0.94
0.85	0.48	1.41	1.34	1.45	0.92
0.99	0.67	0.38	1.60	1.66	0.96
0.78	1.57	1.36	8.44	6.76	1.25
0. 3	0.79	0.74	3.46	2.38	1.46
0.78	0.80	0.23	3.26	3.54	0.92
0.83	0.93	0.33	3.02	3.60	0.84

Results of lab scale plant

In total ammonia concentrations of more than 200 % of the substrate concentration could be reached in the condensed off-steam. Ammonia concentrations in the depleted substrate were between 5 and 10 % of the initial ammonia concentration. The pH of the treated substrate remained stable throughout the process which led to the assumption that the maximum of ammonia extraction was not reached.

As can be seen in Appendix table 11 all resulting NH_4-N concentrations in the condensate (Y_1) were in average 50 % above the expected calculated values (Y_t). Two values were three and more times higher as expected resulting in a standard deviation of 1.0. All of the higher values occurred in sets without increase of pH in the feed substrate. While according to Eq 17 the temperature should have a rather small effect on the achievable concentration in the condensate, pH has a larger influence. However, the $L^{\cdot}/G^{\cdot}$ ratio has the largest impact. Changes in flux of the feed streams during the sets could be a reason for misbalanced values.

ad Rectification in pilot scale plant

Appendix table 13: Additional **i**nput parameters for rectification pilot scales tests

	process time [h]	$\dot{L}_0$ [l/h]	$\dot{G}_0$ [l/h]	pH_0 [-]	X_0 [g/l]
set 1	3	82	33	8.9	4.2
set 1	1	72	20	9	5.8
set 1	3	82	34	8.9	5.8
set 1	2	61	21	8.9	7.4
set 2	1	24	24	9.3	7.4
set 2	1	27	19	9.8	6.6
set 2	2.5	28	17	10.2	6.3
set 2	1.5	29	16	9.3	7.4
set 3	2.5	98	25	8.9	3.6
set 3	nd	94*	24*	8.9*	3.1*
set 3	2	81	23	9	2.1
set 3	3	95	23	8.8	3.5
set 3	nd	42*	15*	8.9*	3.1*
set 3	nd	42*	15*	8.9*	3.1*
set 3	nd	94*	24*	8.9*	3.1*

*Assumption for missing values based on average per set

nd: no data available

Appendix table 14: Results of rectification pilot scales tests

	$\dot{G}_1$ [l/h]	$\dot{L}_0/\dot{G}_1$ [-]	$\dot{L}_N$ [l/h]	X_N [g/l]	pH $_{XN}$ [-]	Y_1 [g/l]	pH $_{Y1}$ [-]	Y_t [g/l]	Y_1/Y_t [-]	$G_0 - G_1$	df	$(G_0 - G_1)$ $/G_0$ [%]
set 1	6	14	109	0.38	5.9	65.4	9.4	48	1.37	27.0	0.75	82
set 1	13	5.7	80	0.17	5.6	23.1	9.3	30	0.78	7.5	0.91	37
set 1	11	7.5	105	0.114	5.8	52	9	38	1.36	23.0	0.78	68
set 1	7	8.7	75	0.16	5.6	74.3	9.3	57	1.31	14.0	0.81	67
set 2	16	1.5	33	>0.001	7.9	8.3	9.3	11	0.76	8.6	0.74	36
set 2	11	2.5	35	>0.001	10.4	15.8	9.3	16	0.98	8.2	0.77	43
set 2	10	2.7	35	>0.001	10.4	16.2	9.9	17	0.95	7.1	0.80	41
set 2	8.6	3.4	36	>0.001	8.95	21.4	7.35	24	0.90	7.3	0.80	46
set 3	3.5	28	120	0.18	5.5	116	9.6	86	1.34	21.5	0.82	86
set 3	2.7*	38	nd	0.1	nd	89.6	nd	92	0.97	21.3	-	89
set 3	2.2	37	102	0.08	5.4	108	9.6	69	1.57	20.8	0.80	90
set 3	2.4	40	116	0.33	5.5	121	9.5	111	1.09	20.6	0.82	90
set 3	2.7*	15.4	nd	1.03	nd	32.5	nd	27	1.22	1.3	-	82
set 3	2.7*	15.4	nd	0.35	nd	43.9	nd	37	1.19	12.3	-	82
set 3	2.7*	35	nd	>0.001	nd	103	nd	96	1.07	21.3	-	89

