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Demonstration of Phosphorus Extraction

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EXECUTIVE SUMMARY

Sewage sludge is produced as an unwanted byproduct of wastewater treatment which can be used as a secondary raw material for construction products. It is also an important source of Phosphorus (P), a critical raw material. With the Phosphorus recovery pilot plant developed within the CINDERELA project we demonstrate that it is possible to recover significant amount of the P (as well as nitrogen and other valuable nutrients) by source-seperation of the urine from the rest of the sewage stream. As a concequence (i) the need for costly P elimination at the wastewater treatment plant, and (ii) the amount of this valuable resource that ends up in the sewage sludge can be reduced.

We have revised the approach for P recovery with respect to what was written in the original CINDERELA proposal to recover P in the form of a nutrient-rich liquid fertilizer rather than as struvite. The revised, complete nutrient recovery approach has a number of advantages, namely (i) that Nitrogen and other valuable nutrients for agriculture are recovered along with the P and (ii) that the full urine stream is treated, removing pathogens and pharmaceuticals, with distilled water as the only by-product.

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EXPLANATION OF ACRONYMS & ABBREVIATIONS

Acronym	Full name
CRM	Critical Raw Material
D	Deliverable
LCA	Life Cycle Assessment
LCC	Life Cycle Cost
N	Nitrogen
NIGRAD	NIGRAD d.d. komunalno podjetje
P	Phosphorus
S-LCA	Social Life Cycle Assessment
SRM	Secondary raw material
TUDelft	TECHNISCHE UNIVERSITEIT DELFT
WHO	World Health Organization
WP	Work package
WWTP	Wastewater treatment plant
ZAG	ZAVOD ZA GRADBENISTVO SLOVENIJE

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1. INTRODUCTION

Sewage sludge, a by-product of municipal wastewater treatment, is one of the most voluminous and cost-incurring waste streams in urban areas[1] (as shown for the Slovenian case, in D3.1);. Paradoxically, it is also a waste stream of great value, often containing high concentrations of Phosphorus (P) - a finite mineral, and irreplaceable nutrient for plant growth and food production.

There are diverse utilization practices for sewage sludge, regulated by the legislation of each country (Figure 1**Error! Reference source not found.**). Traditionally, the most common route to valorize the phosphorus present in sewage has been direct application on arable land, thus recycling this essential nutrient back into the agri-food chain. However, due to socio-ecological constrains this practice is decreasing and, in some cases, being completely eliminated[2] (see e.g. in the Netherlands, where 100% of sewage sludge is incinerated, Figure 1). Therefore, there is a need to valorize the sewage sludge in other ways, all the while recovering the nutrients it contains to recycle them back into agriculture.

Figure 1 Fate of sewage sludge in different EU countries. Data taken from EUROSTAT, for the year 2015. Source: "Sewage Sludge Disposal in the Federal Republic of Germany" [3]

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Dewatered and hygienized, sewage sludge has the potential to be used as a secondary raw material (SRM) in the construction sector. Some characteristics of sewage sludge-based composites to be used as a construction product were assessed in CINDERELA deliverable D3.3. However, ideally, Phosphorus should be recovered from the sewage sludge before it ends up "wasted" in a construction product (Figure 2).

Recovery of P can take place (i) from the sludge streams at the wastewater treatment plant, or (ii) upstream in the wastewater treatment chain, directly from human excreta, before it is diluted and sent to the wastewater treatment plant (WWTP). P can also be extracted from the sludge ash after incineration. This approach is less relevant to our purposes, as CINDERELA studies the use of sewage sludge directly as an SRM. Nevertheless, we also address the option of P extraction from sludge ash below, as there seems to be a trend in promoting the incineration of sewage sludge[2] and sludge ash is also a potential SRM for construction products (see Smol *et al.* 2015[4]).

Figure 2 Sewage sludge produced as an unwanted by-product of wastewater treatment can be used as a secondary raw material for construction products. Phosphorus, a critical raw material, should be recovered from the municipal wastewater before it ends up in the sludge. Left: dewatered sewage sludge at a wastewater treatment plant (source: Monica Conthe), Right: sludge-based composite as shown in D3.3.

For CINDERELA Task 6.1 - Pilot demonstration of extraction of phosphorus from municipal waste – we opted for an upstream P recovery approach, and in this Deliverable - D6.8 - we document the construction and start-up of a demonstration plant for the recovery of phosphorus (and other nutrients) from source-separated urine. Important changes have been made in the implementation of this demo with respect to what was proposed in the original DoA, and we discuss and justify these changes below.

Early upstream extraction of P, nitrogen (N) and other valuable nutrients directly from urine allows us to demonstrate a cascade waste to resource approach: nutrient recovery from wastewater for use where demand is greatest - as fertilizer, followed by the use of sewage sludge as secondary raw material for application as construction materials component where nutrients would be of no added value, and irreversibly wasted.

We are aware that this demo showcases only one, of numerous approaches to recover P in the wastewater chain. In reality, no one technology is perfect or overall better than another:

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improvements are still required for all the P recovery pathways, and likely a combination of all of them, depending on local conditions and legislation, will be necessary to transition to a circular P economy. We therefore briefly discuss other P recovery approaches in this deliverable, and refer to relevant literature for more detailed information on these approaches. The difference in carbon footprint and economic sustainability of different P recovery scenarios will be assessed in more detail within WP7, by means of LCA, LCC, and S-LCA analysis (as discussed in section **Error! Reference ource not found.**). Our goal is that this demo will serve as a platform to make the public aware of the importance of P recovery, the existence of different alternatives and the imperative need to transition to a circular economy in both nutrient management and construction practices.

1.1. The importance of Phosphorus recovery

Phosphorus is an essential nutrient for plant growth and, thus, a critical element for agriculture and food production. Unfortunately, the current model for managing P in our food cycle is linear and unsustainable. Modern agriculture relies heavily on the use of mineral fertilizers as a source of nutrients. This is problematic on the two sides of the linear P chain: extraction (where it is scarce) and disposal (where it is in excess).

Extraction: Currently, the major source of P is apatite (or rock phosphate; Figure 3), a finite source which, much like fossil fuels, took millions of years to form but we are rapidly depleting. 90% of apatite is used for fertilizers, and continuing business as usual, with a growing pressure of global population growth, it is estimated that phosphate rock may be depleted in the next 50 to 100 years [5]. Furthermore, mineral phosphate deposits are limited to few locations around the world, and its export depends on fossil fuel intensive extraction and transport.

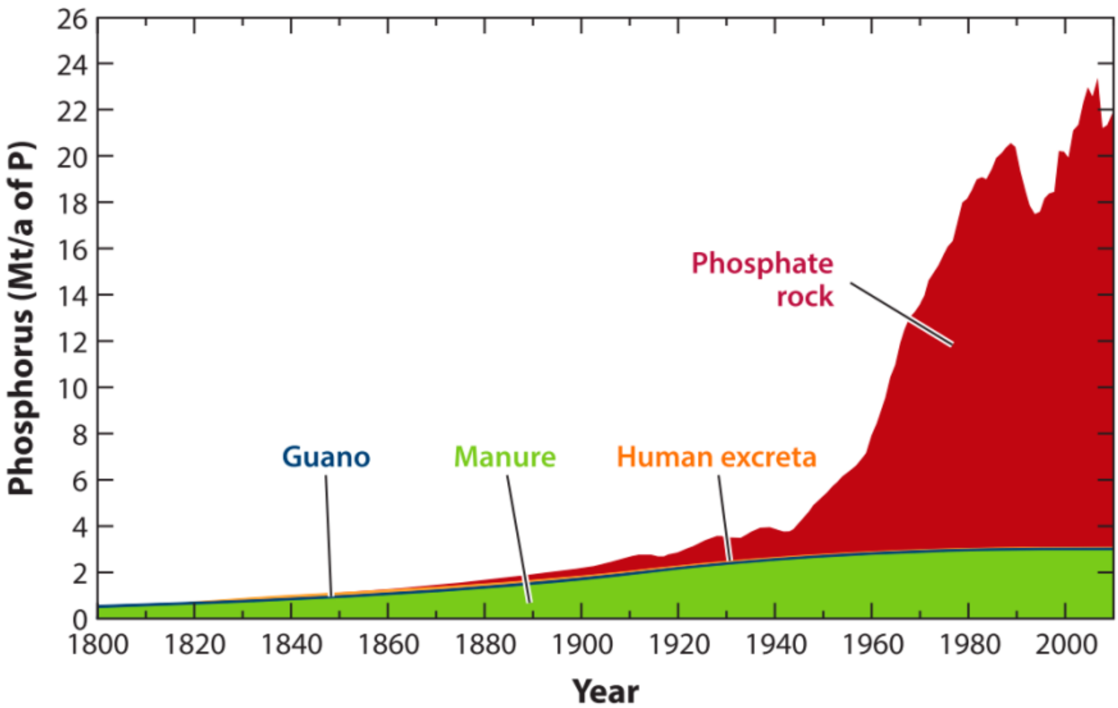

Figure 3 Historical sources of phosphorus for use as fertilizers. Taken from Cordell and White, 2014[6]

Disposal: The intensive mining of P and use of mineral fertilizers is dramatically increasing the input of available phosphorus into natural ecosystems. Large amounts of P are lost at several points in the agri-food chain and end up in water bodies where it contributes to eutrophication, and in the long

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term to a potential anoxic oceanic event [7]. Of the planetary boundaries for human activity set by the scientific community, impacts to the phosphorus biogeochemical cycle (together with the N biogeochemical cycle – to which it is inextricably linked) are already far beyond the established safety threshold to avoid compromising the planetary systems that sustain us (Figure 4).

