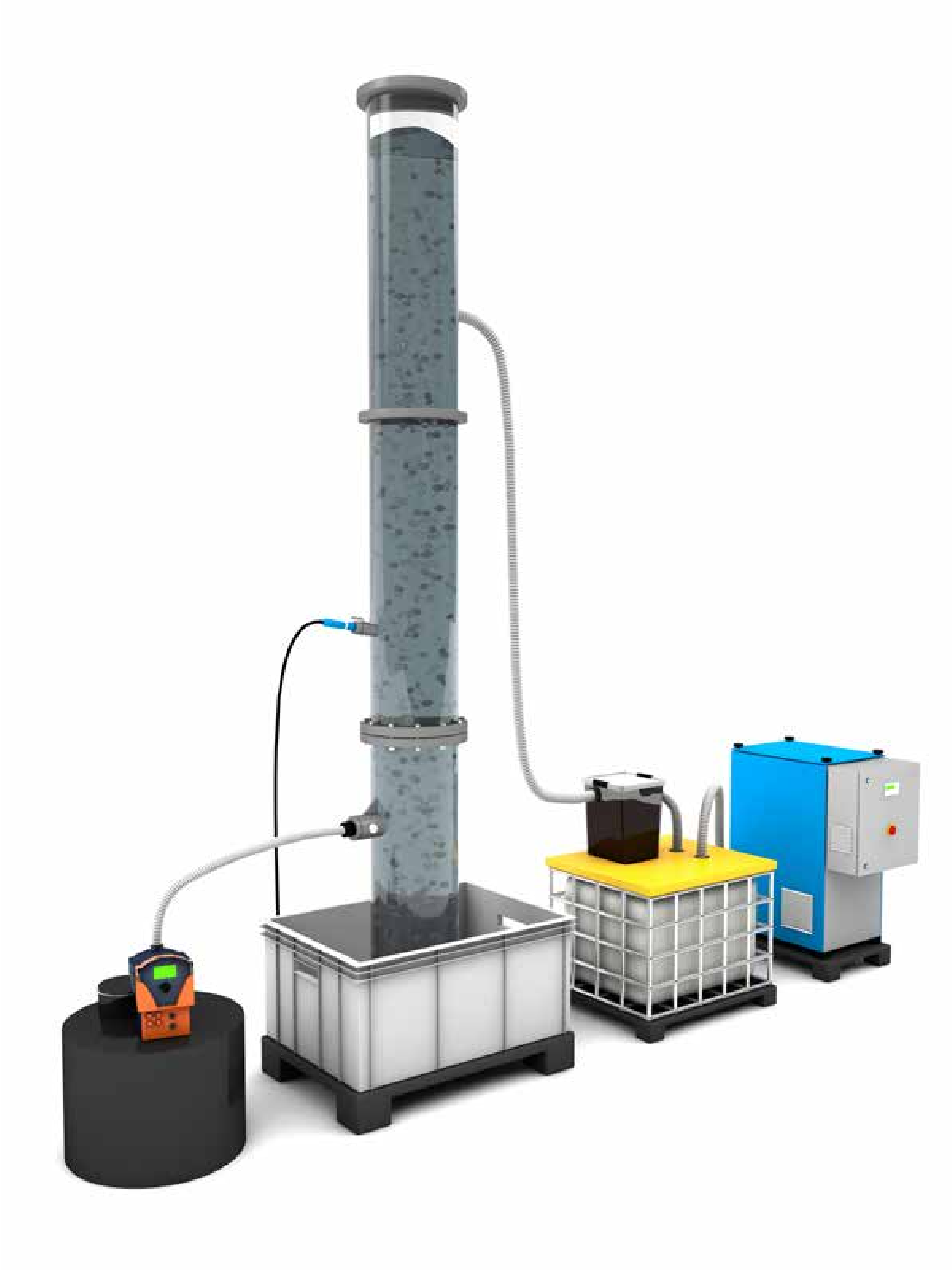
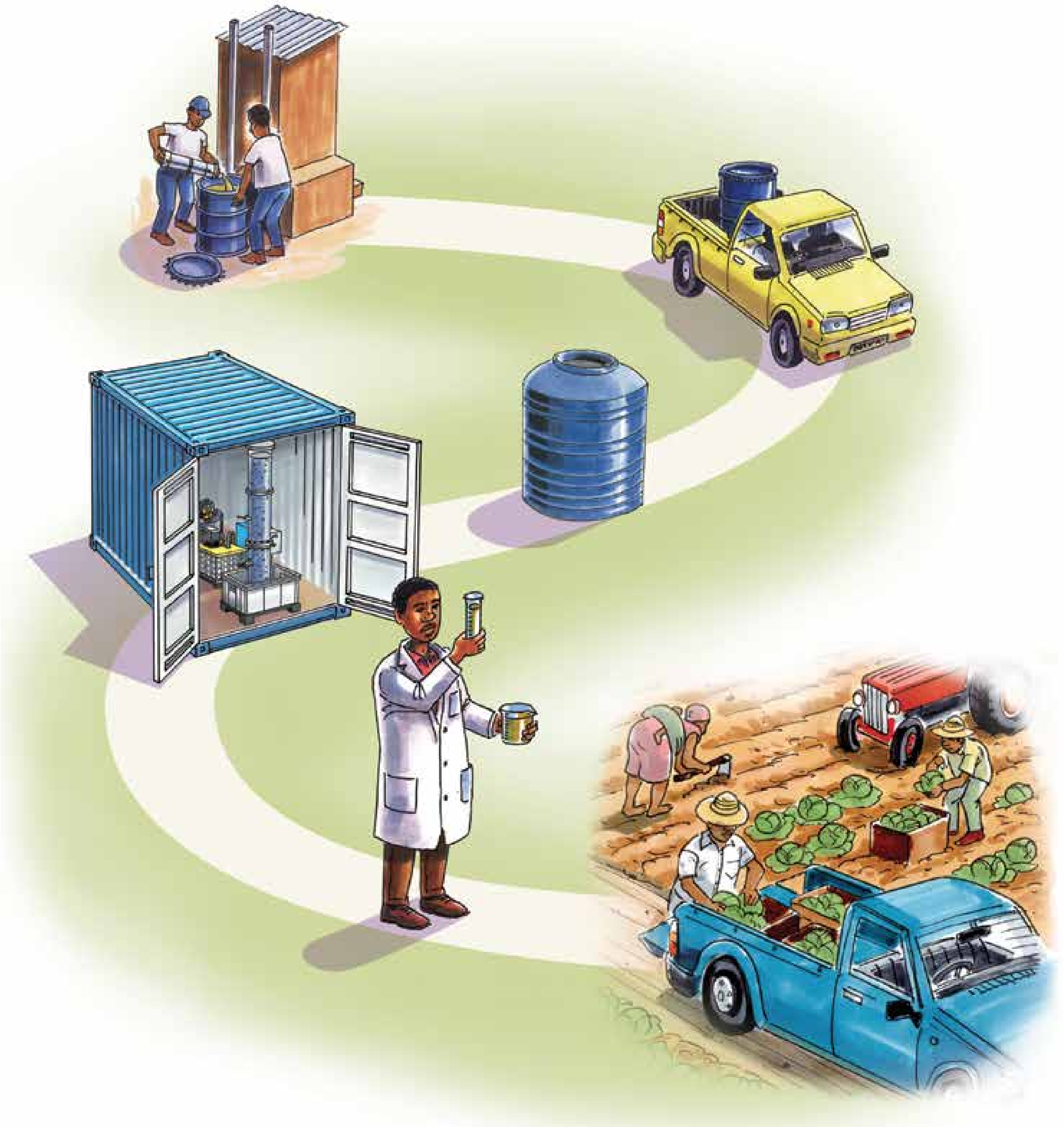
VUNA Handbook on

Urine Treatment





Version: March 2016

Editors: Bastian Etter, Kai M. Udert.

Images: All pictures and illustrations by VUNA Project Team, unless otherwise indicated.

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The VUNA Project was carried out by:

VUNA – Urine Treatment Processes
Table of Contents

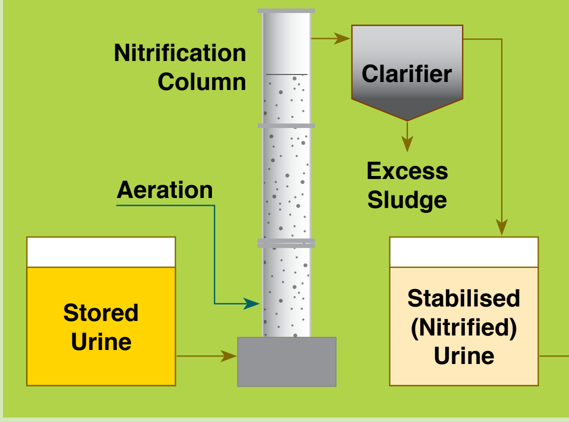
Complete Nutrient Recovery (p. 6)

Recovering all nutrients contained in urine. This process developed as a pilot in the VUNA Project consists of two individual steps:

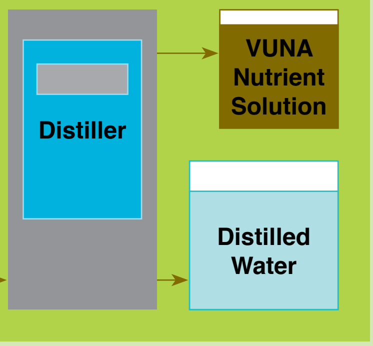
1. Nitrification stabilises the nitrogen in the urine, prevents nutrient losses and malodour.

2. Distillation separates the water and produces a concentrated nutrient solution.

Nitrification (p. 14)



Distillation (p. 14)

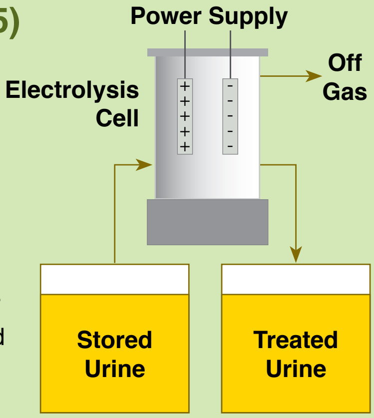


Electrolysis (p. 15)

Electrolysis removes ammonia and organic substances from urine using electrical current.

Laboratory experiments proved that electrolysis is well suited for urine treatment in compact units.

Struvite can be precipitated using electrochemical magnesium dosage (right).

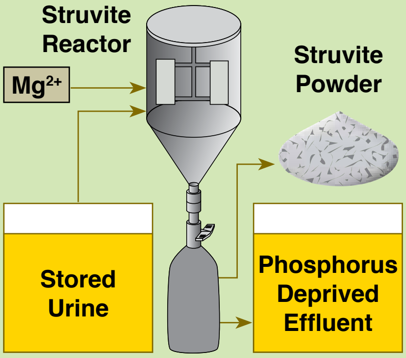


Struvite Precipitation (p. 16)

Struvite is a dry phosphorus fertiliser.

It is extracted from urine after adding magnesium and subsequently filtering out the solids.