*Assumption for missing values based on average per set

nd: no data available

Appendix table 15: Phosphorous content during pilot scale rectification

	P_{feed} X_0 [mg/l]	P_{Ndepl} X_N [mg/l]	P_{cond} Y_1 [mg/l]	C_d / C_0	P^{exp}_{Ndepl} (2 X_t [mg/l]	P^{norm}_{Ndepl} (3 X_N [-]
set 1	431	376	>0.1	87 %	390	0.96
set 2	395	300	>0.1	76 %	315	0.95
set 2	440	326	>0.1	74 %	324	1.01
set 2	431	312	>0.1	72 %	343	0.91

2) The expected concentration of phosphorous in the N-depleted substrate is calculated by $P^{exp}_{Ndepl} = P_{feed} * df$

3) The normed concentration of phosphorous in the N-depleted substrate is calculated by $P^{norm}_{Ndepl} = P_{Ndepl} / df$

Appendix table 16: COD, potassium, sulphur, and phosphorous content during pilot scale rectification

		COD mg O_2/l	Red.(1	K mg/l	Red.(1	K^{norm}	S mg/l	Red.(1	S^{norm}	P mg/l	Red.(1	P^{norm}
Set 3	X_0	2 590		1 198			354			207		
	Y_1	370	14 %	<1	-		58.3	16 %		<2	<1	
	Y_1	1 080	42 %	1.1	0.1 %		83.3	24 %		<2	<1	
	X_N	1 790	69 %	930	78 %	1.06	298	84 %	0.97	162	78 %	1.05
	X_N	1 428	55 %							178	85 %	0.95

1) Reduction regarding initial concentration of feed substrate

Appendix 3.2: ad Evaporation

Appendix table 17: Total solids (TS) and derived concentration factor (feed acidified with H_2SO_4)

Concentrates (* & feed	TS [g/l]	TS *cf*
X_1 (1250)	69	*2.3*
X_1 (1750)	104	*3.5*
X_1 (3975)	224	*7.5*
X_1 (7750)	379	*12.7*
X_0	30	

Distillate (*	TS [g/l]	TS *cf*
Y_1 (1250)	-0.1	*0.0*
Y_1 (1750)	-0.1	*0.0*
Y_1 (3975)	0.6	*0.0*
Y_1 (7750)	0.1	*0.0*

**) evaporation time in brackets*

Appendix table 18: Nutrients during concentration process (feed acidified with H_2SO_4)

	X_0	X_1 (1250)	X_1 (1750)	X_1 (3975)	Y_1 (1250)	Y_1 (1750)	Y_1 (3975)
TN mg/l	4 330	10 100	16 400	27 500	130	40	320
cf_{det}		*2.3*	*3.8*	*6.6*	*3 %*	*1 %*	*7 %*
P mg/l	408	988	1 472	3 037	<2	<2	<2
cf_{det}		*2.4*	*3.6*	*7.4*	*< 0.5 %*	*< 0.5 %*	*< 0.5 %*
K mg/l	1 360	3 290	1 125	2 420	1.57	0.57	1.5
cf_{det}		*2.4*	*0.8*	*1.8*	*0.1 %*	*0.04 %*	*0.1 %*

**) evaporation time in minutes in brackets*

Appendix table 19: Metals during concentration process (feed acidified with H_2SO_4)

	X_0	$X_{N\ (1250)}$	$X_{N\ (1750)}$	$X_{N\ (3975)}$	$Y_{1\ (1250)}$	$Y_{1\ (1750)}$	$Y_{1\ (3975)}$
Na mg/l	2 085	4 945	1 780	3 890	2.13	0.96	3.05
Ca mg/l	7.13	18.3	29.8	67.9	1.56	2.66	0.97
Mg mg/l	0.19	0.84	0.86	1.98	<0.1	<0.1	<0.1
Fe mg/l	1.73	2.46	10.1	11.2	<0.1	4.29	<0.1
Cu mg/l	25.4	65.3	2.38	7.83	0.99	<0.05	0.07
Zn mg/l	3.88	12.0	2.17	3.97	2.76	<0.05	<0.05
Cd μg/l	<1	<1	na(*	<1	na	na	na
Ni μg/l	166	518	na	226	na	na	na
Pb μg/l	131	412	na	98	na	na	na
Gd μg/l	15	42	na	128	na	na	na
Cr μg/l	39	188	na	111	na	na	na