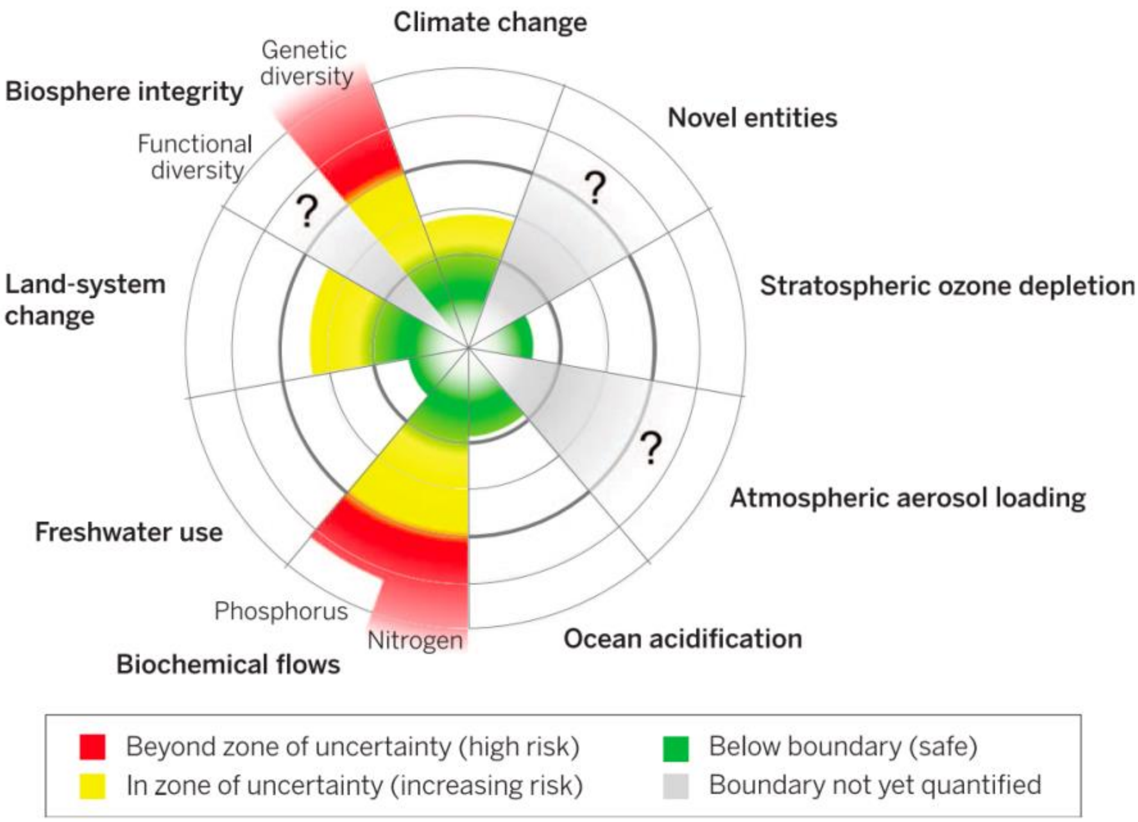

Figure 4 Status of the control variables for seven of the planetary boundaries. Taken from Steffen *et al*. 2015[7]

A circular use of P (and N) is essential to, on the one hand reduce our dependency on fossil and mineral reserves, and on the other avoid the negative ecological impact of "waste" nutrients ending up in the environment. Different types of measures will be necessary to transition to a sustainable use of Phosphorus (Figure 5). "Efficiency" measures are essential, as it is estimated that up to 80% of phosphorus applied in agriculture is lost "from farm to fork"[8], but "Reuse" measures can also contribute significantly. One such measure is recovering P from waste streams and recycling them back into the agricultural production cycle. Relatively little of the P we consume in our diet remains in the human body (i.e. human excreta contains roughly same amount as what we eat), and it has been estimated that recycling P from human excreta could potentially provide up to 20% of phosphorus needs for food production[8].

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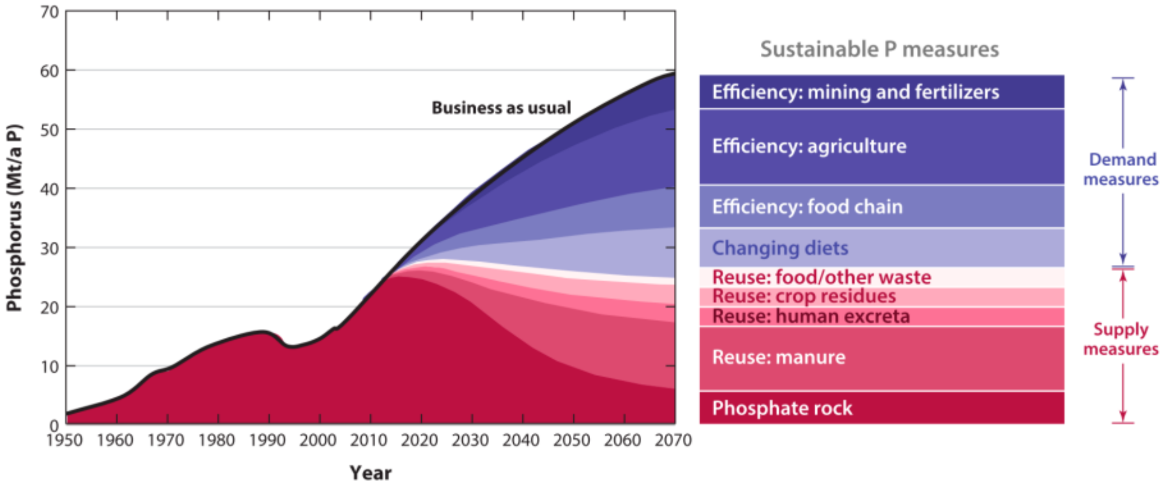

Figure 5 Possible long-term integrated supply-side and demand-side measures for meeting future food security of 9 billion people. Taken from Cordell and White, 2014[6]

Different recovery pathways that can enable us to recycle P from human excreta back into agriculture are presented in section 0. It cannot be emphasized enough, that P recovery reduces the pressure on mineral reserves of P, but moreover, it reduces the impact of lost P on the environment. When recovered upstream of the wastewater treatment process, P recovery also saves energy and resources, by eliminating the need for phosphorus removal at the wastewater treatment plant.

1.1.1 Phosphorus in the EU: a Critical Raw Material

Phosphate rock has been listed as a critical raw material (CRM) by the EU since 2014, based on its economic importance and supply risk (Figure 6). Europe only possesses a small amount of phosphate rock reserves in Finland, and is largely dependent on imports – with Morocco being its main supplier. Currently, much of the phosphorus - more than 90% of which is used in fertilizers and animal feed additives - is lost due to dissipative use, and recovery of P from human excreta would be one of the ways to increase the fraction of secondary materials being functionally recycled to replace primary phosphorus (i.e. to increase the purple arrow on the Sankey diagram in Figure 7, reducing the gray "losses" arrow, and need for imports).

In 2017, the EU included "Phosphorus" as a separate CRM to phosphate rock, referring to elemental phosphorus (P4), or white phosphorus (Figure 6). This is because many "technical" phosphates (e.g. those used in fire safety, lubricants, polymer additives, pharmaceuticals, agrochemicals, catalysts, & metal processing) cannot be produced via phosphoric acid (as are fertilizers and food additives), but via P_4, which is produced in high temperature reducing furnaces. Europe does not have such furnaces, and is totally dependent of imports, mainly from Kazakstan. Although the aim of the CINDERELA demo is to recover P to be used as fertilizer, it is interesting to note that white phosphorus can also ultimately be obtained from human excreta: e.g. the pilot technology RECOPHOS, currently being developed by ICL Fertilizers, recovers P4 from sewage sludge incineration ash (see Figure 9).

Interesting also from the point of view of the CINDERELA pilot, is that other nutrients available in human excreta - magnesium and borate (or boron) - are also listed as CRM, thus adding value to the fertilizer product, if recovered together with P. Potassium, also abundant in human excreta, may also

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be classified as a CRM in the near future (information from the ESPP presentation at the ESCM'2019: European Conference on Sludge Management)

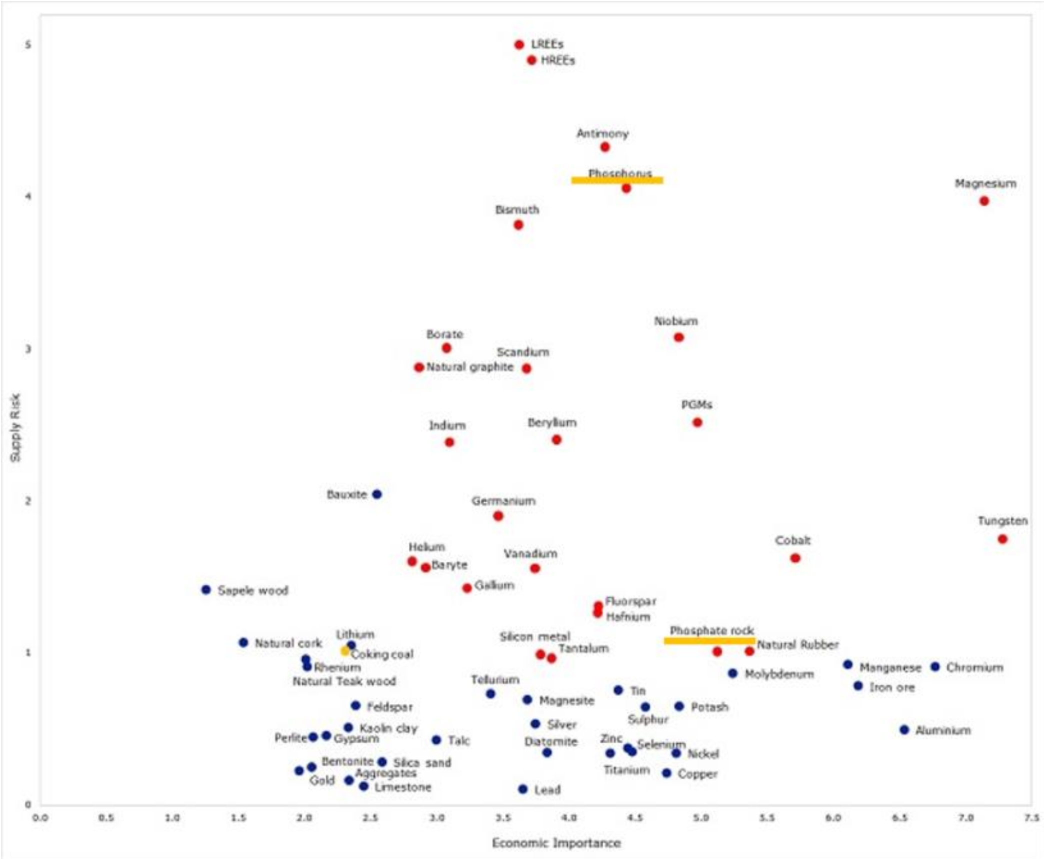

Figure 6 Materials classified as CRM by EU (in red) based on their economic importance and supply risk. Taken from https://ec.europa.eu/growth/sectors/raw-materials/specific-interest/critical_en