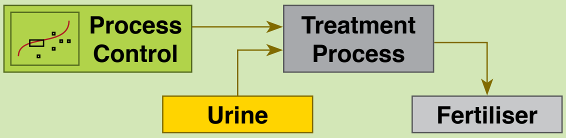
VUNA developed two struvite plants: a manual and a fully automated one.



Process Control (p. 18)

For a stable process operation, an intelligent process control is essential.

VUNA studied new methods to monitor nitrite in urine treatment processes, because the intermediate nitrite compromises process stability.



Other Processes (p. 19)

Beyond VUNA, numerous processes have been studied to recover nutrients from urine. This section presents certain processes including their effectiveness and feasibility.

Stripping

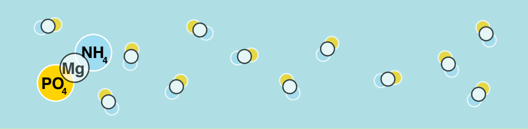
Adsorption

Freeze-Drying

$Ca(OH)_2$ addition

Urine Analysis (p. 20)

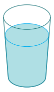
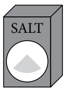
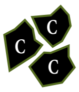
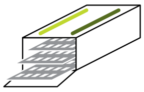
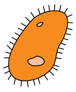
What is urine? Endorse your research with reliable measurements. Refer to this section to effectively sample, measure, or even synthesise urine.



0 Overview

Urine is a complex mixture of numerous substances. All together or individually they can have severe negative impacts on humans and the environment. But, if the right substance is applied at the correct location at an optimum dose, it holds the potential for beneficial reuse. Table 1 attributes beneficial reuses and potential negative impacts to each urine ingredient. The objective of urine treatment is to maximise beneficial reuse while minimising potential negative impacts.

Table 1: *Compounds in stored urine, their reuse potentials and possible negative effects. Adapted from Udert et al., 2015.*

Compound		***Beneficial reuses***	***Potential negative impacts***
	**Water** **(H_2O)**	Recycling, e.g. irrigation	Water volume requires large storage tanks. Water weight makes transport expensive.
N	**Nitrogen** **(present as NH_4^+)**	Fertiliser, Raw product for chemical industry	Smell and toxicity of gaseous ammonia Environmental pollution: nitrate in ground-water, eutrophication of receiving waters and soils, fish toxicity, atmospheric particles.
P	**Phosphorus** **(present as PO_4^{3-})**	Fertiliser Raw product for chemical industry	Environmental pollution (eutrophication) Precipitates block pipes and valves (with calcium and magnesium).
K	**Potassium** **(K^+)**	Fertiliser Raw product for chemical industry	Salinisation of agricultural soils and groundwater
Ca	**Calcium** **(Ca^{2+})**	Fertiliser Raw product for chemical industry	Precipitates block pipes and valves (with phosphates).
Mg	**Magnesium** **(Mg^{2+})**	Fertiliser Raw product for chemical industry	Precipitates block pipes and valves (with phosphates).
S	**Sulphur** **(S)**	Fertiliser Raw product for chemical industry	Environmental pollution (in excessively high concentrations only) Smell and toxicity of hydrogen sulfide
B Fe Zn Mo Mn Cu	**Micro-nutrients**	Fertiliser	Environmental pollution (in excessively high concentrations only)
HCO_3^-	**Bicarbonate** **(HCO_3^-)**	None	Precipitates block pipes and valves
	**Sodium chloride** **(NaCl)**	Raw product for chemical industry	Salinisation of agricultural soils and groundwater
	**Bulk organic substances**	None	Pungent smell. Treatment problems, e.g. foaming Organic reduction of sulfate and nitrate: sulfide production or NO and N_2O production (pollutants, climate gases)
	**Pharmaceuticals** **(trace organic compounds)**	None	Human health concerns Environmental concerns
	**Pathogens** **(bacteria, viruses, helminths)**	None	Human health concerns

In VUNA, three urine treatment processes were tested. Table 2 compares these processes. In summary, the processes are:

- The first process is **Struvite Precipitation (p. 16)**, a technology which had already been tested in multiple projects on urine treatment. Struvite precipitation is a simple and fast process for phosphorus recovery. Other nutrients, such as nitrogen and potassium, remain in the effluent and pathogens are not completely inactivated. Therefore, struvite precipitation has to be combined with other treatment processes to prevent environmental pollution and hygiene risks.

- The second process is **Complete Nutrient Recovery (p. 6)**, a combination of nitrification and distillation. This process combination is more complex than struvite precipitation, but it recovers all nutrients in one concentrated solution, ensures safe sanitisation and produces only distilled water and a small amount of sludge as by-products.

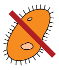
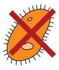
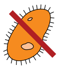
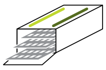
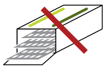
- The third process is **Electrolysis (p. 15)**. This process could be used for very small on-site reactors, because conversion rates are high and the operation is simple, as long as appropriate electrodes and voltages are used. However, nitrogen is removed and not recovered and chlorinated by-products are formed, which can be hazardous for human health.

While urine electrolysis requires further research in the laboratory, struvite precipitation and nitrification/distillation have already been operated at pilot scale. For more details refer to the recommended publication.

Recommended Publications (free download):

- *Udert, K.M., Buckley, C.A., Wächter, M., McArdell, C.S., Kohn, T., Strande, L., Zöllig, H., Fumasoli, A., Oberson, A., Etter, B. (2015)* ***Technologies for the treatment of source-separated urine in the eThekwini municipality.*** *Water SA, 41(2), 212-221.*

Table 2: *Comparison of struvite precipitation, nitrification/distillation and electrolysis according to the research results in VUNA. Adapted from Udert et al., 2015 (see 'Publications').*

Characteristics	***Struvite precipitation***	***Nitrification/Distillation***	***Electrolysis***
Main product	Struvite (phosphate mineral) $MgNH_4PO_4 \cdot 6H_2O$	Concentrated nutrient solution	Liquid without ammonia, organic substances and pathogens
Primary nutrient recovery	P N Medium, mainly phosphate	N P K + all other nutrients High, nearly all nutrients	None, if not combined with other processes
Primary nutrient loss	N K + other nutrients High, most nutrients in the effluent	Low, little ammonia volatilization	N P K + other nutrients High, ammonia is oxidised to N_2
Sanitisation/ disinfection	 Medium, depends on drying	 Complete (during distillation)	 Medium, due to chlorination
Malodour removal	No	Yes	Yes
Pharmaceuticals (trace organic compounds)	 No degradation	 Partial degradation (nitrification)	 Unknown degradation
Energy demand*	🟩 Very low (manual reactor) 🟩 Low (automated reactor)	🟨 Medium ~ 50 $Wh \cdot L_{urine}^{-1}$ nitrification ~ 100 $Wh \cdot L_{urine}^{-1}$ distillation	🟥 High ~ 320 $Wh \cdot L_{urine}^{-1}$ (TDIROF electrode) ~ 1600 $Wh \cdot L_{urine}^{-1}$ (BDD electrode)
Volume reduction	🟦 High Urine to struvite: concentration factor 250 – 630**	🟦 High Liquid volume: concentration factor 20 – 30	No volume reduction
Other outputs	Phosphate depleted urine	Distilled water, excess sludge	Off-gas with chlorinated by-products
Process complexity	🟩 Low (manual reactor) 🟨 Medium (automated reactor)	🟥 High	🟩 Low
Development level	🟦 High	🟦 High	🟦 Low

* Calculated for a total ammonia concentration of 8 g $N \cdot L^{-1}$. The values for electrolysis are based on the experimental values given in the text. These values are most probably too high, because they are based on small-scale laboratory experiments, which have not been optimised for low energy demand.

** Assuming phosphate concentrations between 200 and 500 mg $P \cdot L^{-1}$ in stored urine.

1 Complete Nutrient Recovery

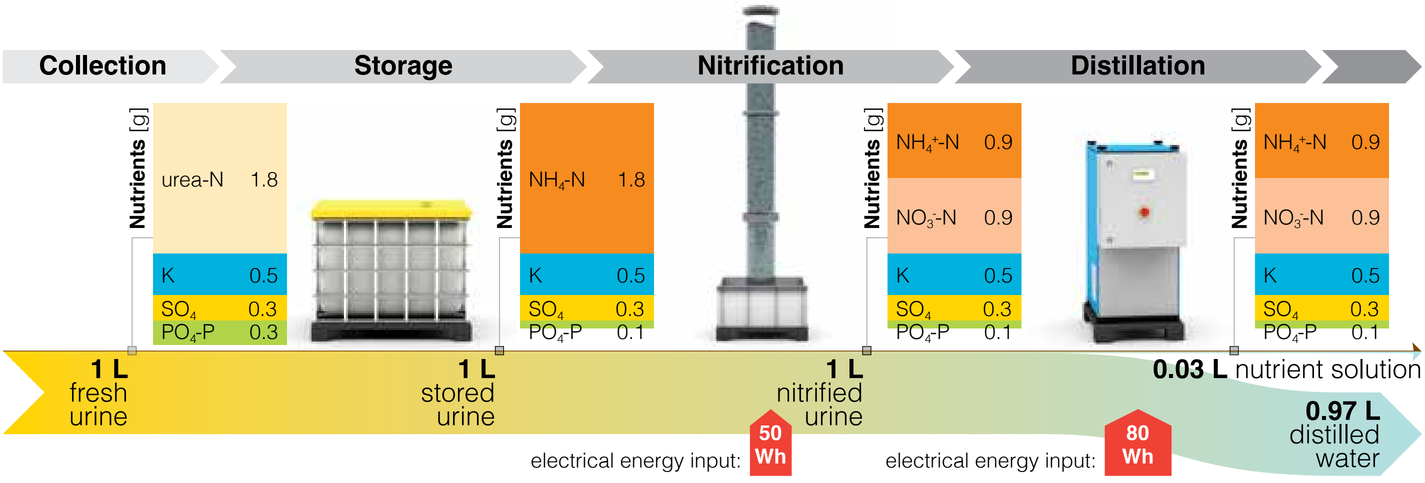


Figure 1: *Mass flow through the combined nitrification/distillation process. All nutrients from urine are recovered as a liquid fertiliser.*

1.1 Concept & Functioning:

Two Process Steps for Complete Recovery

The complete nitrification process developed by the VUNA Project consists of two main process steps (Figure 1):

1. **Nitrification:** Approximately 50 % of the total ammonia (NH_3 and NH_4^+) contained in stored urine is transformed to nitrate (NO_3^-) by bacteria. Simultaneously, about 90 % of the organic matter is degraded. The remaining nitrogen leaves the reactor unchanged as ammonium. The resulting liquid is free from malodour and can be distilled without losing ammonia via volatilisation, i.e. the urine and the nutrients contained in it are stabilised by the nitrification process.

2. **Distillation:** The stabilised urine is distilled to approximately 3 to 5 % of its initial volume. The remaining concentrated liquid contains all the nutrients from urine. At ambient temperature, all nutrient salts stay in solution. Distilled water is recovered and can be reused, e.g. to flush the toilets.