*) na: not analyzed

Appendix table 20: Organic acids during concentration process (feed acidified with H_2SO_4)

		X_0	$X_{N\ (1250)}$	$X_{N\ (1750)}$	$X_{N\ (3975)}$	$Y_{1\ (1250)}$	$Y_{1\ (1750)}$	$Y_{1\ (3975)}$
acetic acid	mg/l	1 660	3 170	6 090	7 770	210	1 160	500
propionic acid	mg/l	105	170	320	780	20	140	80
iso- butyric acid	mg/l	30	8	30	80	10	50	40
n- butyric acid	mg/l	<4	30	70	170	<4	120	90
iso- valeric acid.	mg/l	40	40	40	80	20	80	60
n- valeric acid	mg/l	<2	<2	<2	<2	<2	<2	<2
caproic acid	mg/l	<2	<2	<2	<2	<2	<2	<2

Appendix table 21: Concentrates and distillates from evaporation (feed acidified with H_3PO_4)

	evaporation time [h]	**pH**	**NH_4-N**		**P**		**TS**	
			[mg/l]	*cf_N*	[mg/l]	*cf_P*	[g/l]	*cf_{TS}*
feed X_0		7.3	3 640		1150		8.8	
Concentrate X1	24	5.54	27 000	*7*	33300	*29*	212	*24*
	27	5.38	24 000	*7*	31500	*27*	238	*27*
	53	5.51	32 000	*9*	59400	*52*	465	*53*
	57	5.43	28 700	*8*	58500	*51*	499	*57*
Distillate Y_1	24	5.41	420	*12 %*	5.9	*0.51 %*	0.2	*2.5 %*
	27	9.05	279	*8 %*	0.36	*0.03 %*	0.4	*5.0 %*
	53	8.99	1500	*41 %*	0.22	*0.02 %*	0.4	*4.7 %*
	57	9.24	4000	*110 %*	0.14	*0.01 %*	0.2	*1.8 %*

Appendix table 22: pH, ammonium, and total solids (TS) of concentrates and distillates from N-depleted urine after rectification and evaporation

	evaporation time [h]	pH	TS		NH_4-N		P		K		S	
			g/l	*cf_{TS}*	mg/l	*cf_N*	mg/l	*cf_P*	mg/l	*cf_K*	mg/l	*cf_S*
feed average X_0		6.77			120		170		930		298	
Conc $_{Ndep}$	24	6.03	142	*24.4*	2128	*17.3*	na	*nc*	na	*nc*	na	*nc*
	32	5.74	188	*32.4*	1400	*11.7*	6730	*40*	37100	*40*	11700	*39*
	55	5.82	319	*55.0*	2560	*21.3*	8900	*52*	48100	*52*	15700	*53*
Dist $_{Ndepl}$	24	6.77	0.51		na		na		na		na	
	32	9.44	na		na		<2		1.48		7.3	

Appendix 3.3: ad Removal of pharmaceutical residues

Acidification

Urine from B '06 was acidified with 17 ml HPO_3/l substrate (pH-reduction from pH 9 to pH 4). Already the acidification had some effect on most pharmaceutical residues.

Appendix table 23: Influence of acidification on pharmaceutical residues in urine

	pH 8.9 X_0 [µg/l]	pH4 X_d [µg/l]	X_N / X_0
Bezafibrate	495	413	*0.83*
Ibuprofen	442	354	*0.80*
β-Sitosterol	40	23	*0.56*
Diclofenac	13.9	9.2	*0.66*
Pentoxifylline	5.6	3.9	*0.70*
Carbamazepine	3.5	7.9	*2.26*
Phenazone	2.1	< 1.0	*0*
Phenacetin	1.4	1.4	*1.0*

Appendix table 24: Influence of acidification on pharmaceutical residues in N-depleted urine

	pH 7 X_0 [µg/l]	pH4 X_d [µg/l]	X_N / X_0
Bezafibrate	649	636	*0.98*
Ibuprofen	590	480	*0.81*
β-Sitosterol	87	8.4	*0.10*
Diclofenac	22	16	*0.71*
Pentoxifylline	6.3	7.6	*1.21*
Carbamazepine	14	13	*0.94*
Phenazone	1.8	2.1	*1.17*
Phenacetin	4.5	1.8	*0.40*

The N-depleted substrate was acidified with 2.5 ml HPO_3/l N-depl substrate (for pH reduction from pH7 to pH4) (Appendix table 23). ß-sitosterol was affected the most by acidification. Reductions of more than 40 % and in the N-depleted substrate of more than 90 % were observed. The detected concentration of pharmaceutical residues in ozonated urine and acidified urine is presented in Appendix table 25.