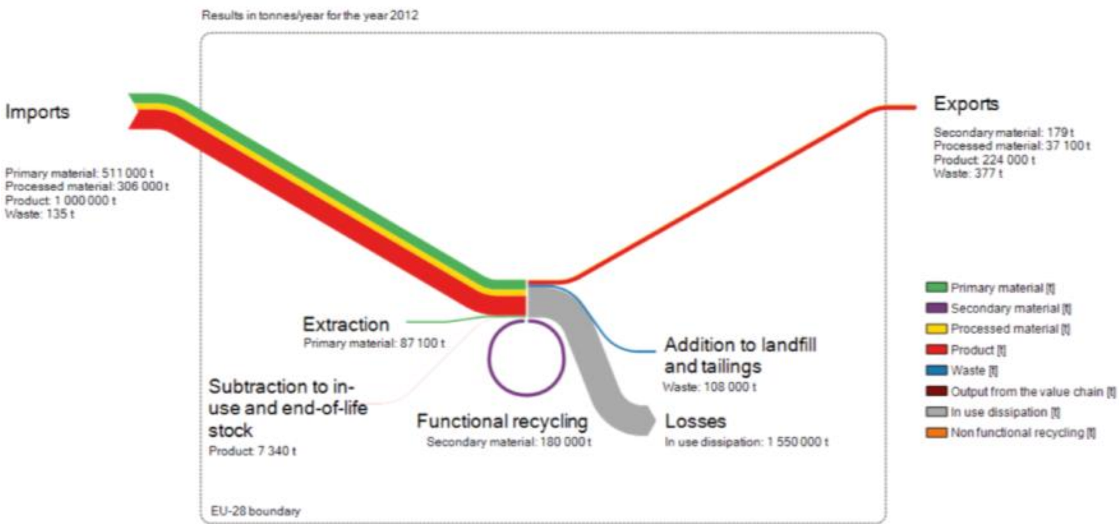

Figure 7 Sankey diagram from phosphate rock in EU, taken from https://ec.europa.eu/growth/sectors/raw-materials/specific-interest/critical_en

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1.2. Phosphorus recovery pathways

Phosphorus can be recovered from different waste streams, through different technologies, obtaining different P products which can be recycled back to food production. Figure 8 maps the flow of phosphorus from mining to disposal. Four phosphorus recovery pathways can be identified: (1) direct reuse of human excreta in agriculture (2) P recovery in the form of struvite, extracted from the sludge streams of the wastewater treatment process, (3) direct sewage sludge application to arable land, and (4) P extraction after incineration of the sludge.

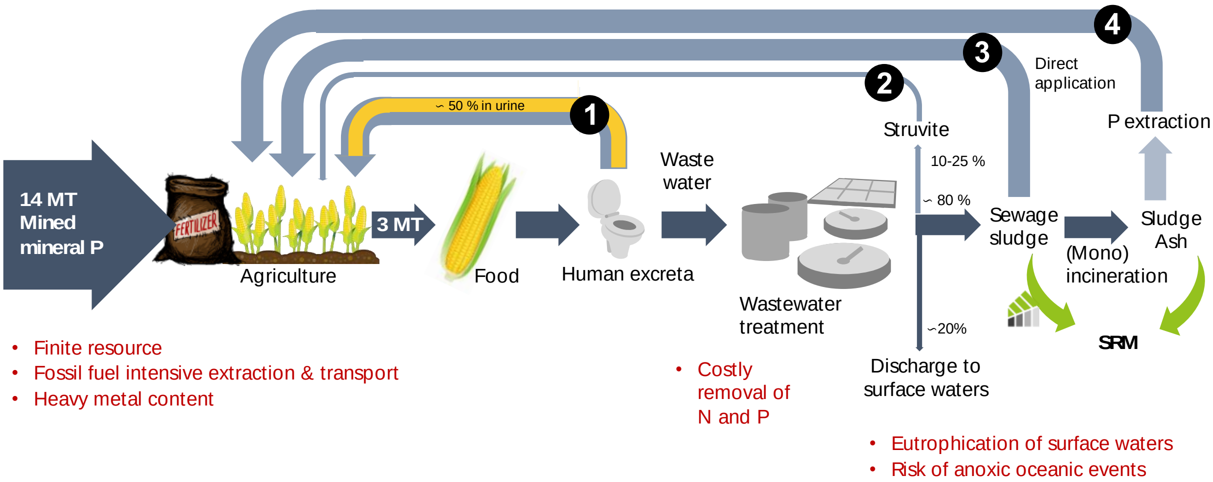

Figure 8: Phosphorus chain - from mining to disposal - and different recovery pathways.

Note that:

- as mentioned earlier, up to 80% of P used in agriculture is lost “from farm to fork”. There are losses which are out of the scope of the P recovery technologies presented here.
- pathways 2, 3 and 4 can only recover P from human excreta entering a centralized sewer system, but the World Health Organization (WHO) estimates only 33 % of P from human excreta worldwide enters a functioning WWTP.
- wastewater (i.e. human excreta diluted with flush water and greywater) has too low of a P concentration for efficient P recovery, therefore pathway 2, P recovery at the WWTP, can only take place from the more concentrated sludge streams
- roughly 10-20% of P entering the WWTP is allowed to remain in the effluent, a fraction which can thus, not be recovered and enters natural ecosystems.
- different technologies for pathways 1,2 and 4 are available, with varying degrees of maturity.
- as mentioned earlier, each country has a different legislation regulating pathway 3, direct application of sewage sludge on arable land.
- the CINDERELA P recovery demonstration plant makes use of pathway 1, direct use of human excreta, via source separation of urine.

Also very important to note is that the P products obtained from each P recovery pathway are very different, some being more suitable for certain agricultural practices, some for others. Struvite is a very different type of P fertilizer than sewage sludge or source-separated urine for example. Even within the category “sewage sludge”, we cannot consider one homogeneous product, since the form in which we find P, and therefore its bioavailability, will depend on the type of wastewater treatment process it originates from (e.g. if P removal is chemical or biological), which, in turn, also affects the

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form in which P is found in sludge ash after incineration. This further reinforces the statement that there is no one perfect pathway for P recovery, but rather, that different pathways should be used to complement each other, and to adapt to different local conditions, agricultural practices, etc. (please refer to van der Kooij *et al*. 2020[8], for a detailed socio-ecological-technological analysis of different P recovery routes, and to Kratz *et al.* 2019[9], for a review on the agronomic performance of P recycling fertilizers).

1.2.1 Recovery via source-separated urine (upstream of the WWTP)

Urine makes up only ~1-2% of the volume of the sewage going to a wastewater treatment plant yet contains ~55% of its phosphorus content, in a concentrated stream low in pathogens and heavy metals. Thus, separation of urine at the source before it joins the sewer system is an attractive hotspot to recover and important fraction of P in human excreta. This P will not reach the wastewater treatment plant, and in this way will not end up in the sewage sludge or discharged to the environment. Furthermore, source-separation of urine reduces the burden on the wastewater treatment process by reducing the P load in wastewater. Wastewater treatment plants (WWTP) are forced to cope with increasingly stringent P discharge limits and P elimination is costly and requires either the addition of chemicals (e.g. ferric chloride, aluminium sulphates) and/or increased reactor volumes for enhanced biological phosphorus removal (EBPR). Aside from P, urine contains ~80% of the nitrogen in sewage, which can also be recovered (further reducing the burden of nitrogen removal from WWTP), and other essential nutrients (potassium, boron, …) for plant growth.

Perhaps the greatest drawback or limitation to this approach is the source-separation in itself, as conventional toilets do not allow for the separation of urine from flush water and feaces.

For recovery of the total P in human excreta, source separation of urine can be combined with composting of faeces. An alternative to source separation of urine is nutrient recovery from blackwater (i.e. the combined stream of urine, faeces, and flushwater), which is the approach used in the Buiksloterham case study (see section 0).

1.2.1.1 Struvite from source-separated urine

Struvite precipitation is one of the most thoroughly researched and understood methods of nutrient recovery from source separated urine, with a number of tested pilot projects[10] [11] [12] [13]. Given the high ammonia and phosphate concentration in urine, along with its high pH (after urea hydrolysis), only addition of a Magnesium source, in low tech reactor systems, is necessary to precipitate nearly all the phosphate as struvite ($MgNH_4PO_4 \cdot 6H_2O$). The main advantage of such an approach is its low degree of complexity, and the fact that P is recovered as a compact solid product, easy to transport. However, while nearly all the dissolved phosphate can be removed, the vast majority of the nitrogen, the potassium and sulphur (along with other micronutrients) remain in the effluent.

1.2.1.2 Complete nutrient recovery

Source-separated urine can be used directly, without treatment, as a fertilizer. Important disadvantages to this practice are, however: (1) the necessity of transporting of large volumes (urine is still a quite diluted fertilizer), (2) the volatilization of ammonia when urea is hydrolyzed, resulting in loss of nitrogen to the atmosphere, along with a pungent odor. There are also rising concerns about the pharmaceuticals present in urine, which would then be potentially reintroduced into the food chain.