Moving Bed Biofilm Reactor – Principles

A moving bed biofilm reactor (MBBR) holds a large number of small plastic biomass carriers (Figure 2) to provide a large surface for bacterial growth. By providing this surface, slow-growing bacteria, e.g. nitrifying bacteria, can be retained in the reactor. The selection occurs as bacteria growing on carriers benefit from a longer retention time, whereas bacteria growing in suspension are washed out faster. MBBR are typically filled to about 50 % of their bulk volume with biomass carriers. Lower filling rates proportionally reduce the surface available for bacterial growth, thus result in lower conversion rates. Higher filling rates (above 60 %) impede proper mixing of the reactor contents and consequently suffer from a limited oxygen availability in some parts of the reactor vessel.

Biomass Carriers

Biomass carriers can have different shapes and sizes. In general, they are designed to supply a maximum surface area while still allowing oxygen to diffuse to lower biofilm layers. They are mostly manufactured from high-density poly-ethylene (HDPE) using extrusion. Their density is adjusted to be slightly inferior to the media they will have to treat, such that they float beneath the surface in absence of any agitation. A thorough aeration of the reactor vessel then stirs the reactor content including the biomass carriers.

In principle, carriers can be purchased from various suppliers. The VUNA reactors used the Kaldnes K1 type carriers with the following characteristics (www.anoxkaldnes.com):

- Specific surface area: $500\ m^2/m^3$
- Maximum fill: 30 to 65 %
- Weight per m^3: $152\ kg/m^3$
- Number of units per m^3: 1 029 000
- Surface per unit: $4.86\ cm^2$
- Percentage of hollow space: 93 %

Figure 2: *Polyethylene biomass carriers provide a large surface for nitrifying bacteria to grow.*

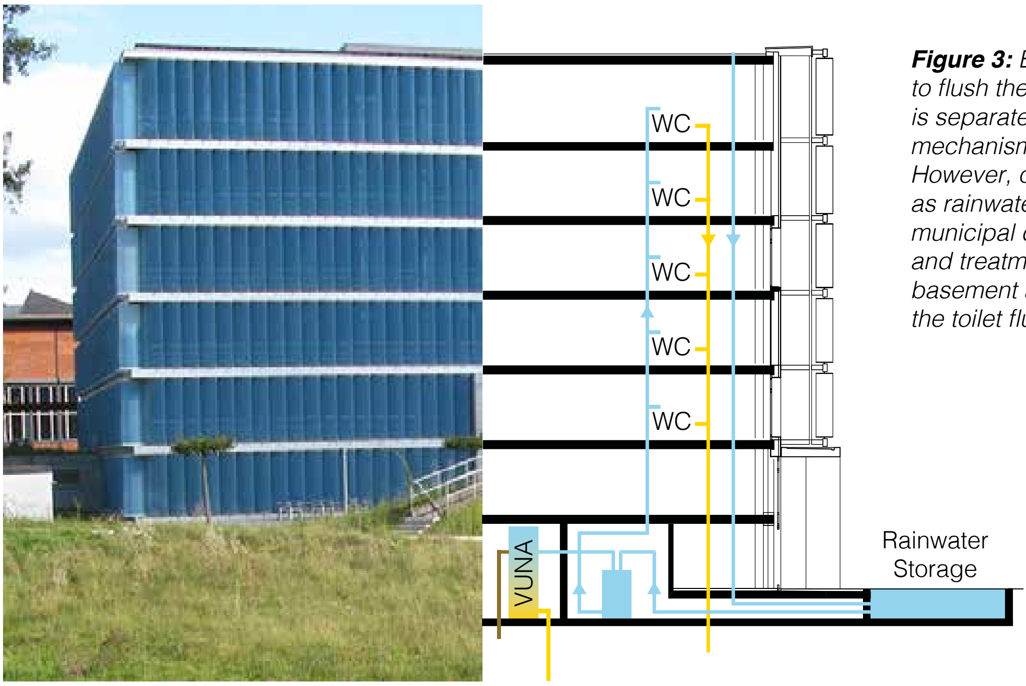


Figure 3: *Eawag's main building uses rainwater to flush the urine-diverting toilets. Although urine is separated from flush water with a special valve mechanism, some water enters the urine pipe. However, only few precipitates form in the pipes, as rainwater is not as hard as water from the municipal distribution system. The urine storage and treatment system is located in the building's basement and the distilled water is recycled into the toilet flush system.*

1.2 Reactor Construction

The following section describes what components are necessary to convert urine to fertiliser at the scale of a building or a neighbourhood. Though the general components and their functioning are outlined, it cannot replace the engineering required to install and operate such a system. The system will have to be carefully adapted to new situations. Furthermore, the technology is still being developed further, so components may vary or be replaced in the future.

System Components

The complete nutrient recovery process developed within the VUNA Project was designed to treat approximately 50 to 100 L of urine per day. This corresponds to the average urine flow at Eawag's main building, Forum Chriesbach (see Links). The VUNA Project also replicated the system twice in South Africa, i.e. once at the Newlands-Mashu Field Test Site and once at the eThekwini Prior Road Customer Care Centre.

The overall system consists of the following components described in the subsequent paragraphs:

1. **Urine storage tank:** Attenuates fluctuations and ensures complete urea hydrolysis (the nitrifying bacteria require ammonium as a substrate, because they cannot degrade urea).

2. **Nitrification column:** An aerated tank containing the nitrifying bacteria mainly on biomass carriers.

3. **Intermediate storage tank:** Stores the (partially) nitrified urine before it is distilled.

4. **Vacuum distiller:** Concentrates the urine and separates distilled water.

5. **Final product storage and recycling:** holds the concentrated nutrient solution and/or distilled water or recycles the water.

Integration into Building Environment

When assessing the potential of integrating the complete nutrient recovery system into a new environment, the full system has to be taken into account: From the urine collection from the toilet users to the use of the final products, i.e. the concentrated nutrient solution and the distilled water. The complete installation has a footprint of approximately 5 m^2, whereas the room accommodating it should not be smaller than 10 m^2. Although the three different VUNA pilots are similar, the building environment varies from one place to another:

- **Eawag:** Urine is collected from 31 urine-diverting toilets and 7 waterless urinals and piped directly to the storage tank in the basement. Rainwater is used to flush the toilets (Figure 3).

- **Newlands-Mashu:** Urine is trucked from household urine-diverting dehydration toilets and stored in large tanks on site.

- **Customer Care Centre:** At present, 3 waterless urinals are connected to the system located in the underground parking. At a later stage, when new urine-diverting flush toilets will be available, urine will be collected from toilets too.

Links

Forum Chriesbach – Eawag's main building:
> www.forumchriesbach.eawag.ch

Brochure with cross section through the building:
> www.eawag.ch/fileadmin/Domain1/About/Nachhaltig/forum-chriesbach/dl/flyer_e.pdf

Newlands-Mashu Research Site – PRG/EWS field research:
> prg.ukzn.ac.za/field-sites/newlands-mashu-research-site

Nitrification Reactor

In the VUNA project, transparent PVC columns were used as vessels for the nitrification reactor. As the reactors were to be used as a demonstration, the process had to be well visible to visitors. However, other plastic materials, such as PP or PE are also suitable for the reactor construction. The column was filled with a bulk volume of 60 % biomass carriers to form a moving-bed biofilm reactor (MBBR).

The volume of the nitrification reactor has to be calculated based on the average ammonium concentration in the stored urine and the maximum nitrification rate. In the VUNA reactors, the maximum nitrification rate varied from 100 to 800 mg/L/d (as ammonium-nitrogen NH_4-N transformed into nitrate NO_3-N per litre of reactor volume per day).

As the ammonium concentration in urine also varied from 2 000 to 4 000 mg/L (as ammonium-nitrogen), one column of 120 L liquid volume was able to treat from 6 to 100 L urine per day. Important factors influencing the maximum nitrification rate are temperature and the maturity stage of the biofilm. To operate under ideal conditions, temperature should not drop below 20°C. Better rates were obtained at higher temperatures comprised between 25 and 30°C. The VUNA reactors repeatedly featured very high rates (up to 800 $mg \cdot L^{-1} \cdot d^{-1}$) within months of start-up (see 1.3 Nitrification Start-up Procedure, p. 10), before they dropped back to rather low rates (100 mg/L/d) due to limited oxygen diffusion in an increasingly thick layer of biofilm.

Vacuum Distiller

To concentrate a solution, distillation is a well-known process. In the case of nitrified urine, water is the component with the lowest boiling point. Hence, at a certain temperature, water evaporates and leaves behind an increasingly concentrated solution. Alternatively, an increased concentration of the nutrient solution can also be achieved with one of the following processes:

- Reverse osmosis: High pressure forces water through a membrane, which retains dissolved salts.
- Humidification: a transfer from the liquid to the gas phase below the boiling point of water.
- Membrane distillation: A gas-permeable membrane lets water vapour pass at a lower temperature difference.

For the VUNA pilots, we opted for an industrial vacuum distiller, because it was readily available and easy to embed into the nutrient recovery plant. In the generated vacuum inside the distiller at about 0.5 bar, the water in urine starts boiling at approximately 85°C. This had the significant advantage of being below the recommended maximum operating temperature set by safety concerns (see 1.9 Process Safety, p. 13).

We selected a Prowadest vacuum distiller produced by the company KMU-Loft Cleanwater located in Hausen in Southern Germany (see Links). The distiller uses vapour compression to generate heat and recovers heat from the condensing distillate to achieve an optimal energy efficiency. An additional heat exchanger transfers the heat from the condensed distillate to the inflowing urine, thus cooling the distillate and pre-heating the urine to be treated. The distiller is able to distil approximately 30 L urine per hour at a power uptake of 3 kW, resulting a specific energy consumption of 100 Wh/L treated urine. Compared to the nitrification reactor, the distiller's capacity is about one to two orders of magnitude higher. However, no smaller distillers featuring equally high energy efficiency are available on the market. With the alternative processes described, alternative sources of energy, e.g. solar or off-heat, may be used to reduce the use of electricity.