Appendix table 25: Influence of ozone on pharmaceutical residues in acidified urine

	stored urine pH 9					acidified stored urine pH 4				
	feed X_0 [µg/l]	0.97 gO_3/l		1.59 gO_3/l		feed X_0 [µg/l]	0.5 gO_3/l		0.86 gO_3/l	
		X_N [µg/l]	*X_N/X_0 [%]*	X_N [µg/l]	*X_N/X_0 [%]*		X_N [µg/l]	*X_N/X_0 [%]*	X_N [µg/l]	*X_N/X_0 [%]*
Bezafibrate	495	159	*32*	47	*10*	413	249	*60*	97	*23*
Ibuprofen	442	163	*37*	57	*13*	354	266	*75*	209	*59*
β-Sitosterol	40	40	*99*	-	*nc*	23	-	*nc*	-	*nc*
Diclofenac	14	-	*nc*	-	*nc*	9.2	-	*nc*	-	*nc*
Pentoxifylline	5.6	1.7	*30*	-	*nc*	3.9	4.1	*105*	1.9	*49*
Carbamazepine	3.5	-	*nc*	-	*nc*	7.9	3.8	*48*	3.1	*39*
Phenazone	2.1	-	*nc*	-	*nc*		-	*nc*	-	*nc*
Phenacetin	1.4	-	*nc*	-	*nc*	1.4	-	*nc*	-	*nc*

Reduction rates at the more or less same O_3-consumptions were except for ß-sitosterol in the same range. In case of carbamazepine the initial concentration were so low that comparison seems to be not significant. ß-sitosterol seemed to be degraded mainly by direct O_3-reaction since as well as during the ozonation of N-depleted urine as well as during ozonation of acidified urine removal was significantly higher than in the untreated stored urine.

Ozonation of N-depleted substrate at low pH

Also N-depleted urine was acidified from initially pH 7 to pH 4. The N-depleted urine and the acidified N-depleted urine were treated with ozone. Again ozone uptake was reduced by nearly half the amount by acidification. The results are presented in Appendix table 26.

Appendix table 26: Influence of ozone on pharmaceutical residues in acidified N-depleted urine

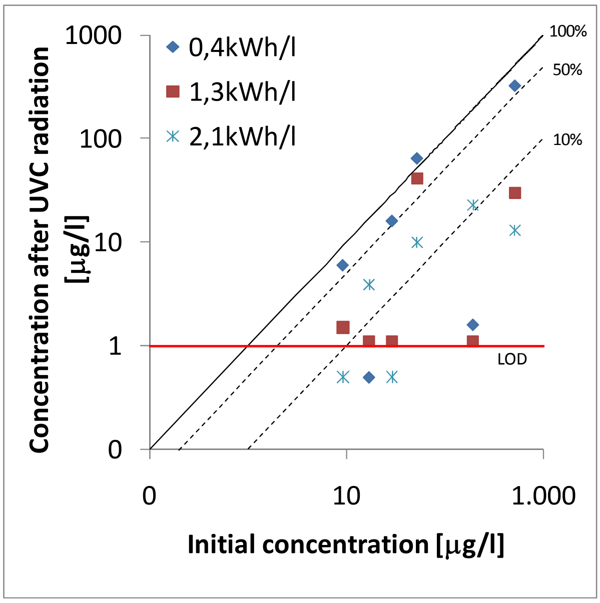
	pH 7	1.37 gO_3/l		1.94 gO_3/l		pH 4	0.63 gO_3/l		1.26 gO_3/l	
	X_0 [µg/l]	X_N [µg/l]	*X_N/X_0 [%]*	X_N [µg/l]	*X_N/X_0 [%]*	X_0 [µg/l]	X_N [µg/l]	*X_N/X_0 [%]*	X_N [µg/l]	*X_N/X_0 [%]*
Bezafibrate	649	335	*52*	167	*26*	636	362	*57*	147	*23*
Ibuprofen	590	390	*66*	274	*46*	480	326	*68*	265	*55*
β-Sitosterol	87	28	*32*		*0*	8.4		*0*		*0*
Diclofenac	22		*0*		*0*	16		*0*		*0*
Pentoxifylline	6.3	6.6	*105*	3.5	*56*	7.6	7.1	*93*	3	*39*
Carbamazepine	14	6.6	*46*	3.6	*25*	13	7.7	*57*	5.1	*38*
Phenazone	1.8		*0*		*0*	2.1		*0*		*0*
Phenacetin	4.5		*0*		*0*	1.8	1.6	*89*		*0*