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For these reasons, different approaches have been developed to stabilize, and avoid ammonia volatilization of urine, to reduce its volume for easier transportation, and to remove traces of pharmaceuticals potentially present in the urine. The CINDERELA demo, based on the VUNA technology[14], uses nitrification for stabilization of the urine, and vacuum distillation for volume reduction (as described in section 2.2), but e.g. ion exchange, or lime addition are other alternatives to avoid ammonia volatilization; and reverse osmosis, forward osmosis, convective evaporation, membrane distillation, are other alternatives for volume reduction (please see Chipako and Randall, 2020[15] for an up-to-date review).

1.2.2 P recovery via struvite precipitation (or other technologies) at the WWTP

Struvite ($MgNH_4PO_4{\cdot}6H_2O$) precipitation can be used to recover P from the sludge lines in wastewater treatment. Struvite can from spontaneously in certain parts of the wastewater treatment plant, as magnesium (Mg), ammonium (NH_4^+) and phosphate (PO_4^-) are all present in the wastewater and tend to crystallize. The crystallization process can be enhanced by the addition of Magnesium (which is present in lower concentrations than phosphate or ammonia) and by increasing the pH (which can be achieved by addition of a base, or simply by means of aeration).

There are numerous full-scale examples of P recovery in the sludge line of WWTP by struvite crystallization (e.g. Airprex®, Ostara Pearl®, Phosphaq®, etc.). Limitations to this technology for P recovery are that (1) only a relatively small fraction can be recovered: limited to 5-25% of the total P load to the WWTP and (2) this option is only possible in plants with enhanced biological P removal (EBPR) and anaerobic digestion of the waste activated sludge. In fact, the main driver for struvite production (currently) is not the recovery of P in and of itself, but to reducing risk of pipe clogging, abrasion of machinery, by avoiding uncontrolled precipitation of struvite in pipes and equipment, and reducing cost of sludge dewatering (which is easier with less P in the sludge). Nevertheless, due to its low complexity, it is a “low hanging fruit” for P recovery, and different technologies are being developed to recover a greater fraction of P (e.g. technologies which enforce P dissolution, like Wasstrip®, can increase recovery to up to 50%). The “GWRC Phosphorus Compendium Final Report 2019-March-20” presents an extensive and up-to-date review of different processes available on pilot scale and full-scale: E.g. Airprex©, ANPHOS©, CRYSTALACTOR©, ELOPHOS©, PEARL© (see Figure 9: Re-dissolution modules and Recovery modules).

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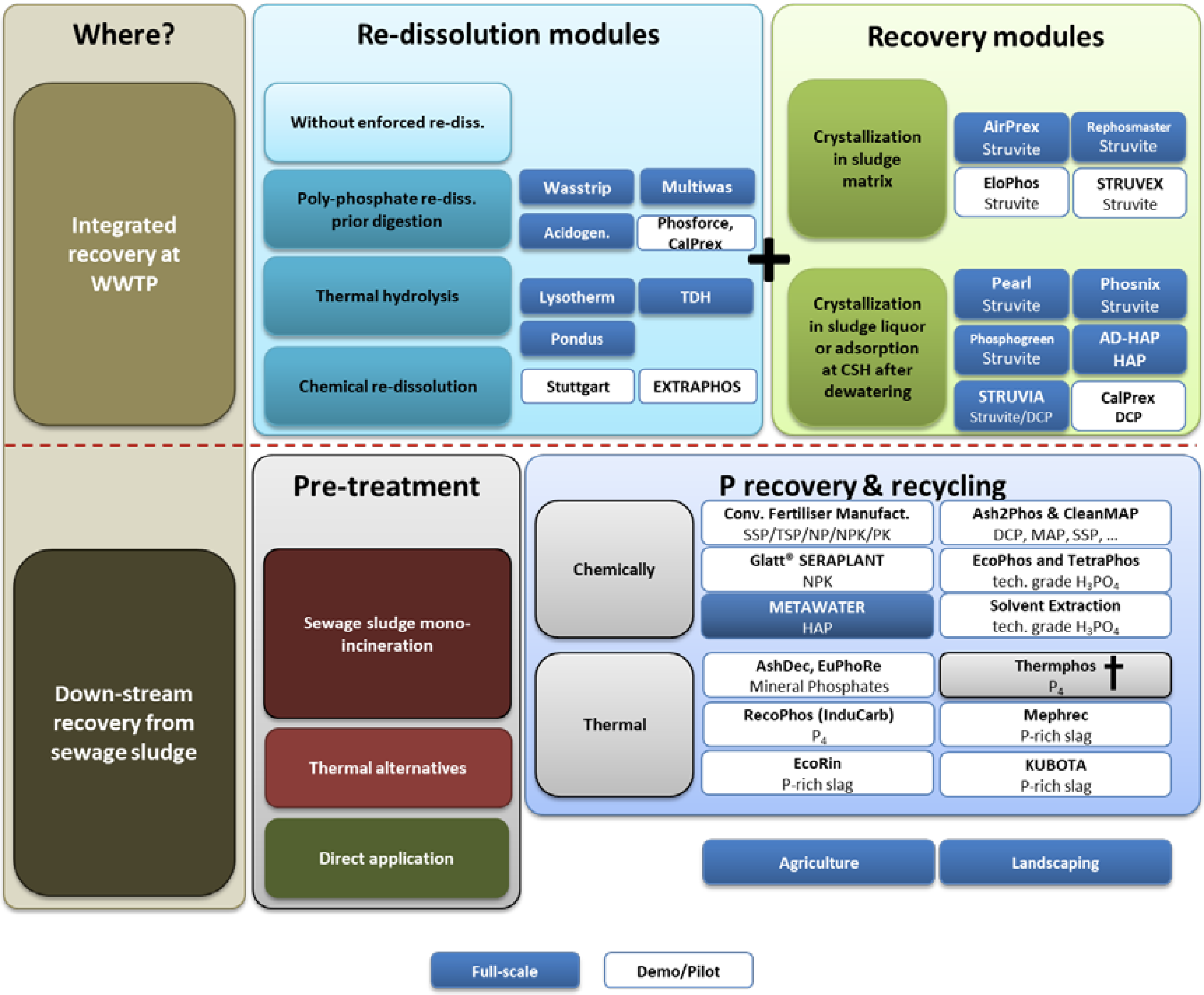

Figure 9: Prominent and already applied concepts for P recovery and recycling from sewage sludge taken from "GWRC Phosphorus Compendium Final Report 2019-March-20"[16]

For wastewater treatment plants that rely on chemical phosphorus removal – i.e. precipitating phosphate (PO_4^-) in the wastewater with Aluminum or Ferric salts, rather than EBPR, research is being performed on recovering ferric phosphates (e.g. vivianite) from the wastewater treatment process by means of magnets (see Vivimag website for more information). This is an important line of investigation since chemical phosphorus removal is widely applied but, further, the presence of iron can also limit the recovery efficiency for struvite in EBPR plants[17].

1.2.3 P recovery from sewage sludge (downstream of WWTP)

1.2.3.1 Direct application on land

In Europe, approximately 25% of P from human excreta is brought back to agriculture via sewage sludge application, after the sludge is dewatered, stabilized and hygienized[8]. However, different countries have different legislation regulating the extent to which this is possible.

Although direct application of sewage sludge on arable land is a straightforward way to recycle P together with organic matter and other nutrients back to agriculture, concerns regarding this practice include: (1) the presence of heavy metals or organic contaminants and (2) the bioavailability

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of the P in the sludge. In fact, the form in which P is present in the sewage sludge, and consequently, how and whether P becomes available to crop, is variable and very much dependent of the type of wastewater treatment process used (E.g. chemical precipitation of P with Al of Fe makes the P less available).

1.2.3.2 P extraction from sludge ash

Sludge ash, resulting from the mono-incineration of sewage sludge (i.e. when sewage sludge is not co-incinerated without with other wastes), has a high P content (roughly 9% on average in Europe[16]), and therefore seems like a promising stream for P recovery. However, considerations to take into account for this pathway are (1) mono-incineration is a must, as mentioned, (otherwise P content is too diluted), (2) thermal or chemical extraction of the P from the sludge ash is necessary as P in the ash is not directly available to plants, and the ash has a high concentration of heavy metals.

There are different technologies available for P extraction from sludge ash, with different degrees of maturity (see Figure 9: P recovery & recycling). Please refer to “GWRC Phosphorus Compendium Final Report 2019-March-20”[16] for an extensive review of the different processes currently being developed and implemented: E.g. Ash2©Phos, EcoPhos©, TetraPhos©.

1.3. P recovery case studies in Amsterdam

As described in the DoA, three existing pilot cases in Amsterdam served as a benchmark case studies from which to define and design Demo 1. The waste stream, recovery process and product obtained for each of these cases is indicated in (Figure 10). The time schedule of the preparations and site visits leading up to the final design and implementation is shown In Figure 11.

Experts in the field consulted, aside from those involved in the 3 case studies, include: Hamse Kjerstadius (part of the the Run4Life H2020 project), Cristian Kabbe (co-founder of the European Sustainable Phosphorus Platform – ESPP), Grietje Zeeman (senior advisor at LeAF), and Mark van Loosdrecht.

Key points identified from the case studies and talks with experts, motivating some changes to proposal in DoA, and discussed further in the following section were: (1) gender inclusion (in De Ceuvel, and Waternet urine collecting events for Foosvatje, only male urinals were used) (2) end use of the fertilizer product (there was no clear market/outlet for the struvite produced at De Ceuvel and Buiksloterham) and (3) recovery of nitrogen and other nutrients.