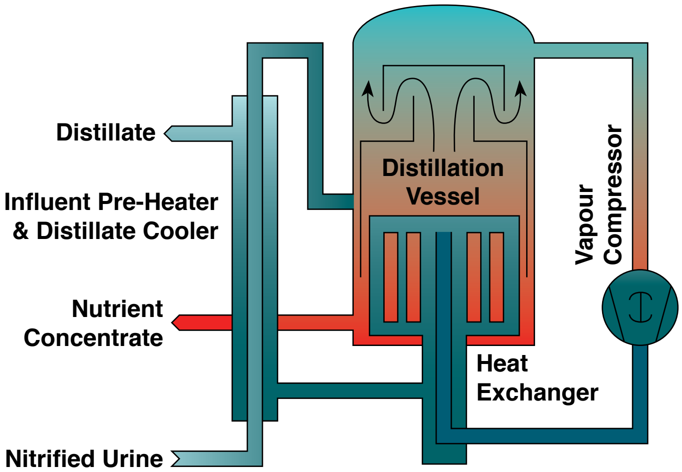


Figure 4: *The Prowadest distiller used in the VUNA pilots operates at a reduced pressure of approximately 0.5 bar to lower the boiling point to 85°C. It generates heat through vapour compression and recovers heat in integrated heat exchangers.*

Recommended Publications:

- *Etter, B., Hug, A., Udert, K.M. (2013)* ***Total nutrient recovery from urine – operation of a pilot-scale nitrification reactor.*** *WEF/IWA International Conference on Nutrient Removal and Recovery 2013, 28-31 July, Vancouver, Canada.*
- *Fumasoli, A., Etter, B., Sterkele, B., Morgenroth, E., Udert, K.M. (2015)* ***Operating a pilot-scale nitrification/distillation plant for complete nutrient recovery from urine.*** *Water Science & Technology, 73(1), 215-222.*
- *Udert, K.M., Wächter, M. (2012)* ***Complete nutrient recovery from source-separated urine by nitrification and distillation.*** *Water Research 46(2), 453-464.*

Links

Producer of the vacuum distiller used in the VUNA pilots:
> www.kmu-loft.de

Membrane distillation plants (not yet tested with urine):
> www.solarspring.de

Figure 5: *The complete nutrient recovery pilot plant at Eawag's basement.*

Legend:
1. Nitrificaiton columns
2. Intermediate storage tanks
3. Vacuum distiller
4. Distillate tank
5. Nutrient concentrate tank
6. Process control unit
7. Target computer
8. Aeration control
9. Vent pipe

Instrumentation and Process Control

The following online sensors are integrated into the plant:

- **Urine storage:** level
- **Nitrification reactor:** pH, temperature, dissolved oxygen
- **Intermediate storage tank:** level
- **Distiller:** pressure, temperature, electric conductivity

Data of these sensors is recorded in 1-minute intervals and logged directly into a data base for an easy analysis.

The influent pump is controlled by the pH value in the nitrification reactor. For details refer to the recommended publications.

Air Supply

The air required to aerate the nitrification reactor can either be produced on site with a suitable compressor or can be obtained from a pressured air distribution net, if available in the building.

Piping and Storage Tanks

Although no water is necessary for the complete nutrient recovery process, we recommend installing a water tap for cleaning in the room hosting the reactors and urine tanks.

The nitrification reactor should sit in a retention basin able to retain one complete reactor volume in case of spills or breakages. The room with the reactors should have a floor drain linked to a soak pit or a sewer, to quickly evacuate any spills (Figure 7). The floor drain should immediately be followed by a p-trap to prevent any unwanted odour emissions.

The initial urine storage tank (Figure 6) should be sized in order to attenuate any daily, weekly or even seasonal fluctuations, as the nitrification process requires a steady input. The intermediate storage tank should hold a volume at least equal to one distillation batch. The distillate and concentrate tanks have to be dimensioned based on the further use of each liquid.

Electricity supply

Depending on the reactor size, the influent pump, the air compressor, and the process control unit can be powered with a normal 230 V, 10 A electricity outlet. At a scale beyond 1000 L nitrification volume, a 400 V 3-phase connection will be necessary for the air compressor. However, the distiller used in the VUNA pilots requires a 400 V, 16 A 3-phase connection even in its smallest size.

Ventilation

In order to meet the safety standards (see Chapter 1.9 Process Safety, p. 13), the room has to be sufficiently ventilated. A minimum of one air change rate per hour is recommended. The intermediate and distillate storage tanks should be vented, i.e. directly connected to the off-air pipe of the ventilation system in addition to the normal room ventilation. Sufficient air space for air intake into the room should be provided.

Figure 6: *The urine storage tanks at Eawag each contain 1 000 L urine to attenuate daily and weekly fluctuations. The urine dosage pump (orange/blue) is installed on the tank.*

Figure 7: *The nitrification column at eThekwini's Customer Care Centre sits on a basin to retain spills. A floor drain acts as a back-up for any spills that do not end up in the basin.*

Figure 8: *A sludge inoculum is taken from the nitrification basin at eThekwini's Norther Treatment Works.*

1.3 Nitrification Start-up Procedure

VUNA started up three pilot nitrification plants: the first one at Eawag's main building near Zurich, the second one at the Newlands-Mashu Field Test Site in eThekwini, and the third one at EWS' Customer Care Centre in downtown Durban. All pilot plants were nearly identical.

The main drivers of the nitrification process are the nitrifying bacteria. As every living being, they need to adapt to new environments and need to be taken care of, i.e. provided with ideal growth conditions. To initiate the process of growing nitrifying bacteria in urine, we first need to source a suitable bacteria population. In the VUNA nitrification reactors, we used sludge containing nitrifying bacteria taken from municipal wastewater treatment plants (Figure 8). Given that the substrate concentrations in municipal wastewater are about 100 times lower compared to urine, the bacteria first had to adapt to the high concentration in urine. To assure a gradual adaptation and prevent a shock exposure to high nutrient concentrations, we established the following start-up procedure, which was followed in every nitrification plant:

Main VUNA Reactor Parameters

- Total volume of a nitrification reactor: 150 L
- Total liquid volume: 120 L (up to overflow)

Initial Ingredients (as % of liquid reactor volume)

- Biomass carrier bulk volume: 50%
- Nitrifying sludge liquid volume: 20%
- Tap water: fill up to reach 100%
- Calculate the amount of urine necessary to reach a total NH_4-N concentration of 100 mg/L in the reactor.

Start-Up Procedure

- Add biomass carriers, sludge, and tap water to the reactor.
- Adjust the aeration to achieve complete mixing of the reactor contents (including biomass carriers).
- Add the exact amount of urine as calculated (see above).
- Set the pH control to switch on the influent urine pump, when the pH drops below 6.20 and to switch it off again, when the pH increases beyond 6.25.

During start-up, sample at least twice weekly as indicated in the following section, in order to monitor concentrations.

1.4 Steady-State Operation

This section outlines the repeating tasks necessary to guarantee a reliable reactor operation. It is likely that more reliable online sensors will develop in the near future. Hence, longer maintenance intervals will become possible and less human interaction will be necessary.

Aeration

Set the aeration rate to maintain a well stirred nitrification column and a dissolved oxygen level above 4 mg/L. A rotameter can be helpful to indicate the air flow entering the process.

Chemical Analyses

For details on laboratory analyses of chemical parameters to be measured in the process, refer to Chapter 8 Urine Analysis, p. 20. Sample 1 to 2 times per week:

- **Influent:** pH, COD, NH_4^+, NO_2^-, NO_3^-, PO_4^{3-}
- **Reactor content:** pH, COD, NH_4^+, NO_2^-, NO_3^-, PO_4^{3-}

It is assumed that the nitrified urine leaving the reactor into the intermediate storage tank has a composition equal to the one in the reactor. Therefore, the sample taken from the reactor represents the nitrification process' effluent.

After distiller operation, if required:

- **Distillate:** pH, EC, COD, NO_2^-, NH_4^+
- **Concentrate:** pH, EC, COD/TOC, NH_4^+, NO_3^-, PO_4^{3-}, K^+, SO_4^{2-}

If the concentrate is mixed in a concentrate holding tank, a composite sample can be taken, before the concentrate is bottled. As micro-nutrients are an important component in the concentrated liquid fertiliser, we also recommend to analyse:

- Boron (B), iron (Fe), zinc (Zn), manganese (Mn)

As potentially harmful substances, the following components have to be monitored in the concentrated liquid fertiliser:

- Na^+, Cl^-, possibly pharmaceutical residues

Maintenance

Weekly to monthly (depending on stability of signals):

- Calibrate pH and dissolved oxygen probe

Yearly:

- Flush urine pipes entering the reactor.
- Calibrate influent dosing pump.
- Distiller maintenance according to supplier's specifications.
- Refill anti-foaming agent in distiller, if required.

1.5 Investment Costs

The complete nutrient recovery plants tested in Durban and Zurich are prototypes and thus relatively expensive. Certain instrumentation was required for research purposes only and can be omitted in industrial plants. We expect the costs to significantly decrease in the future, as plants become more industrial.

Table 3: *Approximative costs of the complete nutrient recovery plants tested in Durban and Zurich. The plants are based on a nitrification reactor of 120 L.*

Component	***Costs in SA [ZAR]***	***Costs in CH [CHF]***
Dosing pumps	4 000	4 000
Air compressor	5 000	5 000*
Nitrification column	7 000	3 000
Storage tanks	4 000	1 000
Distiller	40 000 €	40 000 €
Sensors	40 000	6 000
Process control	140 000	10 000
Piping & installation	100 000	10 000
Total	**780 000 ZAR****	**82 000 CHF****

* Air supply from pressured air network at Eawag; estimated cost for compressor
** Based on average exchange rates for 2012: 1 € = 1.20 CHF = 12 ZAR

1.6 Distillate Reuse

The distillate produced in the distillation process is an important side product at up to 97 % of the initial urine volume. Although one might consider the distillate to be distilled water, it contains some impurities: a residual ammonium concentration of 30 to 60 mg/L is typical. Also, a thin oil layer can sometimes be observed floating on the distillate. It originates from tiny leakages in the vacuum pump.

Considering these impurities, there are different ways to reuse the distillate:

6. **Toilet flush water:** At Eawag, the distillate is pumped back into the toilet flush water storage. Normally, the distillate is diluted by a factor of 100 or more with the rainwater present in the toilet flush water tank. In an extreme case, when the storage is at its lowest level and the distillate of one complete batch is mixed with it, we still obtain a minimal dilution of 1:20. However, even at such low levels, no odours or other compromising effect is observed in the flush water.

7. **Irrigation:** At the Newlands-Mashu site, the distillate is used as irrigation water. The residual ammonium as a minor fertilising effect on the irrigated crops.

8. **Battery water:** Lead-acid batteries used e.g. in motor vehicles occasionally need top-up battery water. A comparison between relevant standards on battery water and the distillate composition shows that the latter requires further polishing to meet the standards (Table 4). Polishing can be achieved with ammonia stripping, i.e. by injecting air into the distillate. The ammonia entrained with the air can subsequently be dissolved in sulphuric acid to produce a nitrogen (and sulphur fertiliser). As a result, the ammonium is stripped from the distillate and an ammonium sulphate solution is produced, which can be used as a fertiliser as well.

Table 4: *Comparison of standards for lead-acid battery water and distillate composition (adapted from specifications given by FNB – First National Battery, South Africa and the Kenya Standard on Water for lead-acid batteries).*

Parameter	***South Africa****	***Kenya*****	***Distillate***
pH	5 to 7	not specified	9 to 11
Colour	transparent	transparent	transparent
Odour	absent	absent	slight odour
NH_4^+ (as N)	5 mg/L	8.2 mg/L	30 to 60 mg/L
NO_3^- (as N)	5 mg/L	5 mg/L	none
EC	30 µS/cm	not specified	500 µS/cm

* First National Battery Specifications

**Kenya Standard DKS 2258:2010: Water for lead-acid batteries – Specification

1.7 Distiller corrosion

In the distiller, all components in contact with any liquid are manufactured from stainless steel (1.4539), except:

- **Sacrificial anode:** A piece of copper bolted to the steam deflector in the main distillation vessel. It had to be removed after excessive copper concentrations were detected in the concentrated nutrient solution. No negative effects on the steel components were observed thereafter.