Appendix table 27: Removal efficiency per g O_3 for acidified and non-acidified urine and N-depleted urine

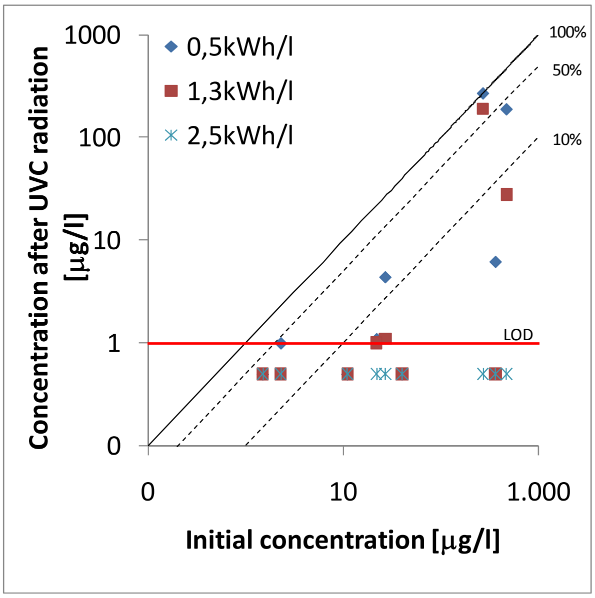
type of substrate	Stored urine				N-depleted urine			
	'regular' pH 8.9		acidified to pH 4		'regular' pH 7		acidified to pH 4	
applied ozone dose [g O_3/l]	1.6	1.0	0.9	0.5	1.4	1.9	0.6	1.3
	removal/ g O_3/l	removal/ g O_3/l	removal/ g O_3/l	removal/ g O_3/l	removal/ g O_3/l	removal/ g O_3/l	removal/ g O_3/l	removal/ g O_3/l
Bezafibrate	57 %	68 %	85 %	79 %	35 %	39 %	72 %	59 %
Ibuprofen	55 %	63 %	46 %	50 %	24 %	28 %	53 %	34 %
β-Sitosterol	nc	1 %	nc	nc	48 %	nc	nc	nc
Diclofenac	nc	nc	nc	nc	nc	nc	nc	nc
Pentoxifylline	nc	70 %	57 %	-10 %	-3 %	23 %	11 %	47 %
Carbamazepine	nc	nc	68 %	104 %	38 %	39 %	71 %	48 %
Phenazone	nc	nc	nc	nc	nc	nc	nc	nc
Phenacetin	nc	nc	nc	nc	nc	nc	19 %	nc

UVC-irradiation for removal of pharmaceutical residues

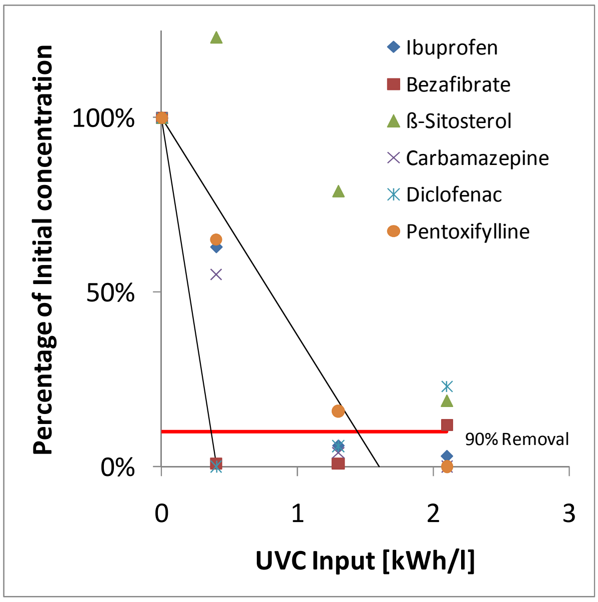
Urine and N-depleted urine were treated by UVC-irradiation as described in *Tettenborn et al.* (*2007*). The results are presented in Appendix figure 2 and Appendix figure 4 for urine, and Appendix figure 3 and Appendix figure 5 for the N-depleted substrate. It has to be noted that the energy input between the two sets was altered. Introduced energy dosages were high.