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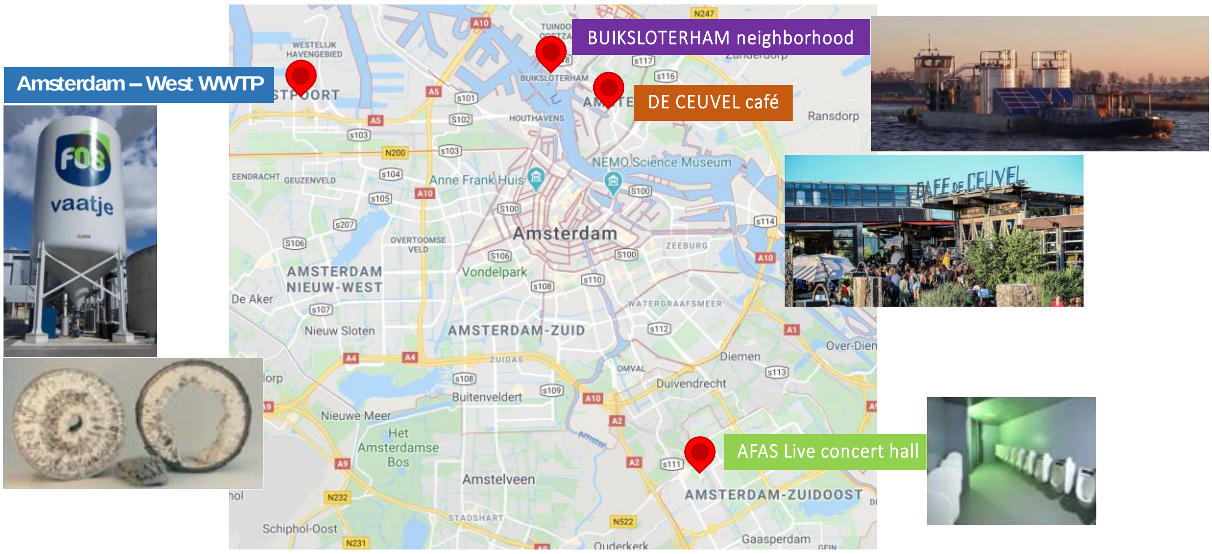

	Household/Neighborhood	Municipal Wastewater Treatment Plant	Sludge incineration facilities
Waste stream	Urine * * Blackwater *	Sludge digestate Sludge liquor * WAS thickening liquor	Sewage sludge ash
Recovery Process	Fluidized bed, or continuously stirred reactor w/ addition of Mg * *	Aerated reactors w/ addition of Mg * Other	Diverse thermal and chemical processes
Product	Struvite * *	Most common: Struvite * Also: Calcium Phosphates	H_3PO_4 Calcium phosphates MAP

* DE CEUVEL
* BUIKSLOTERHAM
* FOSVAATJE

Figure 10: P recovery case studies in Amsterdam. Fosvaatje is the name of the struvite reactor located and the Amsterdam West WWTP. Although its main purpose is to recover struvite from the sludge line of the WWTP, urine from waterless urinals at the AFAS Live concert hall are also transported to the reactor.

TASK 6.1: P RECOVERY DEMO	2019									
	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec
Benchmark studies and site visits	■	■	■	■	■					
Literature reviewn + consulting with experts in the field	■	■	■	■	■					
Site visit Noorderhoek - Desah (equivalent to Buiksloterham - Case Study #2)	■									
Meeting at Waternet headquarters (Amsterdam Foosvatje - Case Study #3)		■								
Site visit De Ceuvel - Metabolic (Amsterdam - Case Study #1)		■								
Site visit VUNA - Eawag (Switzerland)					■					
Design and feasibility studies				■	■	■	■	■		
Design of the container, layout				■	■					
Feasibility study - economical evaluation of VUNA system					■	■				
Design/engineering of lab setup						■	■	■		
Construction and implementation at temporal location NL							■	■	■	■
Installation of container + construction works							■			
Procurement of lab equipment							■	■	■	
Installation of equipment in container								■	■	■
MILESTONE: DEMO ready and operating										■

Figure 11: Time schedule for the implementation of Demo 1

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2. CHANGES WITH RESPECT TO ORIGINAL DOA

An overview of the plans foreseen for Demo 1 in the original CINDERELA DoA, is shown in Figure 12, compared to the revised approach. The pilot plant was proposed to be set up at the NIGRAD office building in Maribor, which has a sewer system built for 200 person equivalents, and would consist of (1) a urine collecting system (one special urine collector per floor) and (2) 200 L struvite reactor in the basement.

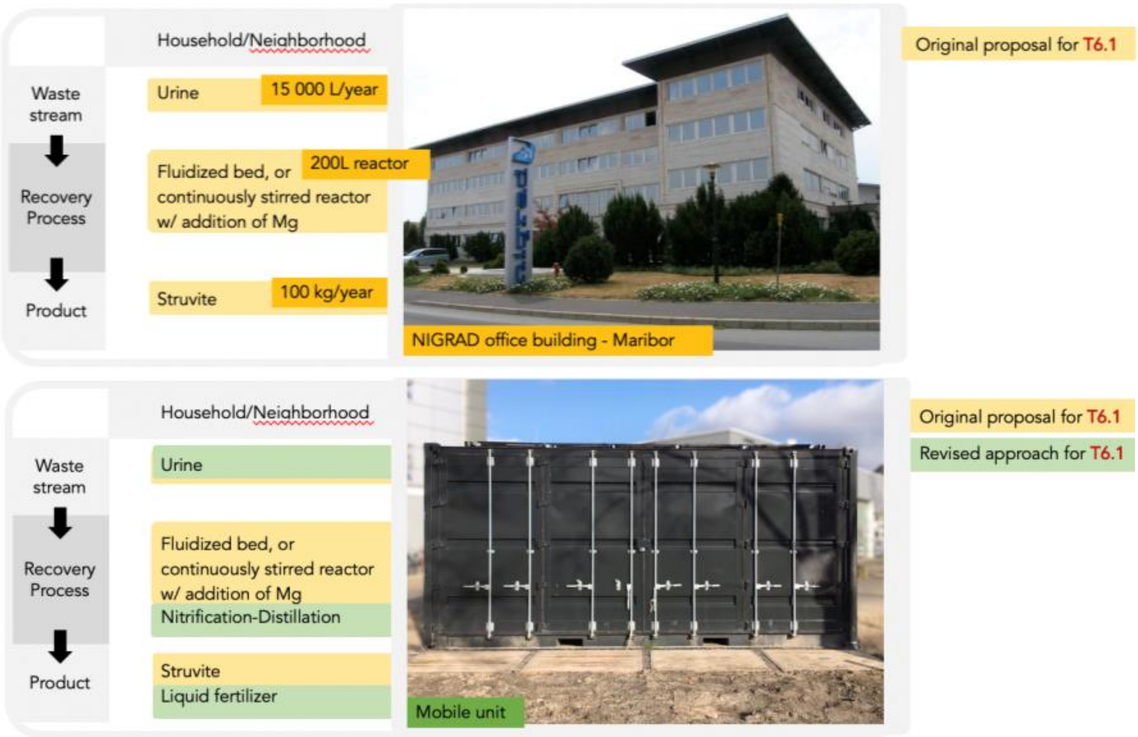

Figure 12: P recovery approach as described in the DoA (above), Revised approach executed (below)

The following sections discuss the motivation behind the changes made, which include:

- **Location:** from a stationary pilot at an office building to a mobile pilot, set up inside a 20ft shipping container, to be used at festivals and public events.
- **P recovery process & resulting product**: from struvite precipitation to complete nutrient recovery as a concentrated liquid fertilizer.

These changes, in turn, entail changes in the

- Capacity of urine treatment, amount of P recovered originally proposed, and end use of the fertilizer.

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2.1. Location and P recovery stream

2.1.1 Mobile vs. stationary demo

After evaluation of the NIGRAD office building layout and sewer system, it was considered that retrofitting the existing piping system for urine separation would require important construction works which would (1) be a significant economic investment and (2) would disrupt the work in the offices.

Two alternatives to the NIGRAD office building, within Maribor, were considered: the Ljudski vrt stadium, which had been reported to have urine separation toilets, and the Zrkovci wastewater pumping station. As far as we are aware, the Ljudski vrt stadium does not currently have urine separation toilets. As expected, the wastewater sampled from the Zrkovci pumping station was too diluted and had a low concentration of P (8.8 mg/l at the time sampled: 28/10/2018 at 12:30). It was therefore decided to opt for a mobile pilot plant approach. The pilot plant, including toilets, could then be used in public locations with large gatherings of people (e.g. at the Lent festival in Maribor) increasing visibility and outreach, and raising public awareness of the need for recovery of valuable resources and transitioning to a circular economy. Furthermore, the need for additional toilets in public locations/gatherings could result in an interesting business model for the urine treatment system.

NIGRAD office building	Municipal wastewater network	Ljudski vrt Stadium	Mobile toilet unit
Retrofitting of toilets and sewer piping not feasible	Phosphorus concentration at accessible pumping station too low	Urine separation toilets not available	To be constructed & optimized in NL, then transported to events, including Lent Festival in Maribor

Another advantage of the mobile unit approach, is that the pilot could be built and optimized over the course of several months in proximity of TU Delft. The Marineterrein of Amsterdam (Kattenburgerstraat 5) has been the location chosen to install the refurbished 20ft high cube shipping container housing the pilot plant. The pilot is currently serving as a toilet facility for the workers of the AMS institute, as well as for visitors to the Marineterrein, and the fertilizer generated will be tested in the urban gardening greenhouse next to the facility (Figure 13). Following operation and optimization of the pilot at this location, the container will be shipped by truck to different events, including the Eurovision Song Festival in Rotterdam in May 2021 and the Lent Festival in Maribor, Slovenia in June 2021, where it will serve as additional toilet facilities for the events.

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Figure 13: 20ft high cube shipping container – housing the P recovery demo - being installed at the Marineterrein in Amsterdam (Kattenburgerstraat 5) in early October 2019.

2.1.2 P recovery from source-separated urine vs. blackwater

We evaluated the possibility of treating blackwater, rather than source-separated urine, as in the case of Buiksloterham (build by the company Desah). The Desah approach in Buiksloterham is minimizing the dilution of the excreta by means of vacuum toilets with low flush volumes, and anaerobically digesting the blackwater (with production of biogas), before P recovery in the form of struvite.