- **Body of the vacuum pump:** Cast steel; according to our experience of 3 years distiller operation, it is the only piece susceptible to corrosion (Figure 9).

The distiller used in the complete nutrient recovery pilots was delivered with a default programmed anti-blockage protection, which activates the vacuum pump at given intervals for 1 second. One-hour intervals were found to be sufficiently short to prevent blockages due to corrosion in the pump.

In general, the distiller was inspected and serviced twice a year, as indicated by the supplier. During the inspections, no solid scaling was observed inside the distiller.

Figure 9: *The cast steel body of the vacuum pump was the only distiller component susceptible to corrosion.*

1.8 Use of Concentrate

Table 5 presents an overview on the concentrated nutrient solution's main compounds.

Table 5: *Measured nutrient contents of the concentrated nutrient solution at Eawag (own measurements).*

Nutrient	***NH_4-N***	***NO_3-N***	***PO_4-P***	***K***	***Na***	***Cl***
Content [%]	2.1	2.1	0.2	1.7	1.3	3.1

Nutrient	***SO_4***	***Ca***	***Mg***	***B***	***Zn***
Content [%]	0.8	0.04	0.004	0.002	0.001

1.9 Process Safety

Compounds causing significant environmental and health concerns occur at three stages of the nutrient recovery process:

- **Ammonia:** When storing large volumes of urine containers, which are not airtight, or when handling large urine volumes in contact with the air, significant amounts of ammonia volatilise. Ammonia is a health hazard if inhaled and can have detrimental environmental effects if released in an uncontrolled way.

- **Nitrite:** During the nitrification process, nitrite may accumulate due to process instabilities. Nitrite is toxic or may volatilise as harmful nitrous acid.

- **Ammonium nitrate:** The main compound of the concentrated nutrient solution is ammonium nitrate. In a dry state, ammonium nitrate is highly explosive. Also, when in solution maximum operating temperatures have to be observed.

Respecting safety procedures during process operation is crucial. Therefore, the following section focuses on urine storage and distillation process. Nevertheless, critical compounds may occur at any other stage of the complete nutrient recovery process and have to be handled with the necessary precautions.

Ammonia Volatilisation

Ammonia is a strong irritant to the respiratory system depending on its concentration. In large urine storage tanks, ammonia can accumulate in the headspace and become a health hazard, if the tanks are opened. When handling urine in large volumes, always wear adequate protective equipment, such as a mask equipped with ammonia filters, gloves and goggles.

Ammonia is highly toxic to aquatic animals. Therefore, extreme care has to be taken not to release urine into water bodies.

Nitrite Accumulation and Nitrous Acid Production

In the case of nitrite accumulation during the nitrification process, care must be taken to completely oxidise all nitrite to nitrate, before the solution is distilled. As nitrite is toxic for plants, it must not be contained in the final fertiliser product. In the case of high nitrite concentrations in the nitrification reactor, nitrogen might convert to nitrogen oxides, which are harmful to humans and the environment. Hence, nitrite accumulation during the nitrification process is not solely an issue compromising process stability, but also affects the environment.

Thermal Stability of Ammonium Nitrate

Ammonium nitrate is thermally unstable, i.e. it decomposes violently, if heated above a critical temperature. Based on the experiments and simulations carried out during the VUNA Project, the following temperatures are critical:

- **170°C:** In solution, ammonium nitrate must not be heated above 170°C to avoid thermal decomposition. However, as long as ammonium nitrate is in solution, a maximal boiling temperature of 130°C will not be exceeded. We therefore recommend not to evaporate the liquid beyond the point, when the first solids start to precipitate.

- **96°C:** If all liquid is evaporated and solid ammonium nitrate remains, 96°C is the absolute safety value (refer to Udert et al., 2015 for details). Thus, one has to ensure that sufficient liquid is present at all times during the distillation process.

Under normal operating conditions in vacuum distillers, the above-mentioned temperatures are not exceeded. The systems tested in VUNA typically operate at 0.5 bar and 80 to 85°C. However, in the case all liquid accidentally be evaporated, the temperature may come close to the critical threshold.

Legal Considerations and Possible Solutions

Given its thermal instability, ammonium nitrate is regulated in most countries. In addition to ammonium nitrate, two compounds in the concentrated nutrient solution can further increase the risk of thermal decomposition: Chloride and organic substances. Therefore, ammonium nitrate fertilisers are regulated:

- **In South Africa:** ammonium nitrate fertilisers are considered and regulated as explosives, if they a) contain > 25% nitrogen or b) contain > 16% of nitrogen and > 0.4% combustible material (RSA, 1956).

- **In the European Union and Switzerland:** ammonium nitrate fertilisers with > 16% nitrogen may only be sold to farmers and professional gardeners (EEC, 2008) and with > 28% nitrogen face a series of other restrictions on pH, chloride content (max. 0.02%), particle size, and combustible material content (max. 0.4%) (EEC, 2003).

The ammonium nitrate concentration in the concentrated solution (4.2% nitrogen) is sufficiently low that no further measures have to be taken to use the solution as a fertiliser. However, if all the water is removed, the ammonium nitrate concentration (21% nitrogen) exceeds the first legal threshold (16% nitrogen). Hence, in Europe the dried solid could only be sold to professionals (other limits do not apply below 28% nitrogen content). In South Africa, the limit for combustible materials would also be exceeded in the dried solid (approximately 0.5%). Thus a solid would have to be declared as an explosive entailing complex legal procedures and difficult handling, except if it was mixed with limestone. This is a common practice for commercial fertilisers. In South Africa the limestone content of the fertiliser would have to be at least 20% (RSA, 1956).

Legislation on ammonium nitrate fertilisers:

- *EEC (2008)* ***Restrictions on the Marketing and Use of 2-(2- methoxyethoxy)ethanol, 2-(2-butoxyethoxy) ethanol, Methylenediphenyl Diisocyanate, Cyclohexane and Ammonium Nitrate.*** *Decision No 1348/2008/EC of the European Parliament and of the Council.*
- *EEC (2003)* ***Regulation No 2003/2003 of the European Parliament and of the Council of 13 October 2003 Relating to Fertilisers.*** *European Parliament and Council.*
- *RSA – Republic of South Africa (1956)* ***Explosives Act.*** *Government of South Africa, Act No. 26 of 1956.*

2 Nitrification

The complete nutrient recovery relies on a functioning nitrification process. Therefore, mechanisms underlying nitrification of urine were thoroughly studied in the VUNA Project: Alexandra Fumasoli (formerly Hug) carried out her PhD research on "Stabilisation of urine with nitrification as pre-treatment for nutrient recovery" (Fumasoli, 2016). The following paragraphs present an overview on her research as well as the relevant publications.

Acid-Tolerant Bacteria

Ammonia oxidation decreases the pH in wastewater. However, ammonium oxidising bacteria (AOB) decrease or even cease their activity at low pH. In spite of that, low pH has been detected in urine nitrification plants, because "The growth of nitrosococcus-related ammonia oxidizing bacteria causes strong acidification in high strength nitrogen wastewater" (Fumasoli et al., 2016). Based on their findings, the team developed guidelines for a "Stable ammonia conversion to nitrate and the prevention of unfavourable system states during nitrification of urine" (Fumasoli et al., 2016).

Pilot scale nitrification

Details on nitrification rates, bacterial growth and further mechanisms in the pilot nitrification reactor are summarised in the publication "Complete nutrient recovery from urine in a pilot-scale nitrification/distillation plant." (Fumasoli et al., 2015).

Complete vs. Partial Nitrification

In her master's thesis, Corine Uhlmann compared the dynamics of partial nitrification – as it occurs in the VUNA pilot plants, limited by the natural alkalinity in urine – with complete nitrification, i.e. a conversion of 100% ammonium to nitrate by adding a base (Uhlmann, 2014). She found that complete nitrification did neither improve nor aggravate the process stability in comparison to partial nitrification. It was rather temperature fluctuations that had a destabilising effect on nitrification performance.

Publications on Urine Nitrification:

- *Fumasoli, A. (2016)* ***Stabilization of urine with nitrification as pre-treatment for nutrient recovery****. PhD thesis, ETH Zurich.*
- *Fumasoli, A., Morgenroth, E., Udert, K.M. (2016)* ***Stable ammonia conversion to nitrate and the prevention of unfavorable system states during nitrification of urine.*** *In preparation.*
- *Fumasoli, A., Weissbrodt, D., Wells, G.F., Bürgmann, H., Mohn, J., Morgenroth E., Udert K.M. (2016)* ***The growth of nitrosococcus-related ammonia oxidizing bacteria causes strong acidification in high strength nitrogen wastewater.*** *Submitted to Environmental Science & Technology.*
- *Fumasoli, A., Etter, B., Sterkele, B., Morgenroth, E., Udert, K.M. (2016)* ***Complete nutrient recovery from urine in a pilot-scale nitrification/distillation plant.*** *Water Science & Technology, 73(1), 215-222.*
- *Uhlmann, C. (2014)* ***Dynamics of complete and partial nitrification of source-separated urine.*** *Master's thesis, ETH Zurich.*

3 Distillation

To concentrate the nitrified urine, distillation was selected as a process. Michael Wächter carried out his post-doctoral research on the topic. To learn more about urine distillation, refer to the publications outlined in the following paragraphs.

First laboratory distillation experiments

In laboratory experiments, Michael Wächter delivered the proof of concept that the combination of nitrification and distillation works as the complete nutrient recovery process. The publication on "Complete nutrient recovery from source-separated urine by nitrification and distillation" (Udert & Wächter, 2012) documents these experiments and assesses the technology's potential in terms of energy efficiency, recovery rates, and process safety.

Process Safety

As safety is of concern for the entire nutrient recovery process, the essential facts are given in the previous chapter (1.9 Process Safety, p. 13). For a comprehensive documentation of safety aspects during distillation, as well as a description of the underlying experiments, refer to the publication "Safety assessment for production and storage of nitrified and concentrated fertilizer from human urine" (Wächter et al., 2016).

Selective Crystallisation of Sodium Chloride

Samuel Huber examined in his master's thesis (Huber, 2011), how sodium chloride can be removed from nitrified urine by selective crystallisation. Given that both sodium and chloride compromise the quality of a fertiliser product, we want to produce a fertiliser with minimal sodium and chloride content. In his research, Samuel Huber showed that a selective crystallisation is possible, if the process is kept with a strictly controlled temperature range (Wächter et al., 2016).

Publications on Urine Distillation:

- *Huber,S.J. (2011)* ***Temperature dependent removal of sodium chloride (NaCl) from synthetic nitrified urine.*** *Master's thesis Karlsruhe Institute of Technology.*
- *Udert, K.M., Wächter, M. (2012)* ***Complete nutrient recovery from source-separated urine by nitrification and distillation.*** *Water Research 46(2), 453-464.*
- *Wächter, M., Huber, S., Kluge, J., Mazzotti, M., Udert, K.M. (2016)* ***Selective crystallization of sodium chloride (NaCl) from partially nitrified urine.*** *In preparation.*
- *Wächter, M., Schwaninger, M., Gmeinwieser, T., Udert K.M. (2016)* ***Safety assessment for production and storage of nitrified and concentrated fertilizer from human urine.*** *In preparation.*

4 Electrolysis

In electro-chemical urine treatment, an electrical current passing through the urine transforms certain substances. The technology has the advantage that it can be integrated into existing systems, as it requires only a small space. In addition, one can modify process parameters, such as current or potential easily, to achieve the desired transformation. The main drawbacks are high energy consumption and the release of by-products.