Appendix figure 2: Concentrations of pharmaceutical residues in urine after UVC-irradiation.

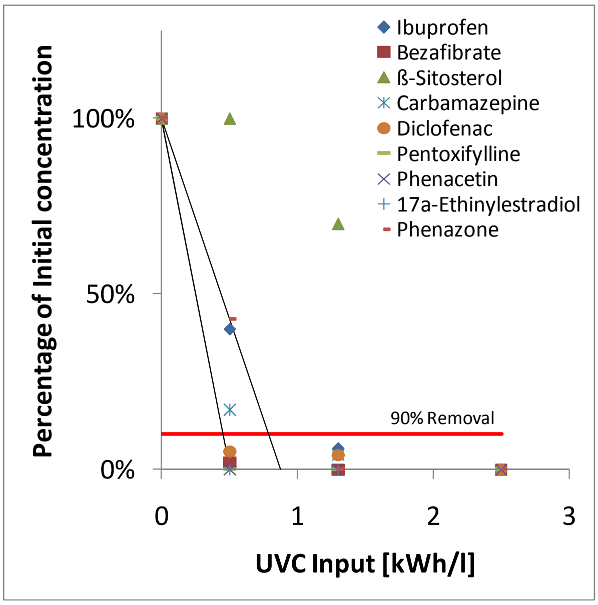


Appendix figure 3: Concentrations of pharmaceutical residues in N-depleted substrate after UVC-irradiation.



Appendix figure 4: Concentrations of pharmaceutical residues in urine during UVC-irradiation.

Red line indicates 90 % removal



Appendix figure 5: Concentrations of pharmaceutical residues in N-depleted urine during UVC-irradiation.

Red line indicates 90 % removal.

In Appendix figure 2 and Appendix figure 3 the concentration after UVC-irradiation is plotted over the initial concentration. The horizontal line is indicating the LOD of most pharmaceutical residues in the urine matrix. Values below LOD are displayed as 0.5*LOD. The 100 % line indicates substances that are not affected by UVC-irradiation. The dotted lines represent 50 % and 10 % of the initial concentration. It can be readily observed that concentrations in nitrogen depleted substrate after rectification and after UVC-irradiation (Appendix figure 3) are lower compared to concentrations in urine after UVC-irradiation (Appendix figure 2).

In Appendix figure 4 and Appendix figure 5 the percentage of the initial concentration of the individual pharmaceutical residues are plotted over the energy input for UVC-irradiation. In this case the horizontal line is indicating a 90 % removal. Assuming a linear reduction of pharmaceutical residues by UVC-irradiation within the first 90 % of removal, the energy demand can consequently be estimated for an average removal rate. To cope with values above the initial concentrations or far away from the average concentrations such as ß-sitosterol in Appendix figure 4 these values were excluded in the estimation of an average removal rate. Based on this agreement the removal rate would still be valid for about 90 % of the detected pharmaceutical residues. In Appendix table 28 the energy demand for a 90 % removal efficiency of 90 % of the detected pharmaceutical residues is presented.

For a 90 % removal rate of 90 % of the detected pharmaceutical residues in urine a UVC energy demand of 1.4 kWh/l (1400 kWh/m^3) was required. For N-depleted urine after rectification / steam stripping the UVC energy demand for a 90 % removal of 90 % of the detected pharmaceutical residues was 0.8 kWh/l (800 kWh/m^3). Although energy requirements of the UVC-irradiation were extremely high an effect could clearly be stated.

Many pharmaceutical residues are known to be instable under photolytic conditions. Several authors reported about e.g. carbamazepine degradation rates of 25–42 % during sunlight irradiation (*Andreozzi et al. 2002*; *Doll & Frimmel 2003*). Of diclofenac *Buser et al.* (*1998*) reported degradation rates of more than 90 % by natural photolytic processes. Both correlate with the observed results in this study. ß-sitosterol as a naturally occurring phytosterol was the one being most persistent against UVC-light which is noteworthy but reasonable.