The appeal of this option is that P is recovered from the feacal fraction as well as the urine, and a more holistic approach to toilet waste handling. Furthermore, biogas as renewable energy source is produced. However, the added level of complexity (production of flammable biogas, + handling of feacal matter) was too great for the current budget and scope, and this approach was discarded. Furthermore, in Buiksloterham, nitrogen will not be recovered.

2.1.3 Toilets for source-separation of urine

Once decided on source-separation of urine, quite some effort was devoted to investigating which types of toilets to use. Previous initiatives have used waterless urinals in public spaces. (e.g AFAS live), however this limits the urine donation to males, and narrows the application of this technology (could not be implemented in new construction buildings for example).

“NoMix” toilets are no longer of the market but the Swiss brand Laufen, together with EAWAG and EOOS, designed a new prototype of urine diversion toilets that we we were given the possibility to test out. In addition of having two new prototypes of the Save! toilet, Laufen also provided a waterless urinal. Dry toilets were not considered, due to to the increased complexity of having to manage the faecal waste.

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2.2. Recovery method

The literature review and benchmark case studies analyzed revealed two "weak spots" of the struvite P recovery approach, which led us to evaluate different P recovery options:

- <u>The end use of product</u>: Struvite is a solid slow release P fertilizer, which can have some advantages for certain agricultural practices. However, it is not an optimal product for urban farming.
- <u>Recovery of N:</u> After struvite precipitation, the P-depleted urine leftover is still a waste stream, with left over nutrients (notably nitrogen). P recovery efforts via struvite precipitation, are increasingly linked to efforts to recover N (e.g. by ammonia stripping).

The simplest alternative considered was the direct use of urine as fertilizer. However, as discussed in section 1.2.1., disadvantages to this approach include: impractical handling of large volumes, volatilization of N, and the presence of pharmaceuticals. Different approaches can be used to stabilize, purify and concentrate urine, but many are still in research and development stage. The VUNA, or nitrification-denitrification approach, however, has already been extensively researched, implemented on the full-scale and commercialized.

The main differences between struvite precipitation and the VUNA (or Nitrification-Distillation) approach are summarized in Figure 14. Complete nutrient recovery from urine, as developed by VUNA, requires:

(1) Stabilization of the urine in a nitrification reactor: to avoid bad odor and volatilization of ammonia. Nitrogen is stabilized as ammonium nitrate. A large part of the pharmaceuticals, and the compounds that produce odor are removed in this step.
(2) Purification of the urine with and activated carbon filter: for removal of pharmaceuticals.
(3) Concentration of the urine with a distillation system: to reduce the volume of fertilizer to be transported, and to recover distilled water.

The product obtained is and ammonium-nitrate liquid fertilizer.

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Characteristics	*Struvite precipitation*	*Nitrification/Distillation*
Main product	Struvite (phosphate mineral) $MgNH_4PO_4 \cdot 6H_2O$	Concentrated nutrient solution
Primary nutrient recovery	P N Medium, mainly phosphate	N P K + all other nutrients High, nearly all nutrients
Primary nutrient loss	N K + other nutrients High, most nutrients in the effluent	Low, little ammonia volatilization
Sanitisation/ disinfection	Medium, depends on drying	Complete (during distillation)
Malodour removal	No	Yes
Pharmaceuticals (trace organic compounds)	No degradation	Partial degradation (nitrification)
Energy demand*	Very low (manual reactor) Low (automated reactor)	Medium ~ 50 $Wh \cdot L_{urine}^{-1}$ nitrification ~ 100 $Wh \cdot L_{urine}^{-1}$ distillation
Volume reduction	High Urine to struvite: concentration factor 250 – 630**	High Liquid volume: concentration factor 20 – 30
Other outputs	Phosphate depleted urine	Distilled water, excess sludge
Process complexity	Low (manual reactor) Medium (automated reactor)	High
Development level	High	High

Figure 14: Comparison of struvite precipitation versus Nitrification/Distillation (or VUNA) taken from VUNA Handbook on Urine treatment[18]. Degradation of pharmaceuticals in nitrification is partial, but can be complemented with filtration through activated carbon.

For the CINDERELA demo case, we favored a complete nutrient recovery approach over the original struvite approach based on the following points:

- Nitrogen, Potassium and valuable secondary nutrients (boron, iron, nickel, …) are recovered along with the P.
- The full waste stream is treated, with full removal of micropollutants, pathogens, and bad odour.
- The liquid high-grade organic fertilizer produced can be directly used for hydroponics/urban farming.
- Distilled water is obtained as a valuable by-product.

The main drawbacks, when comparing struvite precipitation with nitrification-distillation are the higher level of complexity, and the energy demand. We will evaluate if the burden of a higher cost, need for skilled labor, and energy demand will be offset by the advantages of this process by means of LCA, LCC and S-LCA in WP7 (see section **Error! Reference source not found.**).

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2.3. Capacity of the demo

The change in approach from struvite precipitation to nitrification-distillation resulted in some changes with regard to the capacity of urine treatment and amount of P recovered originally proposed. Because the system for nitrification-distillation is more complex than the struvite precipitation system, a lower flow of urine (i.e. L/d) can be treated for the same budget. The capacity of urine treatment is, thus, lower than originally planned, designed for a maximum capacity of 30L/d of urine, which amounts to a maximum of 10,950 L assuming 365 days of operation (*vs*. 15,000L in the DoA).

The expected outcome of the current demonstration unit compared to the outcome predicted in the CINDERELA proposal is presented in Table 1Table 2. As a consequence of the lower urine treatment capacity, less P than originally planned, will be recovered. The original proposal stated 100 kg of struvite produced per year, which corresponds to 12.6 kg of P. Assuming the urine P concentration used in the DoA calculations (840 mgP/L if 100 kg struvite are recovered from 15,000L urine), this means that with a maximum of 10,950L of urine treated per year we would obtain a maximum yield of 9.2 kgP. Nevertheless, it is likely that the actual amount recovered will be lower than this. A thorough review of literature carried out during the design of the demo indicates that the P concentration in (stored) urine, while variable, is more likely to be in the range between 200 and 400 mg/l P. The average P concentration (and its variability) in the urine recovered in our demo will be assessed, contributing to the existing body of literature and allowing for more accurate estimations of the recovery potential of such nutrient recovery systems.

With regard to nitrogen recovery, Table 1 shows that the amount of N recovered with the complete nutrient approach is almost 10-fold higher than the with the struvite precipitation approach, even while treating almost one third less of the urine volume. 100kg of struvite per year amounts to 5.7 kg N recovered per year. Assuming an average N concentration of 4.4g/L in urine, 15,000 L of urine contain 66 kg of N. This means that via struvite precipitation, less than 10% of the available nitrogen is recovered in the struvite crystals. In contrast, with the complete nutrient recovery approach, all nitrogen (with negligible losses) in recovered in the form of ammonium nitrate.

Table 1 Quantity of waste and material production in one year of demonstration of P extraction: Original proposal (Amendment - AMD-776751-2) *vs*. revised proposal.

	Country	Waste processed	Material production	Use
DEMO 1 (1 year of production)	SI	15,000 L of urine	0.1 on of struvite = 12.6 kg P = 5.7 kg N	As fertilizer in NIGRAD's green area
DEMO 1 (1 year of production) **- revised**	NL + SI + Other	10,950 L of urine	548 L of liquid fertilizer = 9.2 kg P = 48.2 kg N	As fertilizer for urban gardening

* 1 kg of Struvite ($NH_4MgPO_4*6H_2O$) contains 1.26 kg of P and 5.7 kg N. This can be calculated based on the stoichiometry of the compound.

It is difficult to give an estimation on the population equivalent (PE) that our demo can serve, given that its main goal is to be used and festivals and large public events. Assuming the volume of urine obtained per visit to the toilet is on average 300 ml, this would mean that the pilot could receive on average 100 visits per day. Nevertheless, there is a buffer/storage tank before the nitrification reactor of 300L, which gives room for some variations in this number.

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2.4. Fertilizer use

The DoA proposed using struvite produced in NIGRAD's green area. Given the change in location, we plan to first use the fertilizer produced at the initial location of the demo, to be tested in the AMS Institute's urban garden. It remains to be determined, if the fertilizer could also be given away/sold at the festivals and events, and the business model to be built around it.

Regarding the expected quality of the fertilizer produced: fertilizer test by VUNA with their AURIN product showed that, for ryegrass grown under controlled conditions, urine-based fertilisers performed just as well as reference commercial chemical fertilisers (please see VUNA's Final Project Report 2015[19] for details). We are informed by private communication with VUNA that, since then, AURIN has been tested and used successfully on different crops including tomatoes, corn, wheat, eggplant, lettuce, colza and basilicum, in France, Switzerland and Germany.

The fertilizer product, if the pilot is operated correctly, is expected to be safe with regard to presence of potential pathogens and pharmaceuticals. The distillation stage of the urine treatment process for concentrated nutrient solution, acts as a form of pasteurization (high temperature during a prolonged period), and will inactivate pathogens in urine. Pharmaceuticals in urine are partly degraded during urine nitrification, but are removed further (>90% for all tested compounds by adsorption onto activated carbon (please see VUNA's Final Project Report 2015[19] for details)

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3. DEMONSTRATION PLANT

3.1. Engineering and design

The demonstration plant was designed to have a capacity of 30L/d of urine. VUNA, the spin-off company from EAWAG, assisted and advised on the design. Special care was taken to minimize the cost of the setup and to select and design the equipment to be as visual, informative and appealing to the public as possible. A new prototype of Save![1] urine separation toilets, commercialized by Laufen, are included in the demonstration plant.