In the VUNA project, Hanspeter Zöllig did his PhD research on "Electrolysis for the treatment of stored source-separated urine" (Zöllig, 2015). He designed, conducted, and analysed extensive laboratory experiments to understand the processes underlying electro-chemical urine treatment. The chemical reactions occurring during electrolysis can be grouped into three main processes (Figure 10) at three different levels of anode potential. Besides the anode potential, another important parameter influencing the process is the electrode material. The following paragraphs summarise Hanspeter Zöllig's research on these various aspects of electro-chemical urine treatment.

Direct Electro-Chemical Ammonia Oxidation

At low anode potentials, ammonia is directly oxidised to nitrate and gaseous nitrogen. In their publication "Direct electrochemical oxidation of ammonia on graphite as a treatment option for stored source-separated urine" (Zöllig et al., 2015), the team showed that graphite anodes are suitable, as they are sufficiently selective, i.e. they only convert the targeted substances, in this case ammonia. In addition, graphite is available at a low price. However, due to the low potential, the reactions are comparably slowly. In another series of experiments, the team identified an "Inhibition of direct electrolytic ammonia oxidation due to a change in local pH" (Zöllig et al., 2015). They had experimented with electrodes coated in thermally decomposed iridium oxide films (TDIROF), which had been shown to be catalytically active for direct ammonia oxidation. To prevent such an inhibition, the researchers propose a modified electrolysis cell design to create favourable hydraulic conditions.

Indirect Electro-Chemical Ammonia Oxidation

In an intermediate range of anode potentials, ammonia is oxidised indirectly, i.e. chlorine mediates the reactions from ammonia to nitrate or gaseous nitrogen. Refer to "Electrochemical oxidation of ammonia and organic substances in real stored urine – a polishing step for phosphorus recovery from urine? " (Zöllig et al., 2016) for details.

Formation of Chlorination By-Products

At higher anode potentials, the chlorine produced reacts with organic substances in the urine to form harmful by-products. Along with Hanspeter Zöllig two master's students studied by-products formation: Christina Fritzsche and Annette Remmele. For details refer to their master's theses or to the publication summarising the results "Formation of chlorination by-products and their emission pathways in chlorine mediated electro-oxidation of urine on active and inactive anodes" (Zöllig et al., 2015).

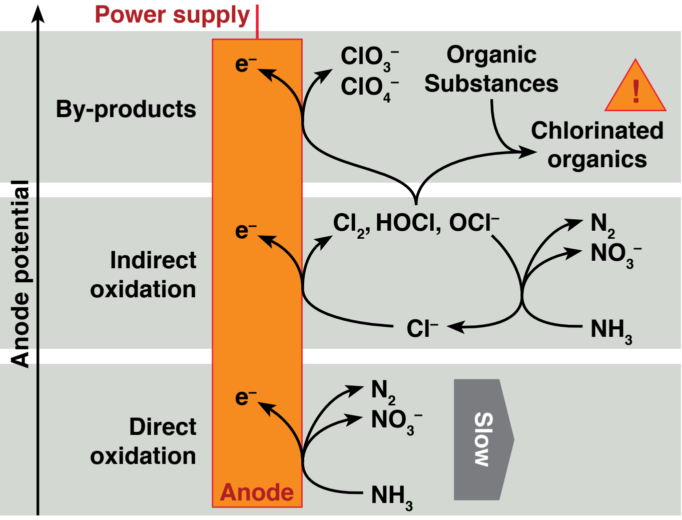


Figure 10: *Processes occurring at an anode in urine vary depending on the anode potential. At low potentials, ammonia is converted to nitrate and gaseous nitrogen at very low rates. At intermediate potentials, the conversion is accelerated by indirect oxidation through chlorine. At higher potentials, organic substances react with chlorine to form harmful by-products. (Concept: Hanspeter Zöllig).*

Publications on Electro-Chemical Urine Treatment:

- *Fritzsche, C. (2012)* ***The formation of chlorinated organics during electrolytic urine treatment.*** *Master's thesis, ETH Zurich.*
- *Remmele, A. (2013)* ***The influence of anode material and current density on the emissions of disinfection by-products (DBPs) during electrolytic treatment of stored urine.*** *Master's thesis, ETH Zurich.*
- *Zöllig, H. (2015)* ***Electrolysis for the treatment of stored source-separated urine.*** *PhD thesis 22854, ETH Zurich.*
- *Zöllig, H., Fritsche, C., Morgenroth, E., Udert, K. M. (2015)* ***Direct electro-chemical oxidation of ammonia on graphite as a treatment option for stored source-separated urine.*** *Water Research 69, 284-294.*
- *Zöllig,H.; Morgenroth,E.; Udert,K.M. (2015)* ***Inhibition of direct electrolytic ammonia oxidation due to a change in local pH.*** *Electrochimica Acta 165, 348-355*
- *Zöllig, H., Remmele, A., Fritzsche, C., Morgenroth, E., Udert, K.M. (2015)* ***Formation of chlorination by-products and their emission pathways in chlorine mediated electro-oxidation of urine on active and inactive anodes.*** *Environmental Science and Technology, 49(18), 11062-11069.*
- *Zöllig, H., Remmele, A., Morgenroth, E., Udert, K.M. (2016)* ***Electrochemical oxidation of ammonia and organic substances in real stored urine – a polishing step for phosphorus recovery from urine?*** *In preparation.*

5 Struvite Precipitation

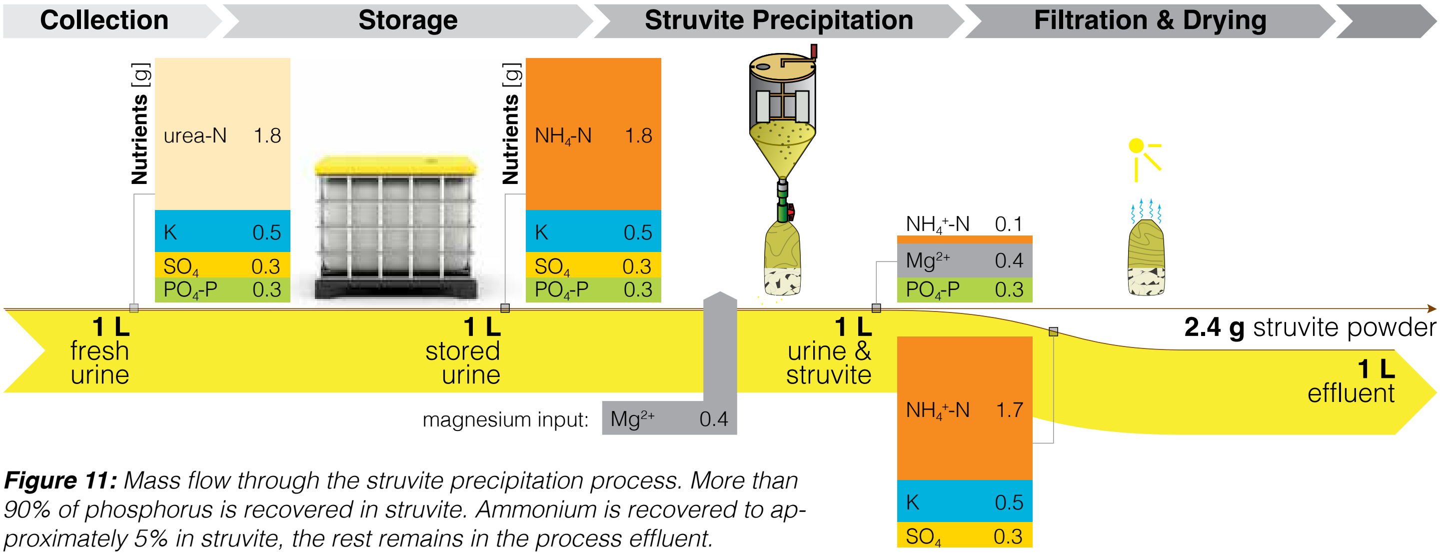


Figure 11: *Mass flow through the struvite precipitation process. More than 90% of phosphorus is recovered in struvite. Ammonium is recovered to approximately 5% in struvite, the rest remains in the process effluent.*

To produce struvite ($MgNH_4PO_4\cdot 6H_2O$) from urine, one has to add magnesium and filter the precipitated powder from the liquid. The product is predominantly a phosphorus fertiliser (12.5%) containing some magnesium (10%) and nitrogen (5%), which can easily be applied to crops and releases the nutrients slowly over time to cover an entire crop period. Besides struvite, phosphorus-depleted urine is generated as a secondary product (Figure 11). Thus, struvite precipitation has to be combined with another technology to achieve full treatment. Nevertheless, the technology suits various environments, given that it operates with or without electricity. Also, the technology can simply be switched on and off on demand, hence it is able to process fluctuating urine volumes.

In the VUNA Project, a team from the University of KwaZulu-Natal and the eThekwini Municipality around Maximilian Grau, Sara Rhoton, Lungi Zuma, Chris J. Brouckart, and Chris A. Buckley developed two struvite reactors for field applications. The following paragraphs present an overview of their work and direct you to relevant documents and publications. For an overview and comparison of different processes, refer to “Technologies for the treatment of source-separated urine in the eThekwini Municipality” (Udert et al., 2015).

Manually Operated Struvite Reactor

The first struvite reactor in eThekwini was adapted from the design of a manual low-cost struvite reactor in Nepal (Etter et al., 2011). The construction manual of the Nepalese reactor is accessible on the website mentioned in the Links Section. Contrary to the Nepalese reactor, which had been build from galvanised iron, the reactor in eThekwini was manufactured from PVC, and was thus corrosion-proof. A sight glass and sturdy pipe connections enabled a convenient operation (Figure 12) under all conditions.

For complete account on the struvite reactors in eThekwini, refer to the conference contributions “Nutrient recovery from urine: Operation & optimization of reactors in eThekwini” (Grau et al., 2012) or “Field operation of a simple struvite reactor to produce phosphorus fertiliser from source-separated urine in eThekwini” (Rhoton et al., 2014).

Automated Struvite Reactor

Based on the manually operated struvite reactor, the team continued developing an automated reactor, to improve efficiency and facilitate handling. In the manually operated reactor, magnesium has to be dosed according to laboratory measurements of the phosphorus concentration in urine. Or, in case no laboratory measurements be possible, the magnesium dose had to be increased to assure that a maximum of phosphorus precipitated. Hence, if magnesium and urine dosage could be automated, the process would no longer require laboratory testing nor excessive magnesium dosage, which would lead to losses.

The team thus ran extensive trials and coded computer simulations to automate the magnesium dosage with the help of turbidity and electrical conductivity measurements. The process is described in “Development of a fully automated struvite reactor to recover phosphorus from source-separated urine collected at urine diversion toilets in eThekwini” (Grau et al., 2015).