Appendix table 28: Energy demand for 90 % removal during UVC-irradiation

all values in kWh/l	**stored urine**	**N-depl. urine**
Ibuprofen	0.97	0.75
Bezafibrate	0.36	0.46
β-Sitosterol	(2.33)	(2.25)
Phenacetin (*	- (*	0.45
Carbamazepine	0.80	0.54
Diclofenac	0.36	0.47
17α-Ethinylestradiol (*	- (*	0.45
Phenazone	- (*	0.79
Pentoxifylline	1.39	0.45

*) Phenacetin, 17α-ethilylestradiol, and phenazone were not detected in the initial substrate of stored urine

Estimation of wastewater load

Wastewater processed in German municipal treatment plants in 2001, including domestic, industrial, and rain water (Brenk et al. 2006) divided by connected German population: $(10.5*10^9\ m^3/a)/(80*10^6)/(365d/a)=360\ l/p/d$

Appendix 4. ad Implementation potential

Appendix table 29: Base parameters of scenarios 1 to 4

Scenario	**1**	**2**	**3**		**4**		
coll. from	wlu pub	wlu pub	wlu pub	wlu office	wlu pub	wlu office	udt office*
Volume based coverage equivalents [capita]							
	730	*2200*	“	8000	“		12000
Collected volume [m^3/d]							
	1	3	“	11	“		8
Loads [kg/d]							
C	2.3	6.8	see scenario 2	54.8	see scenario 3		32.9
N	3.7	11.3		91			54.6
P	0.3	0.8		8.8			5.3
MP	0.5	1.4		11			6.6

* separation ratio: 40 %; dilution grade: 20 %

Appendix table 30: Base parameters of scenarios 5a and 5b

Scenario	**5 a (cov. equ. 25 %)**					**5 b (cov. equ. 80 %)**				
coll. from	wlu pub	wlu office	udt office	wlu private	udt private	wlu pub	wlu office	udt office	wlu private	udt private
Volume based coverage equivalents [capita]										
x 1000	“	89	134	89	134	“	285	427	285	427
Collected volume [m^3/d]										
	“	122	103	122	112	“	390	329	390	358
Loads [kg/d]										
C	see scenario 2	610	412	610	503	see scenario 2	1951	1317	1951	1609
N		1012	683	1012	835		3238	2186	3238	2672
P		98	66	98	80		312	211	312	258
MP		122	82	122	101		390	263	390	322

Appendix table 31: Base parameters of scenarios 5c and 5d

Scenario	**5 c (cov. equ. 25 %) (optimized separation technique)**					**5 d (cov. equ. 80 %) (optimized separation technique)**				
coll. from	wlu pub	wlu office	o-udt office	wlu private	o-udt private	wlu pub	wlu office	p-udt office	wlu private	o-udt private
Volume based coverage equivalents [capita]										
x 1000	“	“	134	“	134	“	“	427	“	427
Collected volume [m^3/d]										
	“	“	142	“	173	“	“	455	“	554
Loads [kg/d]										
C	see scenario 2	see scenario 5a	640	see scenario 5a	823	see Scenario 2	see scenario 5b	2048	see scenario 5b	2634
N			1063		1366			3400		4372
P			102		132			328		421
MP			128		165			410		527

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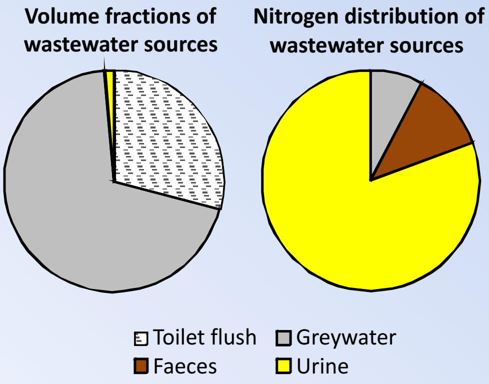
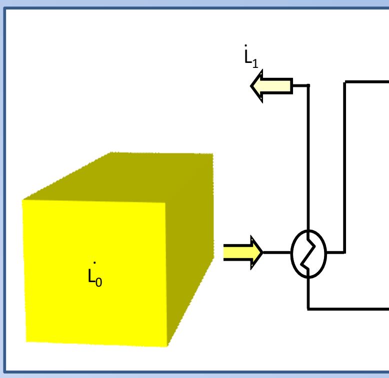
Collectable amounts and concentration of public urinals were measured, to evaluate the potential of a system for separate urine collection and treatment in an urban context. The collected substrate was processed amongst others by rectification and evaporation for nutrient recovery and reduction of micropollutants as pharmaceutical residues. After proving the technical feasibility of the treatment in pilot scale, transport and achievable product values were discussed in respect to scale.





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