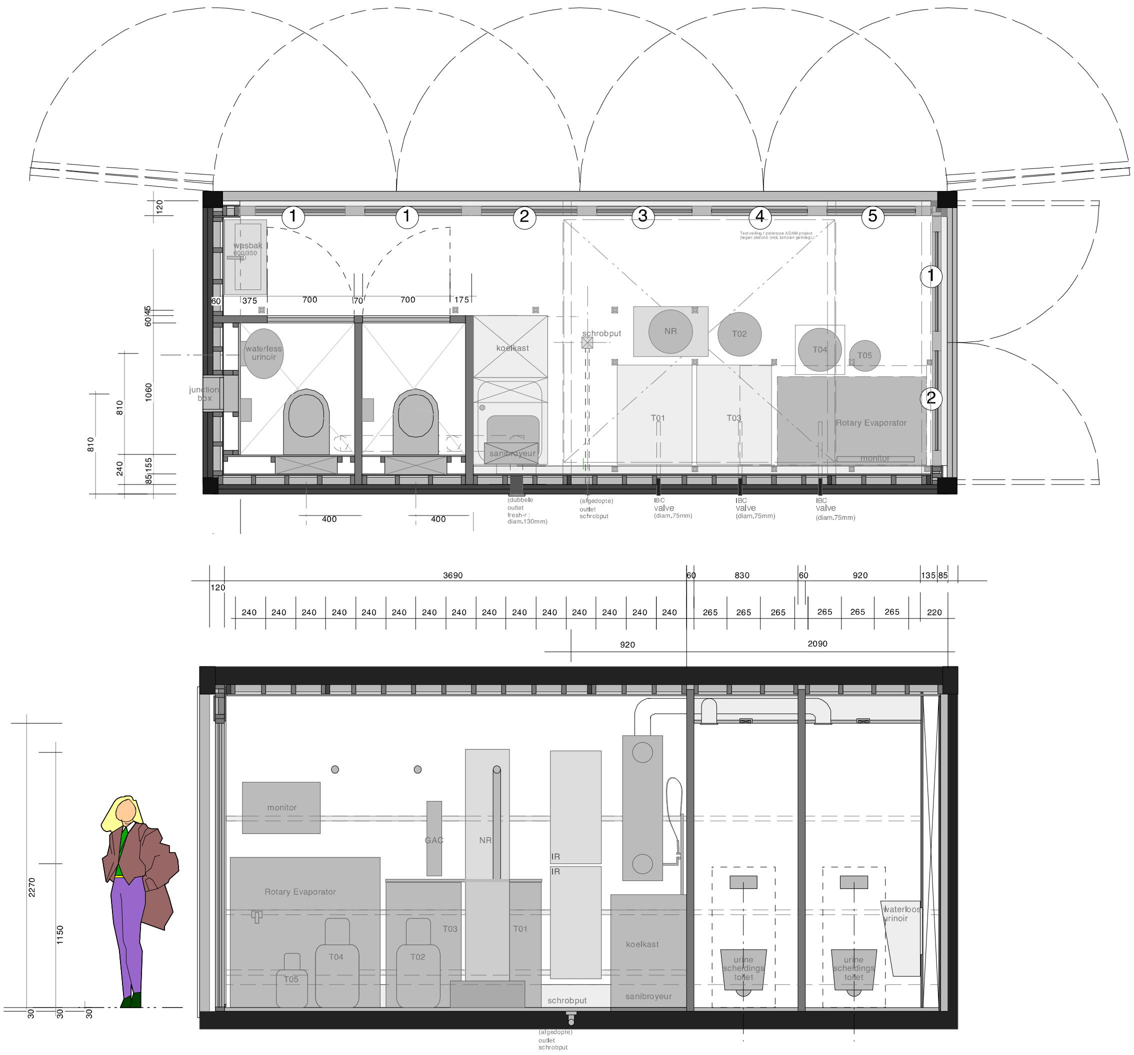

Figure 15: Design of the container: top and side view.

[1] http://www.urinetrap.com/

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3.2. Set-up of the demo

The set-up of the demonstration plant and its different components is schematically depicted in Figure 16. Pictures of the completed pilot, including the toilets and the laboratory setup, are shown below (Figure 17).

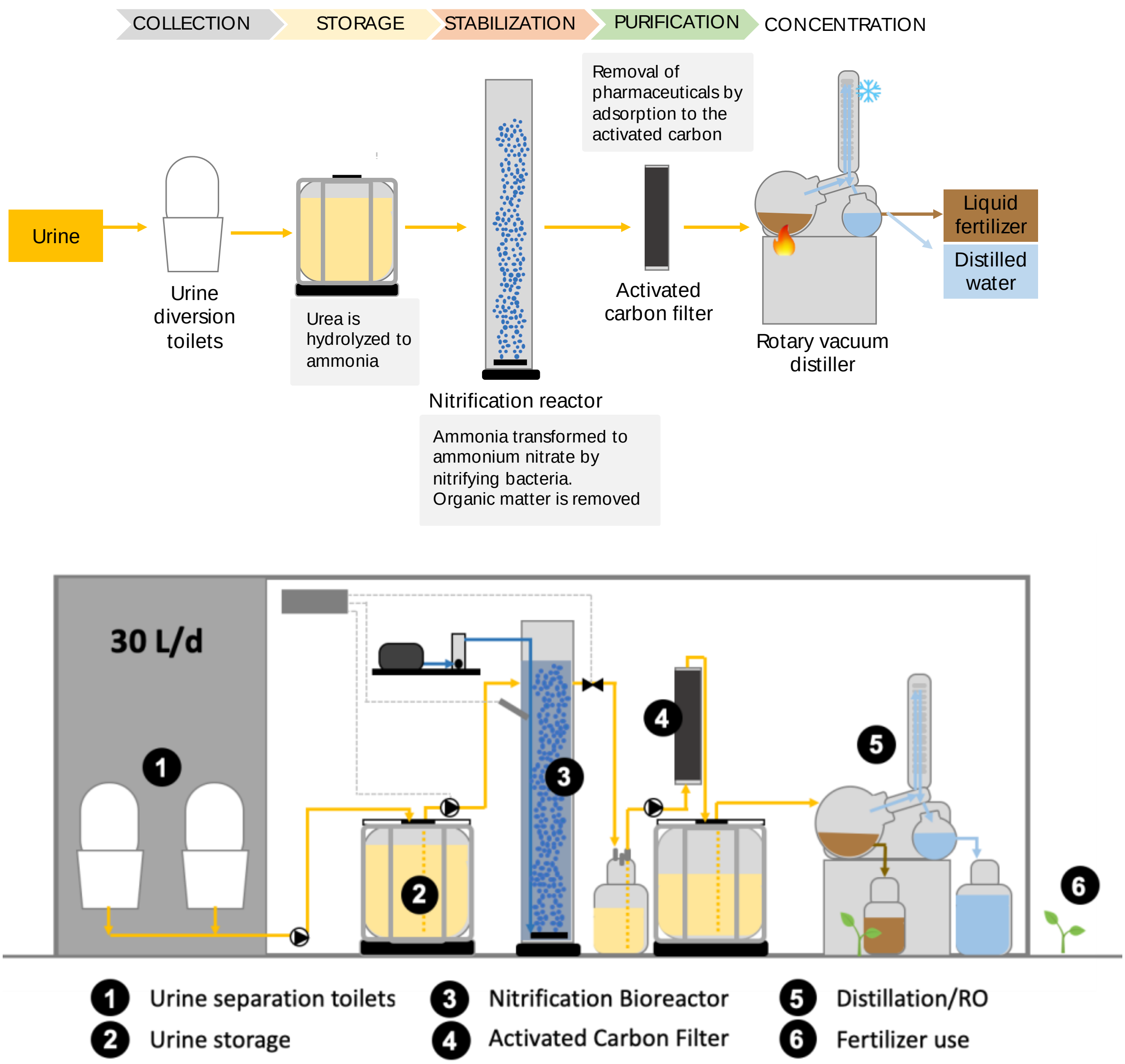

Figure 16: Schematic drawings of demonstration setup for the conversion of urine into fertilizer

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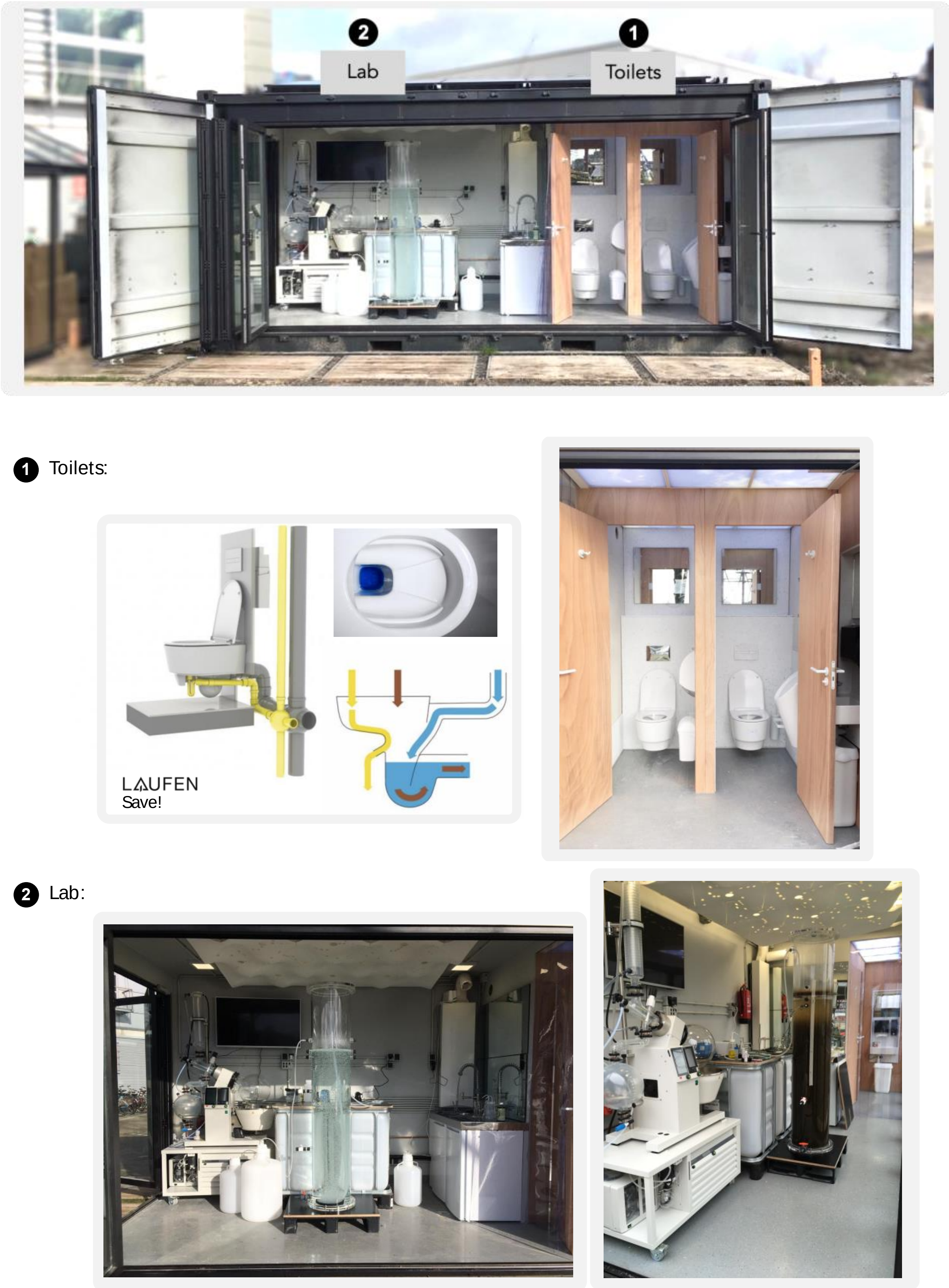