Magnesium Sources

As struvite precipitation requires magnesium, the team also searched for alternative magnesium sources. In their previous project, the researchers at Eawag assessed “Struvite precipitation from urine with electrochemical magnesium dosage” (Figure 14; Hug & Udert, 2013) as well as the option to use “Pretreated magnesite as a source of low-cost magnesium for producing struvite from urine in Nepal (Krähenbühl et al., 2016). Similarly, in South Africa, magnesite rock and calcined magnesite is available from a mine site near Malelane in the province of Mpumalanga. Alternatively “Wood ash as a magnesium source for phosphorus recovery from source-separated urine (Sakthivel et al., 2012) was also studied, but did not yield satifactory results.

Figure 12: *The manually operated struvite precipitation reactor produces struvite from urine at the Newlands-Mashu Field Test Site near Durban. The struvite is retained in a filter bag, which is hung beneath the main reactor vessel.*

Figure 13: *The filter assembly (top) of the automated struvite reactor retains the struvite cake on a plain filter cloth (bottom). Odour emissions are reduced by a completely enclosed filter.*

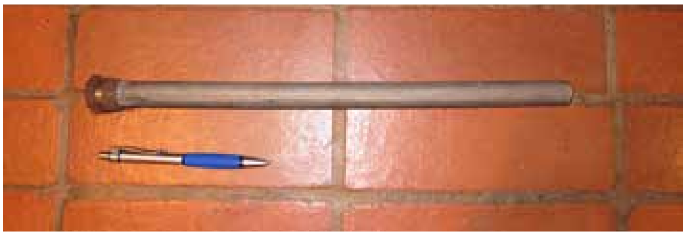


Figure 14: *Alternatively to adding a dissolved respectively soluble magnesium salt as precipitant, a magnesium electrode can be dissolved-electrochemically.*

Links

The Newlands-Mashu Field Research Site with its various nutrient recovery projects, including struvite precipitation:
> prg.ukzn.ac.za/field-sites/newlands-mashu-research-site

STUN – Struvite from Urine in Nepal – VUNA's precursor project developed a low-cost struvite reactor. A construction and operation manual are both available for download on the web:
> www.eawag.ch/stun

European Sustainable Phosphorus Platform – Information on current struvite projects, other phosphorus recycling technologies and phosphorus politics:
> www.phosphorusplatform.eu

Some commercial suppliers of industrial struvite reactors:
> www.ostara.ch
> www.saniphos.eu
> www.nuresys.org
> www.multiformharvest.com

VUNA Publications on Struvite Precipitation from Urine:

- *Grau, M.G.P., Rhoton, S., Brouckaert, C.J., Buckley, C.A. (2015)* ***Development of a fully automated struvite reactor to recover phosphorus from source-separated urine collected at urine diversion toilets in eThekwini.*** *Water SA, 41(3), 383-389.*
- *Grau, M., Etter, B., Hug, A., Wächter, M., Udert, K.M., Brouckaert, C., Buckley, C. (2012)* ***Nutrient recovery from urine: Operation & optimization of reactors in eThekwini.*** *2nd International Faecal Sludge Management Conference, 29-31 Oct, Durban.*
- *Rhoton, S., Grau, M., Brouckaert, C.J., Gounden, G., Buckley, C.A. (2014)* ***Field operation of a simple struvite reactor to produce phosphorus fertiliser from source-separated urine in eThekwini.*** *WISA Biennal Conference, 25-28 May, Mbombela, South Africa.*
- *Udert, K.M., Buckley ,C.A., Wächter, M., McArdell, C.S., Kohn, T., Strande, L., Zöllig, H., Fumasoli, A., Oberson, A., Etter, B. (2015)* ***Technologies for the treatment of source-separated urine in the eThekwini Municipality.*** *Water SA, 41(2), 212-221.*

Further Publications on Struvite Precipitation from Urine:

- *Etter, B., Tilley, E., Khadka, R., Udert, K.M. (2011)* ***Low-cost struvite production using source-separated urine in Nepal.*** *Water Research, 45, 852-862.*
- *Hug, A., Udert, K.M. (2013)* ***Struvite precipitation from urine with electrochemical magnesium dosage.*** *Water Research, 47, 289-299.*
- *Krähenbühl, M.; Etter, B.; Udert, K.M. (2016)* ***Pretreated magnesite as a source of low-cost magnesium for producing struvite from urine in Nepal.*** *Science of the Total Environment, 542, 1155-1161.*
- *Sakthivel, S.R., Tilley, E., Udert, K.M. (2012)* ***Wood ash as a magnesium source for phosphorus recovery from source-separated urine.*** *Science of the Total Environment, 419, 68-75.*

6 Process Control

As indicated in the previous chapter on urine nitrification (2 Nitrification, p. 14), nitrite accumulation is a severe threat to a stable process operation. As currently no methods are available to reliably measure nitrite electronically in the system, the SPIKE Group at Eawag assessed two methods to detect the nitrite concentration:

- **Ultraviolet (UV) spectrophotometric** measurements attributing UV light absorption at a specific wavelength to nitrite concentration.
- **Model-based observers** calculating a nitrite estimate based on other measurable process parameters, such as pH or dissolved oxygen concentration.

SPIKE is the research group for Sensor management, Process Intelligence and Control at Eawag. For the VUNA Project, Alma Mašić and Kris Villez coordinated the studies on nitrite detection. Their comprehensive website (see Link Section) contains most relevant publications for free download.

UV-Spectrophotometry to Detect Nitrite

Monitoring of nitrite is essential for an immediate response to prevent irreversible failure of urine nitrification reactors. Although a few sensors are available for nitrite measurement, none of them are suitable for applications in which both nitrite and nitrate are present in very high concentrations, such as in urine. The SPIKE researchers showed that "Estimation of nitrite in source-separated nitrified urine with UV spectrophotometry" (Mašić et al., 2015) is feasible. The study was backed up with numerous experiments at laboratory and pilot scale. Four master's students worked on the topic: Ana T.L. Santos, Angelika Hess, Elisabeth Grimon, and Katja Briner (see Publications Section for details on their work).

The main challenge of detecting nitrite using spectrophotometric measurements lies in differentiating between the absorption caused by nitrite respectively nitrate. In nitrified urine, nitrate is present at high concentrations. Therefore, detecting nitrite at concentrations, which are 3 orders of magnitude lower, requires specific algorithms taking into account several measured wavelengths.

Model-Based Observers

If a given parameter in a process cannot be measured, other inherent, easily measurable parameters can serve as a basis for an estimate. The process is described in a computer-model, which at its turn, serves to calculate the wanted estimate based on the measured parameters. This is called a model-based observer.

The SPIKE team studied how one can estimate the nitrite concentration in the nitrification reactor knowing the momentary and past pH as well as the dissolved oxygen concentration. Their results are best summarised in "Model-based observers for monitoring of a biological nitrification process for decentralized wastewater treatment" (Mašić & Villez, 2014).

Figure 15: *A UV-spectrophotmetric probe in the laboratory.*

Publications on Process Control in Urine Treatment:

- *Briner, K. (2015)* ***Calibration of a UV-Vis spectrophotometer for simultaneous estimation of nitrate and nitrite in nitrified urine.*** *Master's thesis ETH Zurich.*
- *Hess, A. (2015)* ***Feasibility of UV-Vis spectrophotometry for nitrite estimation in urine nitrification systems.*** *Master's thesis, ETH Zurich.*
- *Mašić, A., Santos, A., Etter, B., Udert, K.M., Villez, K. (2015)* ***Estimation of nitrite in source-separated nitrified urine with UV spectrophotometry.*** *Water Research, 85, 244-254.*
- *Mašić, A., Santos, A., Etter, B., Udert, K.M., Villez, K. (2015)* ***Estimation of nitrite concentration in a urine nitrification reactor by means of UV spectrophotometry.*** *IWA Conference on Nutrient Removal & Recovery, 18-21 May, Gdansk, Poland.*
- *Mašić, A., Villez, K. (2014)* ***Model-based observers for monitoring of a biological nitrification process for decentralized wastewater treatment.*** *IWA Conference on Ecotechnologies for Wastewater Treatment, 23-25 June, Verona, Italy.*
- *Santos, A.T.L. (2014).* ***Evaluation of UV-spectrophotometry for estimation of nitrite and nitrate in nitrified urine.*** *Master thesis, Universidade Nova de Lisboa.*

Links

SPIKE – the research group for Sensor management, Process Intelligence and Control at Eawag (most publications online):
> www.eawag.ch/en/department/eng/main-focus/sensors-and-automation

7 Other Processes

The processes used to recover or transform nutrients from urine in the VUNA Project were: nitrification, distillation, electrolysis, and struvite precipitation. Researchers around the world have tested other processes to recover nutrients from urine. This section presents an overview on the main processes, which have been studied, in case you need to evaluate suitable processes for your urine. For an extended overview on urine treatment processes, refer to the respective chapter in Larsen et al. (2013).

Ammonia Stripping

Given that struvite precipitation recovers mainly phosphorus, but only limited amounts of nitrogen, additional treatment has to be provided, in order to achieve a satisfactory effluent quality. Stripping has been tested in various projects to recover ammonia from ammonia-rich wastewater. In this process, air is forced through urine to entrain the gaseous ammonia. In most cases, the air is then injected into a tank containing sulphuric acid, which at its turn, reacts with the gaseous ammonia to form an ammonium sulphate solution. An ammonium stripping plant was installed at a wastewater treatment plant near Eawag for the "Recovery of nitrogen and phosphorus from urine by struvite precipitation followed by combined stripping with digester sludge liquid at full scale" (Morales et al., 2013).

Stabilisation with Lime – $Ca(OH)_2$

As part of the Autarky Project (see Link Section), "A novel approach for stabilizing fresh urine by calcium hydroxide addition" (Krähenbühl et al., 2016) has been developed. The process is to be integrated directly into the toilet (Figure 16), as lime inhibits the degradation of urea into ammonium. Thereby, lime addition stabilises fresh urine chemically and pre-treats it for further treatment via humidification.

Publications on Other Urine Treatment Processes (Beyond VUNA):

- *Krähenbühl, M., Randall, D.G., Köpping, I., Larsen, T.A., Udert, K.M. (2016)* ***A novel approach for stabilizing fresh urine by calcium hydroxide addition.*** *Submitted to Water Research.*
- *Larsen, T.A., Udert, K.M. and Lienert, J. (2013)* ***Source separation and decentralization for wastewater management.*** *IWA Publishing, London, UK.*
- *Maurer, M., Pronk, W., Larsen, T.A. (2006)* ***Treatment processes for source-separated urine.*** *Water Research, 40, 3151-3166.*
- *Morales, N., Boehler, M.A., Buettner, S., Liebi, C., Siegrist, H. (2013)* ***Recovery of nitrogen and phosphorus from urine by struvite precipitation followed by combined stripping with digester sludge liquid at full scale.*** *Water 2013, 5, 1262-1278.*
- *Schielke-Jenni, S. (2015)* ***Decentralised urine treatment with the nitritation/anammox process.*** *PhD thesis ETH Zurich.*

Links

The Autarky toilet combining stabilisation of urine by lime (CaOH) addition with humidification:
> www.autarky.ch

Figure 16: *The so-called autarky toilet, which is currently being developed. The future toilet will unite an integrated urine and faeces treatment, as well as a water recycling system. To present, the treatment processes have been tested in the laboratory (Render by EOOS).*

Humidification

In the Autarky Project, the urine stabilised by lime is humidified: flowing through a ventilated pipe, the liquid evaporates at ambient temperature. As the temperature gradient is not as high as, for instance, in distillation, the evaporation per volume is lower. Though, simultaneously, the energy demand is also lower.