Figure 17: Pictures of the pilot plant including the toilet and laboratory.

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3.3. Product obtained

The product from the complete nutrient recovery VUNA process is a dark liquid fertilizer with a faint soil-like smell (Figure 18).

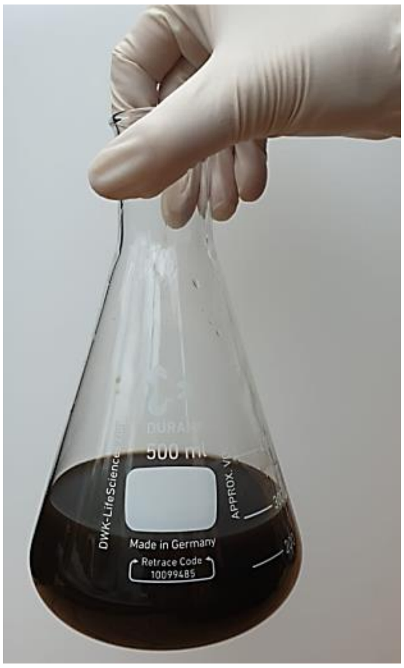

Figure 18: Liquid fertilizer obtained from the VUNA complete nutrient recovery process

The expected composition of the liquid fertilizer obtained is shown in Table 2. The actual average composition of the urine collected and of the concentrated product are yet to be determined.

Table 2 Urine composition (based on literature) and potential recovery in liquid fertilizer

	Urine	**Liquid Fertilizer product**
	g/L	g/L
Total Phosphorus	0.3	6[1]
Total Nitrogen	4.4	88[1]
Sulfate	0.7	14[1]
Potassium	1.4	28[1]
Sodium	1.7	34[1]
Chloride	2.9	58[1]
Calcium	-	0.4[2]
	mg/L	mg/L
Magnesium	-	< 4[2]
Iron	-	0.6[2]
Manganese	-	0.4[2]
Boron	-	17.4[2]

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	Urine	Liquid Fertilizer product
Cobalt	-	0.1[2]
Copper	-	0.4[2]
Chromium	-	0.2[2]
Zinc	-	14.2[2]
Cadmium	-	<0.05[2]
Nickel	-	<0.1[2]
Lead	-	0.27[2]

[1]this is potential (theoretical) recovery. Still to be determined by analysis of product.
[2]based on AURIN fertilizer product commercialized by VUNA

4. FUTURE WORK

4.1. R&D

There is an existing body of literature on source-separation of urine, complete nutrient recovery, and the use of urine (or derived products) as fertilizer. We aim to contribute to the growing knowledge on these topics by looking into:

- Urine source-separation:
  - User acceptance
  - Efficiency of new prototype of urine-diversion toilets: dilution by flush water, maintenance required to avoid clogging
- Nitrification of urine:
  - Optimization of operation to enhance process stability and avoid nitrite accumulation
  - Emissions of nitrous oxide (greenhouse gas that can be produced under certain conditions
- Alternatives to distillation:
  - Using reverse osmosis (RO) or reversed electrodyalisis (EDR) as an alternative to distillation
- Fertilizer use:
  - Use in hydroponic systems/urban farming

4.2. Monitoring and Sustainability - WP7

The CINDERELA P recovery demo showcases only one, of numerous approaches to recover P in the wastewater chain. The difference in carbon footprint and economic sustainability of different P recovery scenarios will be assessed in more detail within WP7, by means of LCA, LCC, and S-LCA analysis. In deliverables D.7.4 “Environmental LCA assessment report for each pilot demonstration” and D.7.5 “LCC and S-LCA assessment report for each pilot demonstration” sustainability will be assessed accordingly to the metrics and methodology included in deliverable D.7.2 “LCA, LCC and S-LCA methodology report”. Life cycle techniques will be applied to compare the P recovery scenario of the Cinderela demo case with traditional alternatives for wastewater treatment. A specific LCA, LCC and S-LCA study will be performed in order to identify the sustainability compared performance. Furthermore, it will carry out a feasibility study of the Environmental Technology Verification (ETV) for the proposed technology in deliverable D7.3 ETV generic protocol for SRM based construction products and as a summary of all the information, a report in form of a “sustainability footprint declaration” will be included in the final deliverable D7.6 Final Report including “Sustainability Footprint Declarations” and Policy Recommendations Paper.

4.3. Communication, exploitation, dissemination - WP8

Special care has been taken in the design of the CINDERELA P recovery demo, to make it as visual, informative and appealing to the public (e.g. the outside of the container has been painted by a street artist (Figure 19), the front façade of the container can be fully opened to showcase what’s inside - Figure 17, there is a screen on the wall that can project information,…). We chose a “mobile” pilot unit approach, to be able to transport and install the setup at different public events, in which visitors can make use of the toilets and witness how urine is converted to fertilizer in the lab setup. Planned event include Eurovison in Rotterdam, Lent Festival in Maribor in 2021. Our goal is to use this demo as a platform to make the public aware of the importance of P recovery, the existence of

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different alternatives and the imperative need to transition to a circular economy in both nutrient management and construction practices.

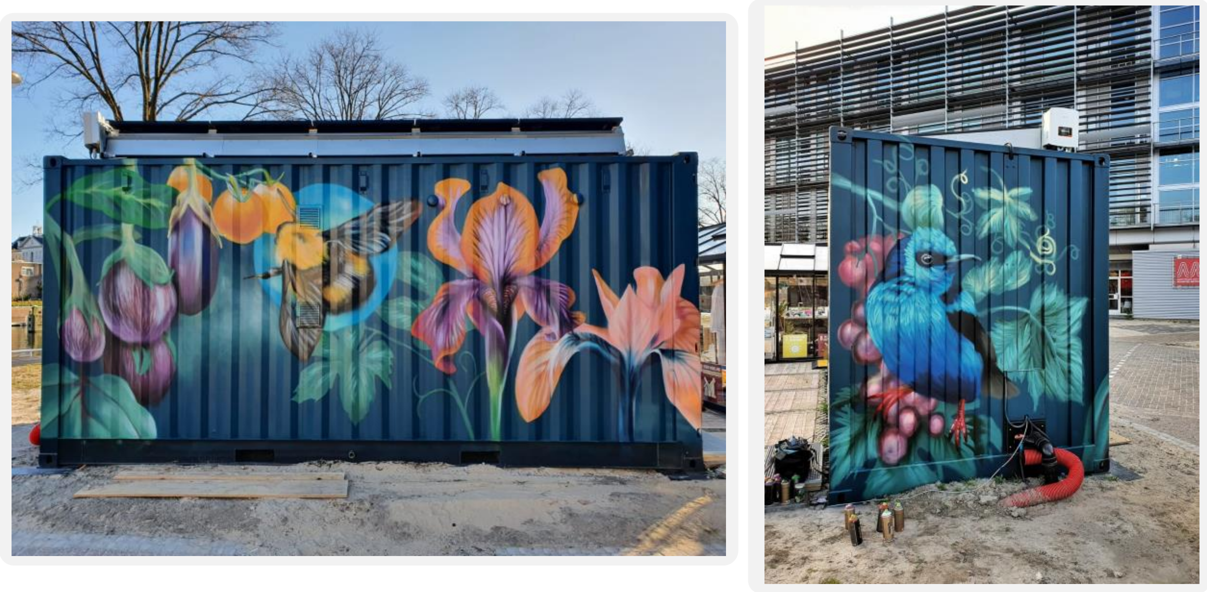

Figure 19: Street art on the shipping container housing the demo plant.

In addition to the dissemination events, and all the communications surrounding it, we aim to publish 2 papers in popular science journals, and 1-2 in peer reviewed journals.

5. Conclusion

A demonstration plant, composed of two toilets and a laboratory set-up designed to treat and recover phosphorus and other nutrients from 30L/d of urine, has been built inside a refurbished 20ft high cube shipping container, currently located in Amsterdam. The product of complete nutrient recovery is a liquid fertilizer which will be tested for hydroponics and food production.

Early upstream extraction of P, nitrogen (N) and other valuable nutrients directly from urine allows us to demonstrate a cascade waste to resource approach: nutrient recovery from wastewater for use where demand is greatest - as fertilizer, followed by the use of sewage sludge as secondary raw material for application as construction materials component where nutrients would be of no added value, and irreversibly wasted.

The demo will be transported to public events like Eurovision 2021 and the Lent Festival in Maribor, to be used as a platform raise public awareness on the importance of converting what has been traditionally considered waste into valuable resources, of thus transitioning to a circular economy in both our management of nutrients, and the construction sector.

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