Anammox

"Decentralised urine treatment with the nitritation/anammox process" (Schielke-Jenni, 2015) was tested as an option to eliminate nitrogen from urine. Although the process works under certain conditions, it has shown a limited performance.

Other Processes

Although published a decade ago, the review "Treatment processes for source-separated urine" (Maurer et al., 2006) still provides an extensive overview of potential urine treatment processes and their respective advantages and drawbacks. The review evaluates the following processes, which are not described in this manual: freeze-thawing, reverse osmosis, acidification, ion-exchange, electro-dialysis, nano-filtration, ozonation.

Other Substrates

Most of the described processes are suitable to treat other waste streams with high ammonium content, such as liquid manure, landfill leachate, or digester supernatant. On the other hand, processes, which have been tested for ammonium-rich waste streams, might be suitable to treat urine too.

8 Urine Analysis

To understand the functioning of urine treatment processes, we have to be able to precisely measure chemical parameters at various stages. Our experience has shown, that chemical analysis of urine differs from analysis of other substances, given the complex composition of urine. This chapter presents how to obtain reliable results from your chemical analysis in urine treatment processes. In certain cases, it is necessary to produce synthetic urine, to study specific mechanisms. With synthetic urine, all ingredients are well know, and it is thus easier to break down an observation or measurement to its origins. Thus, the chapter also presents recipes for synthetic stored and nitrified urine.

Publications on Urine Composition:

- *Fumasoli, A., Etter, B., Sterkele, B., Morgenroth, E., Udert, K.M. (2016)* ***Complete nutrient recovery from urine in a pilot-scale nitrification/distillation plant.*** *Water Science & Technology, 73(1), 215-222.*
- *Udert, K.M., Wächter, M. (2012)* ***Complete nutrient recovery from source-separated urine by nitrification and distillation.*** *Water Research 46(2), 453-464.*
- *Udert, K.M., Larsen, T.A., Gujer, W. (2006)* ***Fate of major compounds in source-separated urine.*** *Water Science & Technology, 54(11-12), 413-420.*
- *Udert, K.M., Larsen, T.A., Biebow, M., Gujer, W. (2003)* ***Urea hydrolysis and precipitation dynamics in a urine-collecting system.*** *Water Research, 37, 2571-2582.*

8.1 Typical Concentrations

Concentrations of nutrients and other ions in urine vary greatly, depending on the physical conditions, diet, environmental factors influencing every individual. In the VUNA Project, urine was collected at various places from numerous individuals over an extended period of time. Therefore, fluctuations are not as strong as in individual or small urine samples. However, the concentrations we measured are repeatedly lower than certain literature values (e.g. literature values compiled in Udert et al., 2006).

The concentrations measured at Eawag (Table 6) are thought to be representative for an office building. Contrarily to a housing unit, concentrations in an office building might be lower, given that the most concentrated urine is excreted just after people get up in the morning and urinate the first time in a day. Hence, the urine collected throughout the day, is expected to contain slightly less nutrients.

In addition to the daily fluctuations, flush water has an important effect on urine concentration. At Eawag, the urine-diverting toilets do require some flush water to convey the faecal matter into the conventional sewer. A small proportion of this flush water may infiltrate into the urine pipe, if the urine-diverting valve is not perfectly water-tight. As men use waterless urinals with no risk of water infiltration into the urine pipe, less water enters the men's urine collection tank, thus the nutrient concentration in the men's tank is significantly higher than in the women's tank.

Table 6: *Concentrations of ions in stored urine used for various experimental series. 1) Urine used in VUNA laboratory nitrification and distillation experiments (Udert & Wächter, 2012); 2) Urine used to assess the precipitation dynamics in separate urine pipes (Udert et al., 2003); 3) Women's urine used in VUNA pilot nitrification and distillation experiments (Fumasoli et al., 2016); 4) Men's urine used in VUNA pilot nitrification and distillation experiments (ibid.); 5) Reference values calculated from literature (Udert et al., 2006). Standard deviations are given as ± values, wherever available*

Component	***1. Lab nitrification***	***2. Modelling***	***3. Women's tank***	***4. Men's tank***	***5. Literature***
	mg/L	*mg/L*	*mg/L*	*mg/L*	*mg/L*
Total ammonia (as N)	2 390 ± 250	1720	1 990 ± 420	4 140 ± 870	8 100
Total phosphate (as P)	208 ± 49	76	106 ± 17	242 ± 23	540
Calcium	16 ± 3	28	13.5 ± 11.0	N/A	0
Magnesium	< 5	1	< 4	N/A	0
Potassium	1 410 ± 320	770	854 ± 143	1 470 ± 130	2 200
Sodium	1 740 ± 360	837	881 ± 239	1 760 ± 90	2 600
Sulfate	778 ± 184	292	308 ± 87	708 ± 109	1 500
Chloride	3 210 ± 530	1400	1 630 ± 400	2 980 ± 440	3 800
Total inorganic carbon	1 210 ± 220	N/A	1 020 ± 250	2 080 ± 260	N/A
Dissolved organic carbon	1 830 ± 360	N/A	N/A	N/A	N/A
Dissolved COD	4 500 ± 910	1 650	2 010 ± 540	3 860 ± 870	10 000
pH [–]	8.69 ± 0.11	9.0	8.9 ± 0.1	9.0 ± 0.1	9.1

8.2 Synthetic Urine Recipes

For laboratory experiments, synthetic urine may replace real urine, if all parameters have to be constant and well controlled. This chapter presents a collection of recipes to produce synthetic urine in the laboratory. These recipes reduce the components contained in urine mainly to the most important minerals. Depending on the recipe, only urea (fresh urine – Table 7) and acetate (stored urine – Table 8) are added as organic compounds. The recipe for nitrified urine (Table 9) does not contain any organic compounds, as nitrification degrades most organic substances.

Synthetic Fresh Urine

Table 7 indicates a standard recipe for fresh urine, i.e. imitating typical concentrations as urine leaves the human body. Urea is included in the recipe as main organic compound. Alkalinity, ionic strength, and pH were calculated using PHREEQC.

Table 7: *Recipe for synthetic* ***fresh*** *urine (left) and corresponding ion concentrations (right). Adapted from Udert et al., 2006.*

Component	**Quantity**
Water (H_2O)	1 L
Urea	16.0 g
NH_4Cl	1.80 g
$Na_2SO_{4\ \text{anhydrous}}$	2.30 g
$NaH_2PO_{4\ \text{anhydrous}}$	2.90 g
KCl	4.20 g
$MgCl_2$	0.370 g
$CaCl_2$	0.510 g
NaCl	0.183 g
NaOH	0.230 g

Notes: *Charge balance on Na. Urea not included in simulation. Alkalinity calculated with final acids SO_4^{2-}, $H_2PO_4^-$, H_2CO_3, NH_4^+. No complexation with citrate nor oxalate.

Ion	**Concentration**
Urea	0.27 M
NH_3	0.0337 M
PO_4	0.0242 M
K	0.0563 M
SO_4	0.0162 M
Cl	0.1101 M
Na	0.0654 M
Mg	0.0039 M
Ca	0.0046 M
Alkalinity*	0.022 M
Ionic strength*	0.183 M
pH*	6.2

Synthetic Stored Urine

Table 8 indicates a recipe for stored urine, i.e. after urea has been hydrolysed into ammonium and carbon dioxide. Also, some phosphorus has precipitated as struvite and hydroxylapatite along with the calcium and magnesium. Acetate is included as the main organic compound. Alkalinity, ionic strength, and pH were calculated using PHREEQC. When preparing this recipe, take extra precautionary measures:

Caution!

The NH_4OH solution is corrosive: work in a laboratory fume hood! Add the NH_4OH solution with a volumetric cylinder or a glass pipette, but not with a micro-pipette with plastic tips, as the solution will dissolve the plastic. Add NH_4HCO_3 as last substance, to prevent CO_2 production.

Table 8: *Recipe for synthetic* ***stored*** *urine (left) and corresponding ion concentrations (right). Adapted from Udert et al., 2006.*

Component	**Quantity**
Water (H_2O)	1 L
NH_4-Acetate	9.60 g
$Na_2SO_{4\ \text{anhydrous}}$	2.30 g
$NaH_2PO_{4\ \text{anhydrous}}$	2.10 g
NaCl	3.60 g
KCl	4.20 g
NH_4OH_{sol} (25% NH_3)	13.0 mL
NH_4HCO_3	21.40 g

***Notes:** Alkalinity calculated with final acids HSO_4^-, $H_2PO_4^-$, HAc, H_2CO_3, NH_4^+. No complexation with citrate nor oxalate.

Ion	**Concentration**
Acetate	0.125 M
NH_3	0.568 M
PO_4	0.0175 M
K	0.0563 M
SO_4	0.0162 M
Cl	0.118 M
Na	0.111 M
CO_3	0.271 M
Alkalinity*	0.584 M
Ionic strength*	0.637 M
pH*	8.87

Synthetic Nitrified Urine

For experiments on distillation or fertiliser trials, it might be of interest to directly produce a synthetic urine, which imitates the qualities of nitrified urine. The recipe in Table 9 is based on the theoretical concentrations in stored urine (Udert et al., 2006). We assumed that all nitrogen in fresh urine was hydrolysed to ammonia, 25% of the total ammonia volatilised and 50% of the remaining ammonia was oxidised to nitrate.

Table 9: *Recipe for synthetic* ***nitrified*** *urine (left) and corresponding ion concentrations (right). Adapted from Supplementary Information in Udert & Wächter, 2012.*

Component	**Quantity**
Water (H_2O)	1 L
NH_4NO_3	19.2 g
$NaH_2PO_4 \cdot 2H_2O$	2.7 g
KCl	3.4 g
$KHCO_3$	1.1 g
$Na_2SO_{4\ \text{anhydrous}}$	2.3 g
NaCl	3.6 g
HCl 32%	0.4 mL

Ion	**Concentration**
NH_4-N	3.4 mg/L
NO_3-N	3.4 mg/L
PO_4-P	0.5 mg/L
K	2.2 mg/L
SO_4	1.5 mg/L
Cl	3.9 mg/L
Na	2.5 mg/L
TIC	0.13 mg/L

Publications Including Synthetic Urine Recipes

- *Udert, K.M., Wächter, M. (2012)* ***Complete nutrient recovery from source-separated urine by nitrification and distillation.*** *Water Research 46(2), 453-464.*
- *Udert, K.M., Larsen, T.A., Gujer, W. (2006)* ***Fate of major compounds in source-separated urine.*** *Water Science & Technology, 54(11-12), 413-420.*

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Project Partners: